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INDEX OF VOL. XVI.—1897.

INDEX OF AUTHORS' NAMES.

N.B.—In this Index (P) indicates that the matter referred to is an abstract of a Patent.
The titles of new books are given in quotation marks.

LIST OF ABBREVIATIONS.

App.	Apparatus
Applic.	Application,-able
Artif.	Artificial
Atmos.	Atmospheric,-sphere
Aurif.	Auriferous
Charac.	Character,-istic,-s
Chem.	Chemical,-s,-istry
Co.	Company
Col.	Colour,-s,-ation,-ing
Comp.	Composition,-pound,-s
Concent.	Concentration,-ing
Decomp.	Decompose,-ition,-ing
Detec.	Detection,-ing
Determ.	Determine,-ation,-ing
Distill.	Distillate,-tion,-ing
Estim.	Estimate,-tion,-ing
Evap.	Evaporate,-tion,-ing
Exam.	Examine,-ation,-ing
Expts.	Experiments
Extract.	Extraction,-ing
Ferment.	Fermentation,-ing
Formn.	Formation
Gen.	Generation,-ing
Impt.	Improvement,-s
Infl.	Influence
Manuf.	Manufacture,-ing
Mat.	Matter,-s
Minl.	Mineral,-s
Modif.	Modification,-fying
Nat.	Natural
Org.	Organic,-ism,-s
Photo.	Photography,-graph,-s
Physiol.	Physiology,-ical
Ppn.	Precipitation
Prep.	Preparation
Prev.	Prevention,-ing
Prod.	Produce,-tion,-ing
Protec.	Protection,-ing
Purif.	Purification,-fying
Qual.	Quality,-tative
Quant.	Quantity,-tative
Rectif.	Rectification,-fying
Separ.	Separation,-ing
Soc.	Society, Société
Sp. Gr.	Specific Gravity
Temp.	Temperature,-s
Util.	Utilisation,-ing
Veg.	Vegetable,-s,-tion
Volum.	Volumetric
Vorm.	Vormals

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INDEX OF SUBJECTS.

N.B.—In this Index, (P) indicates that the matter referred to is an abstract of a patent; (T.R.) indicates that the matter referred to is in the Trade Report.

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INDEX OF NEW BOOKS.

LIST OF ERRATA.

Page.	Column.	Line.	Description.
			1897.
58	2	6	From bottom: for "tested" read "tasted."
400	2	21	From top: for "rags" read "rays."
409	2	23	From top: for "rags" read "rays."
467	1	1	For "Bordes" read "Bordas."
510	2	7	From top. Explanation of Fig 1: for "and" read "α" (alpha).
622	2	10	Second Dissected formula lacks below, "CH_2."
623	2	4	From top: for "Prima seconds" read "Good seconds."
623	2	13	From top: for "1 sq. cm." read "1 sq. decim."
908	2	..	Figs. I. and II. should be reversed, or be numbered II. and I.
974	1	33	From top: add the reference, "This Journal, 1896, 597."

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Collective Index.

A collective index, embracing the whole Journal from the Proceedings of the First Annual General Meeting, 1881, to the close of 1895, is now in preparation, and will be ready shortly. It will contain both a subject-matter and authors' names portion, and will be a volume of about 500 pages, uniform in size with the Journal.

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Feb. 1st.—Mr. E. Andreoli. "Ozone: its Production and Industrial Applications."
Feb. 15th.—Mr. W. J. Dibdin. "The Character of the London Water Supply." Adjourned Discussion.

Meeting held on Monday, January 4th, 1897.

DR. R. MESSEL IN THE CHAIR.

THE SMELTING AND REFINING OF CYANIDE BULLION.

BY ARTHUR CALDECOTT, B.A. (C.G.H. UNIV.).

THE precipitate produced in the MacArthur-Forrest cyanide process by the passage of gold-bearing cyanide solutions through filiform zinc varies in character according to the nature of the ore and the strength of the solutions employed. It is collected by screening in water the contents of the extractors through sieves, of which the meshes may be ¼ inch or less, and then draining off the liquid through a filter cloth. To hasten filtering and drying a filter press may be employed. By this means the bulk of the fine material, with its gold contents, is separated from the coarser portion, which is returned to the extractors. A somewhat coarse sieve is advisable, as the finer particles of zinc, which would otherwise tend to accumulate and clog in the extractors, are thereby removed.

The components of the precipitated gold slimes, as they are called, of which special account must be taken in the conversion into bullion, are shreds of zinc and zinc cyanide, copper from the ore, iron, arsenic, lead, and other metallic impurities of the zinc which are insoluble in cyanide solutions, zincic ferrocyanide, of which the iron is liable to be reduced to the metallic state, zincic hydrate, ferric hydrate, calcic carbonate, sand, and other oxy-compounds which are readily fluxed, and mercury derived from the amalgamated copper plates. From the nature of these constituents they may obviously be divided into those metallic oxy-compounds which readily combine with the fluxes subsequently used, and the metals and cyanide compounds which tend to act as reducers and render the bullion base. These latter it is necessary to oxidize preliminary to smelting.

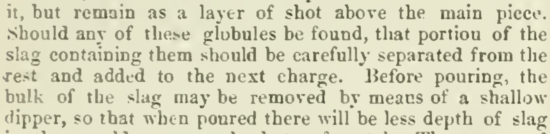
At one time it was common practice on the Witwatersrand to mix the dried slimes with fluxes and smelt at once, but this plan was found to be tedious, to produce very base bullion, and to leave a large amount of gold in the slag; owing to the ammoniacal gases and steam evolved on heating, the crucible was liable to boil over and violently eject the contents, causing loss of gold. By subjecting the slimes to a preliminary roasting on a tray, with free access of air, the volatile compounds were expelled and the base metals oxidized to a great extent. This preliminary roasting, however, of slimes alone, or of slimes mixed with 1 to 3 per cent. of nitre, usually takes a considerable time, as a red heat must be attained with each lot, and there is an appreciable loss of fine gold, which is carried off mechanically in the dust rising when the slimes are handled or stirred during the operation.

To expedite and improve this preliminary roasting the following plan was adopted, and found to give such good results that I have since used it to a large extent. 20 lb. dried slimes, containing 7 per cent. fine gold, were roasted and subsequently smelted in a No. 70 crucible; one pouring only was made, and yielded, besides the bullion, 34 lb. slag, containing 36 oz. fine gold to the 2,000 lb. ton, or 12 per cent. Slag as usually produced in the smelting of gold slimes contains 100 oz. to 200 oz. to the ton, and is crushed and panned, whereby a considerable amount of gold is recovered, but there is also a certain loss of fine float gold; the tailings are then sold to smelters, or put through the mill. This last is a bad plan, as however carefully amalgamation and subsequent concentration and cyaniding be conducted, sufficient gold still remains in the slags to render them three or four times as rich as average concentrates. If, however, the gold contents of slag as produced be reduced to ½ per cent. they can be disposed of to smelters at once, and all losses during crushing, &c. avoided.

The 20 lb. of slimes above referred to were dried until dust just began to be apparent by rising on stirring. They were then thoroughly mixed with 30 per cent. (6 lb.) of their weight of fine dry quartzose sand and 20 per cent. (4 lb.) of finely-crushed nitre, and spread in a layer ¾-inch thick on a large iron tray (6 ft. by 3 ft.) of ½-inch wrought iron, below which a small fire was kindled so as to raise it somewhat above 100° C. A live coal and pinch or two of nitre were then applied to the centre of the layer of slimes mixture, and quiet but intense combustion began, which gradually and regularly spread at the rate of about 6 in. per minute over the whole mass. Dense fumes were given off, and when combustion was complete all volatile materials were expelled, and a red-brown non-dusty product in brittle cakes remained. Three parts of dried slimes yielded 4 parts of this material. When cooled it was mixed with 40 per cent. (11 lb.) of its weight of anhydrous borax and 10 per cent. (2¾ lb.) of dry sodic carbonate, and smelted in a crucible. Fusion took place rapidly without violent

ebullition, and the slag, as stated, was comparatively free from gold. According to the composition of the slimes the above method of roasting, and the proportion of the fluxes may be varied as experience shows. Some slimes may require no heat at all, but combustion once started in the cold will continue, whilst others may require a very slight damping to prevent its being too rapid. The effect produced by the nitre is oxidation of portion of the carbonaceous matter and base metals, and a partial combination with the sand to form a fusible silicate. This serves to cement the particles of the material together and obviates dust. A portion of the zinc is volatilised along with the mercury and carbon dioxide, cyanogen, ammonia, steam, and other gases are evolved. Care should always be taken to use somewhat less nitre than is required to oxidise all the base metals, as, otherwise, during the subsequent fusion, the pots may be corroded and the gold pass into the slag as an alkaline aurate. The sand serves to moderate the combustion during roasting, and by forming a fusible silicate with the potassic hydrate produced by the reduction of the nitre, acts as a flux and protects the crucibles against corrosion by the basic oxides. A thick slag indicates insufficient flux or improper proportions, a corrosive slag requires more sand or borax, or both. Above the roasting tray should be a hood of sufficient size to catch all dust and fume, which is conducted by as direct a flue as possible to a large dust chamber, where the greater portion of the solid constituents are deposited.

In smelting it is advisable to add fresh flux slimes to the crucibles before the surface of their contents is quite fused to lessen possibilities of ebullition. When in perfect fusion and a few minutes before pouring, the contents of the crucibles should be well stirred to cause any globules of metal at the bottom to coalesce. The conical moulds used should be flat, not rounded, at the bottom of the inside, and should be greased and smoked and well warmed, otherwise, on pouring, the bulk of the metal is at once chilled, and solidifies, and the last molten drops are unable to unite with it, but remain as a layer of shot above the main piece. Should any of these globules be found, that portion of the slag containing them should be carefully separated from the rest and added to the next charge. Before pouring, the bulk of the slag may be removed by means of a shallow dipper, so that when poured there will be less depth of slag in the mould to entangle shots of metal. The same end may be attained by using a small conical mould above a tray into which the bulk of the slag overflows. By using dry sodic carbonate and dried or fused borax, which need not be very finely crushed, in place of sodic bicarbonate and hydrated borax usually employed, a great saving of time, labour, and fuel is effected, there being no combined water to drive off during smelting, and the danger of ebullition in the crucibles is reduced to a minimum. By this method a No. 70 plumbago crucible can be used for 10 or 12 pourings equivalent to smelting 2 cwt. of slimes, or a "life" of 36 hours in the furnace. Even then it shows more wear and tear on the outside than corrosion on the inside. In proportioning the fluxes for any particular case the object should be to produce a neutral fluid slag, so that the pots are not corroded, and the gold is not retained. The cost of a little more flux, or the production of somewhat more slag, is a small matter, so long as the greatest possible percentage of gold in the slimes is immediately converted into bullion.



Section of Conical Slimes Shovel.

Above is a sketch of a conical slimes shovel of thin sheet iron for charging the crucibles. It is filled through the opening near the larger end, and the handle being at an angle, permits of the contents being readily delivered into the crucible without spilling or allowing dust to be carried off by the draught of the furnace. In making the shovel, the joining of the two edges of sheet metal should be on the upper side where the opening is. Box furnaces, lined with fire-brick, 22 ins. deep and 20 ins. square, and built with openings level with the floor, are of a suitable size for a No. 70 crucible, and nearly $1\frac{1}{2}$ cwt. of slimes should be smelted in each per 24 hours. Altogether the drying, roasting, and smelting of even a large works should not occupy more than a couple of days. Long-handled basket tongs should be used, and made to fit the crucible. They are hoisted by a thin wire rope passing through a pulley-block on an overhead traveller, and terminating in a hooked rod which is attached to the pivot eye-bolt, by which the two halves of the tongs are joined. Possibly a small quick-action Weston pulley might be used with advantage. The moulds can be made after the type of the wheeled slag-pots used in cupelling silver-lead bullion, or else fitted with trunnions resting on supports, rising above a removable iron tray, on a frame provided with wheels and a handle. By this means, when the crucible is poured, the mould and its fused contents can be wheeled away from the furnace to cool. Any overflow from the mould during pouring is caught in the tray, and when the contents of the mould are turned out they also fall on the tray, which can then be removed.

A species of small reverberating furnace is sometimes also used successfully for smelting with coal; the crucibles come in contact with the flame only, and not with the solid fuel. To remove the greatest possible percentage of gold from the slimes, litharge and a reducer are used at a large rand company; of course this involves subsequent cupellation of the lead bullion. It is probable that in time this method will be very generally adopted where ready facilities for cupellation exist, as very little gold indeed is left in the slag. In companies in outlying districts, however, direct smelting of the slimes in bullion is likely to be practised for some time to come. Refining of gold slimes preliminary to smelting is occasionally done by means of dilute sulphuric acid in wooden tubs. This dissolves the zinc, &c., but removal of the solution of soluble sulphates by filtering and washing or by decantation is a very tedious process unless vessels considerably larger than the usual half-hogsheads be employed. If not thoroughly washed out some of the sulphate is liable to be reduced in the crucible to a sulphide, with formation of a matte. This acid refining is perhaps advisable when so much mercury is deposited in the zinc boxes as to form an amalgam, and it is desired to purify this for retorting to recover the mercury and avoid danger of salivation, were the slimes roasted in the ordinary way. Mr. A. F. Crosse, of the Standard Bank, Johannesburg, informs me that he has successfully experimented with a solution of caustic soda for the removal of the bulk of the impurities in gold slimes by boiling, preliminary to smelting.

Should it be desired to further refine the bullion as above produced, or indeed any base bullion, before casting into bars, nitre may be used to effect this. It is commonly believed that the use of nitre for this purpose in plumbago crucibles is hardly permissible, on account of the corrosion of the pots, with the consequent possibility of the contents finding their way between the fire-bars into the ash-pit. As nitre refining is commonly performed, this hesitancy is justified, but if the following precautions be observed, large quantities of base metal can be readily oxidised and caused to pass into the slag with only slight corrosion of the crucibles. The chief point to be observed is that the nitre must be brought into immediate contact with the molten bullion, so that it does not expend its oxidising power on the plumbago of the crucibles. To effect this, a somewhat large crucible is used, so as to expose as large a surface of bullion as possible. The metal is melted without flux, and when molten, a couple of ounces of nitre are thrown in and immediately pressed gently down and kept in absolute contact with the surface of the molten metal. This is done by means of a plumbago stirrer, fastened to a long, thin bent iron rod. The stirrer is made from an old crucible by sawing a vertical piece out of the side attached to a portion of the bottom, about $2\frac{1}{2}$ in. diameter, which is hence at right angles to the iron rod. Dense fumes of zinc

oxide, &c. are evolved, and when the action ceases the process is repeated until a thick crust of scoria-like oxide forms on the surface. Some flux, consisting of a mixture of 3 parts of dry borax, 1 part of dry sodic carbonate, and 1 part of fine dry sand is added, and when fused the slag is either removed in the usual way, as detailed in the second volume of Aaron's Assaying, or else the pot is poured and the slag detached from the metal, which is replaced in the crucible for the process to continue till it is sufficiently refined. If a borax flux alone be used, there is sometimes considerable difficulty in detaching the layer of slag from the bar after pouring. If the nitre be added to the crucible along with the other fluxes, the greater portion of it never comes in contact with the metal at all, but attacks the crucible and causes violent boiling over of its contents. When, after the final pouring, the slag is solidified, the bar is turned out of the mould and tapped with a hammer to remove the bulk of the slag; the still hot bar is then placed in cold water to detach any slag still adhering.

Boiling of the bars for half an hour in dilute nitric acid, in a heavily enamelled iron dish with handles, considerably improves their appearances, but samples for determining fineness of the bullion should be taken beforehand.

For weighing and mixing slimes, a zinc covered table with raised edges and a row of flux bins at the back is very convenient. Ordinary oval galvanised iron baths with handles are also very useful in handling slimes and slags.

It need hardly be said that the most scrupulous care is required, especially in the earlier stages, in dealing with a material so rich and so easily dissipated as gold slimes. It is probable that the losses which occur and are only partially recovered as "sweeps" from the dust of the floor, which should be cemented, from the flues and fume chamber, from the ash of furnaces, &c. are considerably greater in many instances than are suspected.

Discussion.

Prof. W. Roberts-Austen said that from the assayer's point of view the question of the removal of zinc was of considerable importance. Much of the bullion coming from South Africa, where the cyanide process was largely used, was contaminated with zinc, owing probably to the want of some such process as the author had brought before them. In a triple alloy, in which the bulk was gold, a small proportion of zinc would have a remarkable effect on the relative distribution of the precious and base metal in the mass, which would assay far more accurately if zinc were not present. In fact, with zinc present, it was impossible to get a trustworthy assay, and any process that eliminated zinc and lead from the gold would be of value.

Mr. Arthur C. Claudet remarked that it would have been useful to know the fineness of the bullion after the treatment of the gold slimes, as also the amount of zinc and lead contained in the bullion. With regard to the effect of the presence of zinc in the bullion, he endorsed Prof. Roberts-Austen's remarks; but, in his opinion, it was not zinc only that affected the assay, but the presence of lead exerted a very considerable effect. Most of the mines using the cyanide process used a cheap zinc contaminated with lead, sometimes to the extent of 2·5 to 3 per cent. and more, and the whole of this was necessarily left in the mud with any zinc not removed. An analysis he made three or four years ago of a bar of bullion gave the following results:—

	Per Cent.
Gold	64·30
Silver	6·73
Lead	11·86
Copper	0·97
Zinc	14·93

In a bar of this composition it was difficult and generally quite impossible to get assays to agree by cutting pieces off the bar. Still it was not impossible to get reliable assays from cyanide bars, if the bar was properly melted and a "dip" properly taken from the molten metal in the crucible. The correct way was to use a small ladle and take out enough metal, but so little that it would set immediately, thus preventing excessive segregation of the metals present. Then the pot should be stirred and another dip taken. Such a course gave accurate results if the bar were not very low—say down to 200 or 150. But with a fineness of 500 the dips would not differ more than a half to one millième. The main points to be observed were, first to separate mechanically as well as possible from the slimes the larger pieces of zinc, then treat the slimes with sulphuric acid, as at the Treasury mine in the Transvaal. Having done that, the gold remaining would yield a tolerably pure bullion. The fact still remained, in the zinc boxes pure zinc only should be used, because it was much better to keep impurities out than to have to eliminate them afterwards. He had already gone very fully into this question in a discussion on a paper, in the transactions of the Institute of Mining and Metallurgy, Vol. IV., entitled "Notes on the Treatment of Zinc Box Precipitate," by Robert Gilman Brown, read on May 20th, 1896. The importance of obtaining gold bullion free from lead and zinc, or nearly so, was gone into very carefully in his contribution to this discussion.

Dr. A. F. Fuerst agreed with the previous speakers. By simply roasting the slimes one could get gold 900 fine, and if this process would not do better than that, there was no advantage in using it. He understood from a paper read by Mr. Matthey, lately, that if the amount of silver present in cyanide bullion was less than 30 per cent., it was impossible to get out the gold, because the precious metal concentrated in the middle of the bar.

Prof. Roberts-Austen quite agreed with Mr. Claudet's remarks. In the particular case which he had cited, however, there was very little lead present, or he would have alluded to it.

The Chairman regretted the absence of the author, who was in South Africa. It was possible that some few points in the paper, which had been in the Society's possession some time, might have appeared in print elsewhere; but the delay was unavoidable, and the importance of the gold industry in South Africa fully warranted the bringing forward of the subject now. It was probable, however, that some one of the electrical processes would eventually solve the problem.

A RECORDING PYROMETER FOR INDUSTRIAL USE.

BY PROF. ROBERTS-AUSTEN, C.B., F.R.S.

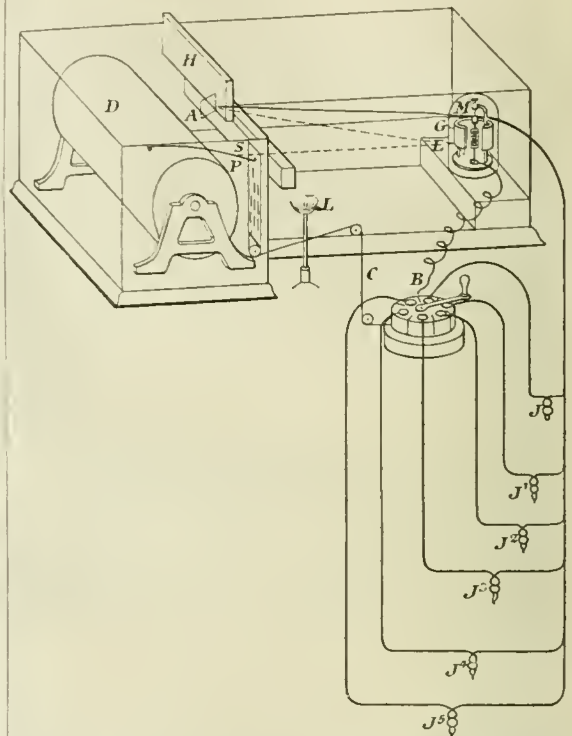
The communication which I have to offer the Society is hardly worthy of being called a paper; but as it may possibly prove to be useful to the members, I have much pleasure in laying it before them. There are practically only two methods which can be readily used in accurate pyrometric work. One of these is the well-known resistance pyrometer devised by Sir William Siemens, or the modification of it devised by Callendar. With this appliance three chemists are at present conducting some most interesting work at Cambridge. Mr. Griffiths, who has attained a degree of accuracy which is simply marvellous, and Messrs. Haycock and Neville, whose work has already been the subject of several communications submitted to another society which meets in this room. I need say nothing about the resistance pyrometer, because, admirable as it is, it is somewhat costly, and need not be employed for work that does not require minute accuracy—let us say, than a tenth of a degree at a thousand degrees—which may be justly claimed for it. The other accurate pyrometric method involves the use of a thermo-junction. The use of the thermo-junction pyrometer, however, dates from 1826, when the elder Becquerel devised and employed a number of thermo-junctions, finally adopting one of platinum and palladium. Joule, in 1859, showed that thermo-junctions might be used for registering the temperature of bars of iron submitted to longitudinal stress. But we owe the extended use of the thermo-junction to H. Le Chatelier, who, by his work from 1885 to the present time, has shown with what extreme accuracy its use may be attended, and who has, in fact, made Becquerel's instrument a very accurate one. Le Chatelier used originally platinum alloyed with 10 per

cent. of iridium, but abandoned that because he found a critical point in the iridium-platinum alloy; and he then employed a junction of platinum wire twisted with another platinum wire alloyed with 10 per cent. of rhodium. In 1873, when so much was thought of the iridium-platinum alloy for metrical standards, Prof. Tate employed a platinum platinum-iridium alloy, and produced his well-known "first approximation to a thermo-electric diagram." That fact has been somewhat overlooked, and I mention it because, for actual industrial working, I have found it well to revert to this platinum platinum-iridium alloy first used by Prof. Tate. Nothing is simpler than its use: it is only necessary to take a thin wire of platinum and twist it with another similar wire alloyed with a small amount of iridium, which is usually 10 per cent. It may be mentioned that a series of alloys are now being produced by Col. Matthey which are very rich in iridium, and it is confidently hoped that their use will enable temperatures to be measured which are higher than the melting point of platinum, to which we are now limited. All that is necessary is to take these wires, plunge their twisted junction into the metal or receptacle the temperature of which is to be ascertained, and the measurement may be effected, provided the free ends of the wires are connected with a galvanometer. The next thing is to calibrate the thermo-junction, and that is done by employing certain known melting points which have already been fixed by the aid of the air thermometer, to which all final references must be made. There is a consensus of opinion surrounding many of these melting points, and we may confidently accept them: the boiling point of water, 100°; the melting point of lead, 326°; the melting point of aluminium, 650°; and, according to the recent determinations of Haycock and Neville and many other observers, the melting point of gold, 1,062°. There is also the melting point of palladium, 1,500°, and of platinum, 1,775°; and, as I have said, Col. Matthey will, I hope, soon communicate to the Royal Society the results of some experiments with alloys of iridium and platinum, which will enable us to measure higher melting points still, at no very distant date. Copper may replace gold with advantage, because it can readily be obtained pure, and its melting point, 1,082°, is sufficiently accurate for practical purposes. The method of calibration is shown in the diagram, only in the case of gold it is always necessary to keep the thermo-junction from direct contact with the metal. Take a small crucible in which gold or copper is melted and allowed to solidify; if the two loose ends of the thermo-junction are connected with a galvanometer, and the junction, suitably protected, is plunged in the metal, the spot of light will traverse the screen, and as the metal freezes the spot will remain fixed till the whole of the metal is solid; that will give the freezing point of gold or of copper, as the case may be, and in that way any particular thermo-junction intended for use may be calibrated. The great advantage of using this instrument for industrial purposes consists in the fact that the spot of light from the galvanometer traversing photographic paper, is able to make a record, and that is done by the aid of a special appliance which I have devised for the purpose, and will now describe.

It consists of a box or camera, which is drawn as if it were transparent. The galvanometer G may be connected with each of the six thermo-junctions (J to J^5) in turn. Each of these thermo-junctions is placed, suitably protected, in a furnace, the arrangement merely indicating the methods of making the connections of the six thermo-junctions with the galvanometer by means of the switch B. The handle of this enables either of the thermo-junctions to be connected with the galvanometer. The light from the gas flame L falls on to a mirror, A, and passes from it to the movable galvanometer mirror M, and is reflected from it through a fine horizontal slit to the drum D. This drum contains the clockwork which causes it to revolve once in 12 hours. Light from the mirror A also falls on to a fixed mirror, E, placed below the movable mirror of the galvanometer, and this enables a datum line to be traced on the sensitised photographic paper by which the drum is covered. The temperature to which any given thermo-junction is raised will be indicated by the position of the line which is traced on the paper by the spot of light falling from the mirror M.

The *number* of the particular furnace under examination is indicated as follows. The switch B is connected by a cord C with a strip of metal P, and this strip, which is

Fig. 1.



supported by a spring S, bears a number of fine horizontal slits. If, for instance, No. 3 furnace is under examination, light from the lamp L will fall through a fine *horizontal* slit in the wall of the box containing the drum, and through *three* slits in the vertical strip P. A triple continuous datum line will be photographed on the drum as long as connection is maintained with No. 3 furnace. If No. 2 furnace is being tested, the datum line will be double, while if the furnace is No. 1, the line will be single.

A slide, H, may be screwed down so as to exclude light from the portion of the camera which contains the drum. This portion may then be removed to the dark room, where the record as traced on the sensitised paper is developed.

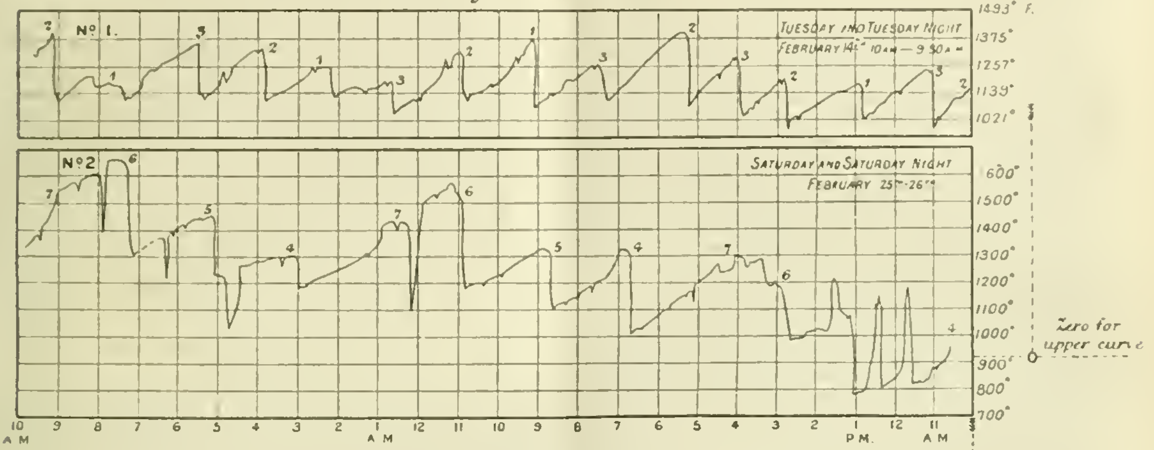
For industrial purposes you have a slight modification, and that is, that instead of a flat glass plate it is convenient to use a drum actuated by clockwork, around which a piece of bromide paper is placed. The drum revolves at a definite pace, and the deviations of the spot of light mark the angular deflection of the mirror and consequently variations in the temperature of the receptacle or of the metal or alloy in which the thermo-junction is placed, the result being that you have little or no difficulty in obtaining an accurate record of what has happened in the immediate vicinity of the thermo-junction. Take a specific case. The chief industrial use of the appliance, so far, has been in connection with the measurement of the temperature of the air supplied to blast furnaces.

The upper curve in Fig. 2 represents a record for $23\frac{1}{2}$ hours of the temperature of the hot-blast supplied to a furnace by a series of stoves, numbered respectively Nos. 1, 2, 3. It is a typical record, and, as it represents an excellent sample of careful and successful gas-firing, it may be well to describe it in some detail.

It will be seen that the record begins near the top right-hand corner, and shows that the temperature of the blast contributed by No. 2 stove (at 10 a.m. on the 14th of February) had an initial temperature of 1,160° F. At the

end of an hour the temperature of the blast had fallen to 955° F., and the gas-man considered that the time had arrived for effecting the connection with a fresh stove, No. 3. As will be seen from the record, this stove contributed blast at an initial temperature of nearly 1,230° F., and the temperature fell in the course of 1 hour and 50 minutes to 1,020° F., when the blast from No. 1 stove was introduced into the horse-shoe main. Inspection of the curve will show the successive variations of temperature. The record, viewed as a whole, shows that the temperature never rose

Fig. 2.



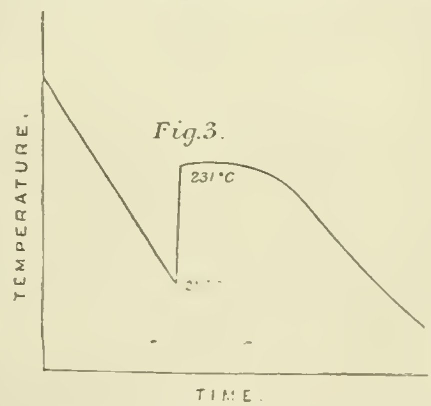
Pyrometer Record.

above 1,400° F., or fell below 950° F. This record shows good, conscientious, careful firing, such as should ordinarily be obtained in works. Complications are, however, introduced by untoward accidents, which, unfortunately, will happen; and the lower diagram, No. 2, Fig. 2, has been specially selected as indicating the existence of a set of conditions which presented much difficulty.

The record refers to stoves No. 4, 5, 6, and 7, and begins at 10.30 a.m. on February 25th. For $2\frac{1}{2}$ hours the temperature of the blast rapidly varied between 1,180° and 730° F., and it is evident the working of the furnace showed that it had a "scaffold," and that cold or "coddled" blast was being freely introduced. This state of things continued for 3 hours longer, when the conditions became more equable. It will be observed that when No. 6 stove is resorted to, the temperature suddenly rises, although it is evident that cold-blast was also being used. Stove No. 6 soon gave place to stove No. 7, and the turn of No. 6 did not come again till 7 hours later, when its temperature proved to be very high (1,570° F.), and the same fact was recorded when No. 6 stove was again used at 7.10 a.m. on the following morning. A consideration of this unsatisfactory record led to the view that No. 6 stove must be receiving a supply of hot, and not of entirely cold air during the period in which it was being heated up. It was found, on investigation, that the valve of No. 5 was leaky, and that hot air from the other stoves could find its way into it—an accident which was of course soon remedied.

The importance of maintaining a constant temperature of blast is manifest. The ratio that the reducing actions bear to the fusing ones varies with the temperature of the blast, and if a furnace has been steadily working at a given rate it will be prejudicial to suddenly increase the activity of reduction in the cooler parts of the furnace, as they would then contribute reduced iron and pasty slag at a greater rate than that at which they could be disposed of in the zone of fusion; hence the tendency of the furnace to choke if the temperature of the blast is suddenly augmented.

One of my old students is present, and he will give you the result of his experience in installing the instrument in works, which shows that in certain cases the appliance may do good service in unexpected ways. I have little else to do except to project on the screen a series of slides illustrating specific cases, to which reference can best be made as they are successively thrown on the screen; but I should like to show some photographs recently obtained, and more particularly one which my friend and assistant Mr. Stansfield secured only this afternoon, of the freezing point of tin. The ordinary freezing-point curve has this form: the temperature of the metal falls until it begins to freeze, then



remains constant. So a curve trace would be a slow fall down to the point when the metal begins to freeze, then a horizontal line during freezing, and then a falling off again when the freezing is complete. But if the angles are sharp, the metal may be considered to be pure. In this case (Fig. 3) you have a remarkable instance of sur-fusion. The line falls 20° below the point of fusion and then suddenly rises, and again falls as the mass cools slowly.

As regards the industrial application of the apparatus, it has been mainly in the relation to metallurgy that the recording pyrometer seems to be useful, and it may be employed in almost every metallurgical operation in conducting which it is desirable to know the temperatures at which products melt or fuse. Is there a copper-smelter who can say that he knows the temperature at which "coarse metal" or "white metal" fuses? However, I am sure that the temperatures are much higher than people usually suppose. In the case of the glass and cement industries, as is indicated by Mr. Redgrave in his book, the appliance would be a most useful acquisition. There must be so many members present who know better than I where and how the appliance would be industrially useful, that I would rather hear what they think than ask them to listen to me any longer.

Discussion.

Mr. Percy C. Gilchrist, F.R.S., was convinced that the beautiful apparatus described by Prof. Roberts-Austen was capable of very wide application. In the metallurgy of steel there were innumerable problems still awaiting the patient investigator. For instance, the difference in temperature between steel hot enough to clear the ladle and make good commercial ingots, and the steel that made skull in the ladle. If an instrument could be produced which could be used by a works foreman for this purpose it would have an almost unlimited application. It would be most interesting to determine the difference in temperature between soft acid and soft basic steel of similar composition as regards Si. C, and Mn. Again, it would be most useful in the Bertrand-Thiel process—a process which he believed would enable one to produce considerably over 100 of steel from 100 of pig,—as it would be most valuable to ascertain, as a matter of fact, and not of mere opinion, whether the temperatures employed in these primary and finishing furnaces were different to the temperatures hitherto employed in the basic open-hearth. He would venture to call attention—by no means, however, in opposition to the beautiful instrument described by Prof. Roberts-Austen, but as a rough-and-ready means of arriving at approximate results—to a little appliance which he had brought with him. It was simply a pair of blue spectacles cut into two at the bridge and with a tube each side of the glasses. An observer looking at a piece of red-hot iron with one eye and at iron being welded with the other, could easily calibrate the apparatus: the distance between the two glasses would be a measure of the different temperatures. Again, if one looked on one side at molten pig iron and on the other side at molten steel, one had the data for ascertaining the differences of temperature between the two. He brought this little apparatus forward because it was so difficult to get anyone skilled in the use of the Chatelier pyrometer to go to Austria to register the temperatures used in the Bertrand-Thiel process.

Mr. R. C. Styles said that the instrument before the meeting was already being put into the hands of gasmen at ironworks, who were able to take the readings and were made responsible for them. Mr. Styles then explained at considerable length the details and trials at various ironworks and potteries where the pyrometer had been instrumental in detecting not only the vagaries of furnaces, hot-blast stoves, and kilns, but also the shortcomings of workmen employed in connection with them. Referring to Mr. Gilchrist's question, he thought that although the temperature of molten steel might affect ordinary porcelain tubes, there would be no difficulty in producing tubes which would stand the test.

Mr. R. Forbes Carpenter thought that a constant record of temperatures such as this instrument gave would be very valuable in the Deacon chlorine process, as it was most important that the temperature in the decomposers should not get above 1,000° F.

Mr. A. Stansfield said that tubes suitable for insertion in molten steel could be produced from fireclay, or graphite and fireclay.

Mr. H. G. Greaves observed that Dr. Fenning had found that porcelain tubes could be made to withstand very high temperatures by wrapping them with asbestos and filling the inside with powdered tungsten mixed with carbon. Thus protected, even if the tube cracked the asbestos coating would hold it together.

Mr. W. F. Reid had made experiments with the resistance pyrometer in the cement industry, but had found great difficulties in the way, owing to the alkaline character of the mass, which had to be tested at a white heat. The resistance pyrometer gave accurate results at certain temperatures, but if one went above them it was not reliable. He would like to know the lowest and the highest temperatures at which Prof. Roberts-Austen had found his pyrometer reliable. For many purposes one might want an instrument which was self-recording uniformly. He would also like to know, approximately at least, the cost of the instrument.

Mr. Bertram Blount was certain that manufacturers in many industries would highly appreciate an instrument that would give a reliable measure of high temperatures. In the cement industry, the very haziest ideas obtained as to the temperature reached in the kilns, and an accurate instrument would be of the highest value to manufacturers. He agreed with Mr. Reid as to the difficulties to be encountered, owing to the nature of the materials employed, but thought that they might be obviated if the tubes were covered by a basic sheath. The most reliable method at present available of arriving at information as to temperatures reached in the cement industry consisted in the use of "Seger's cones" (little pyramids of mixtures of silicates which softened at various temperatures pretty accurately determined for each mixture). Such a method, however, could not compare for accuracy with the results obtainable by the instrument before them. He would like to ask Prof. Roberts-Austen how he obtained the pure tin to which he had referred in the course of his remarks.

Mr. W. Thorp enquired whether the wires were liable to undergo change by prolonged use and especially by the action of gases upon them. Also whether in use they were likely to undergo any mechanical change which would necessitate re-calibration. He agreed with Mr. Reid as to the probable usefulness of the apparatus for giving continuous records of fairly low temperatures in a place distant from the point of observation.

Prof. Roberts-Austen, in reply to Mr. Gilchrist's remarks, said that he had made arrangements to send a trained assistant to Austria for conducting accurate measurements, but his plans had been frustrated by the fact that the gentleman engaged had in the meantime been offered other and more remunerative employment. However, the instrument before them was quite at Mr. Gilchrist's service if he would like to take it. He thought there would be no difficulty in determining the temperature of molten steel (taking it to be about 1,500°) if the porcelain tube were encased in one of graphite, as suggested by Mr. Stansfield. The resistance pyrometer of Callendar could be calibrated up to the melting point of gold at least; he had himself, working with Prof. Callendar, calibrated it up to molten silver with ease. He desired to speak in terms of the highest respect of that instrument, and was most anxious that the apparatus which he had brought to their notice that night should not be regarded as a rival instrument. Each was admirable in its way, as had been shown by the work of Mr. Griffiths on the one hand, and Messrs. Haycock and Neville on the other. But if one had a small piece of rare metal it was convenient to be able to put a thermo-junction into it and get its temperature in a way that the resistance pyrometer did not render possible, as, in order to use it, a considerable bulk of metal is necessary. Prof. Dewar was at the present moment employing a thermo-junction for determining those very low temperatures which he had recently attained. For such extreme temperatures on the other side of zero it had been found better to employ the iridium alloy rather than the rhodium. He could not state definitely the price at which the instrument could be produced; the one before the meeting had cost 30*l.*, but it was obvious that economies in its construction could be effected by the use of cheaper materials; and its cost was small when compared with the sums which it had proved itself capable of saving certain manufacturers. It was not patented, and its use, therefore, was at anybody's disposal. Replying to Mr. Blount, if he had used the expression "pure tin" he was only speaking of commercial purity. But bearing in mind the sharpness of the angles of its cooling curve and the dip due to sur-fusion, he thought that it would be admitted that it had a degree of purity which warranted him in speaking of the metal with respect. Mr. Thorp had asked what degree of change the thermo-junction underwent by use; that depended on circumstances. Mr. Stansfield had one particular pet couple which had been carefully used and protected. It had been used up to 1,065°, and in the course of a year had varied only 1° at that temperature.

Mr. Thorp said that what he had wished to know was whether a change was produced by prolonged heating rather than intermittent heating throughout a long period.

Mr. Styles replied that they had used one thermo-couple for six months, but the usual life of a couple was two to three months. No occasion had arisen for recali-

brating them, and the renewal of a wire could be made at a trifling cost.

The CHAIRMAN considered that an instrument such as Mr. Gilchrist had suggested could have but a limited application, owing to the difficulty of obtaining comparative standards of light. He, however, entirely agreed with Mr. Forbes Carpenter that a pyrometer such as had been brought before them must have a wide application in connection with industries such as the Deacon chlorine and sulphuric anhydride processes, in which the reactions which took place were reversible; and would possess great advantages over the resistance pyrometer in that it gave continuous readings, instead of requiring a series of isolated observations. It gave them much pleasure to see the professional element bringing these papers before them, and he trusted that they would have many from the same quarter.

Meeting held on Monday, January 18th, 1897.

DR. R. MESSEL IN THE CHAIR.

THE CHARACTER OF THE LONDON WATER SUPPLY.

BY W. J. DIBDIN, F.I.C., ETC.

THE author has much pleasure in placing before the Society the following results of his experience in regard to the analysis of potable water. As his observations have been more particularly confined to the examination of the character of the water supplied to London, he has naturally taken that supply for the subject of the text, but the main object of the paper is to point out the results of the improved methods employed in the course of the investigation, with the view of submitting them to the criticisms of the Society, as they tend to throw into prominence a point of interest which has hitherto been to a great extent overlooked; this is, that the various matters held in suspension, in presumably pure waters, although small in quantity, are yet often of such a kind as to throw considerable light upon the quality of a sample of water, even after the old methods of analysis fail to distinguish the difference between one sample and another.

For the purpose of recent enquiries into the quality of the water supplied to London, the author has had the opportunity of making daily analyses of the water of the River Thames at the companies' intakes, and of the filtered water supplied by the respective companies drawing therefrom; also of the water supplied by the Kent Company, and by the two companies drawing their supplies from the River Lea and New River.

At first these suspended matters were examined only qualitatively, but it soon became apparent, as the work proceeded, that the quantity was sufficient, if properly collected, to admit of exact determinations, and thus more particularly identify them with the unfiltered river water. The collection was therefore conducted on a much larger scale by subsidence in special glass vessels; but later on it was found that the recently introduced hard filter papers formed an admirable medium by means of which the solid particles could be collected each day and treated with a dilute solution of bichloride of mercury to prevent decomposition, until their microscopical examination, as well as chemical analysis. By this means not only the quality, but the exact quantity was estimated, and thus further light was thrown upon the much vexed question of the analytical examination of water. A modification of this method, in which a specially constructed micro-filter* is employed, has enabled determinations of the relative quantity, in terms of volume instead of weight, of the supended matters to be made on quantities of water too small even for an ordinary analysis. The results of this method are embodied in the table of analyses under the head of "millimetres of suspended matter in micro-filter from one litre of water."

This process was a development of that adopted for collecting the floating particles for the purposes of microscopical examination, and by this means insuring the inclusion of the total number of such particles under one cover glass, thus insuring greater rapidity as well as accuracy.

* *The Analyst*, January 1896.

Previous to this enquiry it does not appear that any such quantitative methods had been employed. It is clear that either vegetable or animal substances may be qualitatively indicated in terms of carbon, nitrogen, ammonia, and so forth; but such methods of expression, though valuable under certain circumstances, convey but little idea of their relative character, or whether they are in a fresh and wholesome condition or in a state of putrefaction.

In the course of some evidence given by the author before the recent Royal Commission on the London Water Supply, he made the following observations:—

"You may have in a tube of water a few floating particles, and when you have carefully isolated these you will find that they are made up of fibres and a number of organisms, and so on, and occasionally a microscopical eel, all of which would tend to show inefficient filtration or storage, as the case may be. Of course, we know that it is with the greatest possible difficulty that you can keep these things out, but, nevertheless, we do not get them (to anything like the same extent) in the Kent Company's water. If the filtration is so efficient as to take out 99·9 per cent. of the organisms of far more minute microscopical organisms than some of these, how is it that these bigger ones get through? It is a matter of surprise that if the filter is of sufficient mesh to stop a marble, how is it that a cannon ball gets through? These things point to the necessity of a more systematic examination of the London water supply. At the present moment London is in a very peculiar position with regard to its protection. In the case of the gas companies, their gas is examined nightly at no less than 23 gas-testing stations, and they are subject to forfeitures in case their gas is below a definite standard; in the case of the water companies, one sample is taken per month, and if it is good, bad, or indifferent it does not seem to matter; no notice is taken. It has often been a matter of surprise to me why there has not been a systematic examination of the water, with such consequences ensuing to the companies as might be considered proper and just if they failed through any neglect. I do not say failed in any case, because it might be an accident, but if they failed through neglect, then they ought to be subjected to the consequences in the same manner as the gas companies are. If in the matter of what concerns our pocket alone—in the matter of gas—it is necessary to have such very strong and stringent precautions as we have, surely one would think that in the matter of water it would be equally advisable that equally good supervision should exist."

In the report of the Commissioners the following observations occur:—

"*As to the Treatment of the Water after its Abstraction from the River.*

"We have further to consider what steps should be taken to ensure the proper treatment of the water that is taken from these rivers for the supply of London. For the water that flows down these rivers must be subjected to certain important processes before it is brought into suitable condition for delivery and consumption. These processes consist of subsidence and filtration, and the quality of the water when delivered depends largely upon the thoroughness with which they are carried out. It does not come within the terms of our reference to lay down what should be the exact regulations as regards filtration, that is to say, what should the proportionate area of the filtering beds, the depth of sand, the frequency of renewal, or the rate at which the water should be allowed to percolate; nor, as regards the subsidence tanks, how many days' storage should be deemed sufficient so as to obviate the necessity of taking in turbid storm water, and to allow of due settlement; but we cannot shut our eyes to the fact that the provision for these purposes differs enormously in the different companies, and in some of them is, to our mind, quite inadequate."

This very pronounced opinion of the Commissioners left no possible doubt as to the correctness of the views placed before them in the above extract. Unfortunately at that time there was no information available as to the exact quantity of matter held in suspension in the water; but the work which has since then been undertaken has enabled this point to be most satisfactorily settled, and we are now in possesssion of reliable data on the point. As will be

seen later on, one of the most satisfactory features of the case is that, while there can be little doubt as to the correctness of the position thus authoritatively laid down in 1893, the improvement which has been effected by the companies since that time has been most marked, and leaves not a shadow of a doubt as to the correctness of the opinion that the companies could and should effect better work in purifying the water from contamination. So far, good; but what guarantee is there that when the present interest in the question is less keen, the condition of things above indicated will not return, unless there is kept up a thorough and complete daily examination of the water supplied by each of the respective companies.

The average results of daily analyses during nearly 12 months ending December 1895, gave the following quantities of suspended matter, in terms of grains per gallon :—

—		Grains per Gallon.					
		Mineral.		Volatile and Organic.		Total.	
Chalk	Kent		0·0007		0·0004		0·0011
Thames	Southwark	0·0031		0·0025		0·0056	
	Lambeth	0·0018		0·0015		0·0033	
	Chelsea	0·0014	0·0016	0·0013	0·0014	0·0027	0·0030
	Grand Junction	0·0010		0·0013		0·0023	
	West Middlesex	0·0005		0·0005		0·0010	
New River	New River		0·0006		0·0005		0·0011
Lee	East London		0·0015		0·0012		0·0027
Thames Intakes	Sunbury		0·4181		0·1179		0·5360

It will be noticed that the average quantity of suspended matters present in the Kent, West Middlesex, and New River supplies were practically identical, namely, 0·0011. 0·0010, and 0·0011 grain per gallon respectively, so that the West Middlesex Company found it possible to so purify and store the water, that in this respect it bears comparison with waters which are generally accepted, in view of their source, as beyond suspicion. This quantity may therefore be looked upon as that unavoidably due to rust and dust, and forms a standard whereby to judge the quality of the remaining supplies. Taking the average quantity of suspended matter found during this enquiry in the water supplied by the Southwark, Lambeth, Chelsea, Grand Junction, and East London Companies as 0·0033 grain per gallon, and deducting therefrom the 0·0011 unavoidable or rust and dust matters, found as above, we have 0·0022 chemically dry grain per gallon as obviously avoidable suspended matters present in the filtered water supplied to consumers. In order that this average factor may be more readily grasped, we may convert it into terms of "mud" of 90 per cent. moisture (the usual percentage present in such matter) actually supplied per annum. Thus the volume of river water supplied by these companies is 131·6 million gallons daily, so that the quantity of avoidable suspended matters present in that supply is equal to 67·5 tons of wet mud per annum. This, however, includes the period during which the suspended matters were at their lowest. If, on the contrary, the estimation is made on the basis of those present in the Southwark Company's supply during the month of April 1895, namely, 0·0385 grain per gallon, the annual average would be equal to 1145 tons.

How far this factor would have been increased had such estimation been made during the bad season of November and December 1891, it is difficult to judge, as such estimations of the suspended matters were not then made; but from the improvement effected in the colour of the water, as well as in its analytical character, it is open to the assumption that the suspended matters largely exceeded those present during 1895, and thus enormous quantities of river *débris* must have been distributed throughout the metropolis during every flood period.

I would direct attention to the new method of estimating the relative volume of suspended matters by means of the micro-filter, which enables such estimations to be made on small samples of water, instead of on many hundreds of gallons, as was the case with these results given in actual weights. Comparison with the gravimetric method shows that for practical purposes the new micro-filter method gives identical results, with the further advantage that the sample is immediately ready for microscopical examination.

The table on page 11 shows the relative quantity of suspended matters present in the various supplies when determined gravimetrically and by the micro-filter, the numbers representing the monthly average of daily estimations.

From that table it will be seen that there is clearly an indication of the quality of the suspended matter from the ratio of mm. in micro-filter per litre of water to grains of actual suspended matter per gallon; thus the average of the Kent Company's water for the six months ending November 1895 gave a ratio of 0·0023 grain per gallon for each mm. in the micro-filter; in like manner the New River Company showed a ratio of 0·0033; the West Middlesex, 0·0022; the Chelsea, 0·0052; the Grand Junction, 0·0057; the East London, 0·0042; the Lambeth, 0·0052; the Southwark and Vauxhall, 0·0056; and unfiltered Thames, 0·0633. From this it is seen that when the water is purer, the weight of suspended matter per unit volume is least—a fact which is evident on microscopical examination; as in the purer waters only a few fibres with a little *débris* were generally present, in contrast with numbers of particles of *débris* and decaying organic matter, with numerous diatoms, &c. in the case of those waters which bear the least resemblance to the Kent.

From a consideration of these tables, another instructive fact is apparent, *viz.*, that the quantity of deposit in the micro-filter becomes referable, within useful limits, to the actual weight found by a gravimetric process of estimation; thus the average depth of deposit in the micro-filter was 0·44 mm. per litre of water, and the average weight of suspended matter found by the gravimetric measurements was 0·0019 grain per gallon, and therefore 1 mm. of deposit from a clean water in the filter equal to 0·0042 grain per gallon. Experiments are now in hand with the view of showing, if possible, that a scale may be used by means of which the micro-filter results may be directly compared with the gravimetric ones now usually made in the case of sewage analyses, &c.

In the course of 12 months' determination of the quantity of suspended matters present in the Kent Company's supply during 1895, I found that the average was 0·0011 grain per gallon. This quantity I have ascribed to unavoidable rust and dust, and it may be safely concluded that any suspended matter less in quantity than this amount may be neglected as of no importance.

Looking to the above comparisons of the estimations by the micro-filter and by the gravimetric method, 0·0011 grain per gallon may be taken as representing one-fourth of a millimetre per litre in the micro-filter; we thus have a standard of comparison which may be usefully employed in such determinations, and we may therefore conclude that if the depth of deposit from 1 litre of water in the micro-filter tube of 2 mm. diameter does not exceed 0·2 mm., it may be safely neglected, unless the microscopical examination reveals the presence of definitely objectionable particles.

My own experience with many waters leads me to think that no serious notice need be taken of any quantity less than 1 mm. per litre, equal to 0·004 grain per gallon, as shown above, or even a larger quantity, unless it is observed there is a definite microscopical evidence of objectionable

Micro-Filter Deposit, 1895. Grains per Gallon.

Company.	June.		July.		August.	
	Mm. in Micro-filter per Litre of Water.	Grains per Gallon.	Mm. in Micro-filter per Litre of Water.	Grains per Gallon.	Mm. in Micro-filter per Litre of Water.	Grains per Gallon.
Kent	0·0	0·0006	0·2	0·0006	0·1	..
New River	0·0	0·0010	0·2	0·0012	0·2	0·0011
West Middlesex	0·1	0·0012	0·2	0·0014	0·2	0·0008
Chelsea	0·4	0·0038	0·5	0·0020	0·3	0·0013
East London	0·4	0·0019	0·3	0·0030	0·2	0·0020
Grand Junction	0·4	..	0·2	0·0037	0·3	0·0031
Lambeth	0·3	0·0026	0·2	0·0020	0·1	0·0018
Southwark	0·7	0·0098	0·7	0·0057	0·4	0·0031
Average	0·3	0·0010	0·3	0·0024	0·2	0·0019

Company.	September.		October.		November.		Average for the Six Months.
	Mm. in Micro-filter per Litre of Water.	Grains per Gallon.	Mm. in Micro-filter per Litre of Water.	Grains per Gallon.	Mm. in Micro-filter per Litre of Water.	Grains per Gallon.	Mm. Grains. Mm. Grains.
Kent	0·8	..	0·4	0·0008	0·5	0·0007	0·3 = 0·0007, or 1 = 0·0023
New River	0·8	0·0013	0·4	0·0009	0·3	0·0008	0·3 = 0·0010, or 1 = 0·0033
West Middlesex	0·7	0·0007	0·6	0·0007	0·4	0·0006	0·4 = 0·0009, or 1 = 0·0022
Chelsea	0·4	0·0024	0·4	0·0016	0·4	0·0013	0·4 = 0·0021, or 1 = 0·0052
East London	1·1	0·0027	1·1	0·0040	0·5	0·0015	0·6 = 0·0025, or 1 = 0·0042
Grand Junction	0·7	0·0018	0·4	0·0013	0·3	0·0016	0·4 = 0·0023, or 1 = 0·0057
Lambeth	0·8	0·0023	0·4	0·0026	0·4	0·0015	0·4 = 0·0021, or 1 = 0·0052
Southwark	1·1	0·0022	0·9	0·0013	0·6	0·0011	0·7 = 0·0039, or 1 = 0·0056
Average	0·8	0·0019	0·6	0·0016	0·4	0·0011	0·44 = 0·0019, or 1 = 0·0042

—	September.		October.		November.		December.		Average for the Six Months.
	Mm. in Micro-filter per Litre of Water.	Grains per Gallon.	Mm. in Micro-filter per Litre of Water.	Grains per Gallon.	Mm. in Micro-filter per Litre of Water.	Grains per Gallon.	Mm. in Micro-filter per Litre of Water.	Grains per Gallon.	Mm. Grains. Mm. Grains.
River Thames at Sunbury.	1·8	0·1718	4·8	0·2020	33·7	1·1775	7·8	1·4870	12·0 = 0·7569, or 1 = 0·0633

contamination and the chemical analysis shows a questionable state of things.

The chemical examination of the character of the suspended matter gave very remarkable results. For the purpose of this comparison the dry matters obtained from the water, either by natural subsidence or filtration, were examined for the nitrogen present in combination with the organic matter. The average percentage quantity found is set out in the following table:—

Nitrogen per Cent. on Volatile Organic Matter.

Chalk	Kent	2·12	
Thames	Southwark	3·44	3·27
	Lambeth	3·42	
	Chelsea	2·97	
	Grand Junction	3·66	
	West Middlesex	2·86	
New River	New River	2·86	
Lea	East London	2·97	
Thames Intakes	Sunbury	4·06	

For comparison, the following results of the estimation of the quantity of nitrogen present in known samples of polluted mud are given:—

Sewage mud	5·20
Known polluted river mud	4·40
Pure river mud from the Fitzroy River, Queensland	2·70

They clearly point to the conclusion that the suspended matters present in three of the companies' filtered supplies approximated more or less nearly in character to those in the unfiltered river waters at the companies' intakes.

Microscopical Character of the Suspended Matters.

The conclusion drawn from the consideration of the relative chemical character of the suspended matters in the unfiltered river water, compared with those in the filtered supplies, is borne out by the results of the daily microscopical examination of all samples collected, the average results of which are set out in the following condensed table:—

*Percentage Number of Samples returned as "Objectionable" * by the Microscopic Examination.*

———	Samples examined.	Returned as Objectionable.
		Per Cent.
Kent	284	18
Southwark	286	47
Lambeth	284	34
Chelsea	282	22
Grand Junction	285	25
West Middlesex	285	24
Average	..	30
New River	286	21
East London	283	30

* "Objectionable" samples are those which contained a comparatively large quantity of *débris*, microscopic eels or anguillulæ, &c., decaying *débris* infested with bacteria, the larger infusoria, such as paramecium, rotifera, &c., epithelium scales, &c.

These percentages were then calculated into total quantities of objectionable matter present on the actual estimation of the weights of suspended matters found, and are thus set out in column 4 of the following table:—

Company.	Average Suspended Matter. — Grains per Gallon.	Microscopical Examination of Objectionable Matters. Percentage in Samples.	Microscopical Examination of Objectionable Matters. Grains per Million Gallons.	Organic Nitrogen in Suspended Matters. Per Cent.	Organic Nitrogen in Suspended Matters. Grains per Million Gallons.	Organic Nitrogen in Suspended Matters. Equal to Organic Matter containing 4·1 per Cent. of Nitrogen, as in Thames at Sunbury. — Grains per Million Gallons.	Average Number of Microbes per c.c.	Average Oxygen absorbed by the Dissolved Matters from Permanganate in Four Hours. — Grains per Gallon.
Kent	0·0011	18	198	0·84	9	220	90	0·018
New River	0·0011	21	231	1·44	16	390	130	0·041
West Middlesex	0·0010	24	240	1·43	14	341	130	0·072
Chelsea	0·0027	22	594	1·87	52	1,268	139	0·064
Grand Junction	0·0023	25	575	2·06	47	1,146	170	0·069
East London	0·0023	30	810	1·35	36	878	277	0·060
Lambeth	0·0033	34	1,122	1·86	61	1,488	291	0·071
Southwark	0·0056	47	2,632	1·68	94	2,293	352	0·078
Intake at Sunbury	0·5360	81	434,160*	1·01	5,414	132,049	16,061	0·128

* NOTE.—The microscopical examination includes with the organic, the mineral matter, which is proportionately larger in the case of the intake waters than in the filtered supplies: hence the objectionable matters appear to be greater by the microscopical examination than those estimated by the nitrogen, which relate solely to organic matter.

The practically complete coincidence between the various methods of examination—namely, the physical, as the estimation of the weight of the suspended matters may be called; the chemical and microscopical examination of their character; the biological examination as to the actual number of bacteria present in a given sample of water; and the strictly chemical examination as to the quantity of oxidisable organic matters dissolved in the water—is very striking.

The unfiltered river water contained a mass of matters of generally indefinite character included under the head of *débris;* but, in addition to these, certain noticeable features were distinct, amongst which the diatomacæ were predominant; entozoic worms, various dyed and undyed textile fibres, such as wool, cotton, silk, &c., were not infrequent; together with numerous infusoria and bacteria.

The results of the examination of the filtered supplies indicated the frequent presence of all these substances, and in the majority of cases it would have been difficult for a careful observer to conclude from the examination of the preparation under the microscope whether it was drawn from the unfiltered river water or from one of the filtered supplies (it being understood that the actual quantity of *débris* taken for examination was equal in each case). Diatoms were almost always present, and not infrequently anguillulæ also; but the most noticeable and objectionable feature was the frequent presence of fragments of matter in a state of decomposition and swarming with bacteria. The presence of such particles cannot be accepted as inevitable in the case of a pure water supply, as, if the filter beds are properly worked, it should be impossible for such matters to pass them, whilst if their presence be due to dust getting into the pure water reservoirs, evidently greater care should be taken to keep them clean. That a particle of dust might be so introduced is not to be doubted, in view of the fact that on a few occasions the Kent supply was found to contain such matters in small quantities, but evidently accidentally so, as compared with their more frequent presence in larger quantities in the filtered river waters.

From the results of the work now put on record before this Society, it is clear that much more may be learnt from the proper collection and examination of the suspended matters, however small, in a sample of water than has hitherto been supposed, and the author ventures to suggest that in future all samples of waters should be so examined.

A great point has been made in recent years of the number of bacteria present in a water, but from the experience now obtained it would seem as if the number of bacteria depended more upon the quantity of suspended matter than on any other factor.

Considerable differences have been obtained in the estimation of the number of bacteria by different observers. It would now seem possible that these differences are occasionally due to the fact that in one sample certain particles of decaying matters may have been present, although absent in another—a result which would account for many hundreds, if not thousands, of colonies being obtained by one experiment, and but few by another, quite apart from the known differences due to varying methods of procedure, temperature, &c.

Bacteriological Examination.

The examination of the daily samples of water for the number of bacteria as determined by the number of colonies developed on gelatine plates, has yielded interesting results, and affords further evidence of the improvement effected in the various filtered supplies.

On page 13 is given a condensed tabular statement of the monthly average number of microbes present in the water collected from stand pipes in various parts of the respective companies' districts.

Having thus reliable data for comparison as to the bacteriological improvement which has taken place in the quality of the water between the years 1892 and 1895, it will be interesting to glance at the still further improvement which took place during the course of the latter year, and of the relation which exists between the quality of the intake waters and the filtered supply.

It will be noticed that the company which supplied water containing the largest quantity of suspended matters—*viz.*, the Southwark and Vauxhall—supplied an average of 352 microbes per cubic centimetre of water throughout the year, this being the largest number of any of the companies. In January, the monthly average was 838, which gradually decreased with the reduction in the quantity of suspended matter during the year to 78 in October, but, unfortunately, rose again to 427 in November, corresponding to the rise of microbes in the Sunbury river water of from 9,900 in October to 45,263 per cubic centimetre in November.

Month.	Kent.	Southwark.	Lambeth.	Chelsea.	Grand Junction.	West Middlesex.	New River.	East London.	Sunbury.
January	89	838	793	127	111	128	274	365	15,135
February	90	545	131	161	201	92	194	318	29,308
March	122	818	238	165	135	294	251	556	29,844
April	135	325	286	188	253	135	114	151	7,080
May	73	170	88	339	284	142	145	78	5,664
June	92	199	204	158	184	231	83	602	16,013
July	79	142	102	95	263	113	83	163	10,774
August	112	164	838	108	119	96	98	123	4,813
September	31	164	124	71	113	80	44	88	2,884
October	40	78	59	61	43	54	56	76	9,900
November	139	427	366	55	169	65	92	494	15,263
Average	90	352	294	139	170	130	130	277	16,061

In 1891–92 the following average results were obtained :—

	4,795	1,594	4,626	3,091	2,612	2,534	2,299	3,387	179,601

The relative value of the various methods of estimating the organic matter in water—*viz.*, the organic carbon and nitrogen or "organic elements," the albuminoid ammonia, and the oxygen absorbed—has been observed in connection with the series of analyses under discussion. The results of 12 monthly averages of daily analyses made during 1892 (which period is selected in preference to 1895, when there was a break in the continuity of the analyses as regards organic elements and albuminoid ammonia) has shown that the oxygen-absorbed method, as used by the Society of Public Analysts, has been found to give the most concordant factor all through. In the case of waters of known origin, such as those supplied to London, it does not appear to be necessary to repeat indefinitely processes which, however valuable in themselves for certain purposes, fail to throw any fresh light upon the general history of the water.

Comparison of the present London Supply with the proposed Supply from Wales.

In order that the exact position of the present supply may be clearly understood, so far as regards the question of relative quality and the extent to which an improvement might be anticipated in the event of the Welsh water being brought to London, I submit the following considerations arising from the comparison of the analyses of the water at present supplied to London with those of the Welsh waters collected by myself in May 1894.

In the following table the analytical factors in each case are placed side by side, so that the specific differences may be seen at a glance. The figures representing the Welsh waters are averages of the samples drawn from the rivers Usk, Edw, Yrfon, Wye, Towy, Ithon, and Clywedog, which were, of course, unfiltered. Those representing the present filtered river water supplied to London are averages of the Southwark, Lambeth, Chelsea, Grand Junction, West Middlesex, and East London. These, however, are given in two columns, one for average summer quality, and the second showing average winter quality. For comparison as a standard, the Kent Company's average supply is added.

On carefully examining the results it will be found that the filtered river water supplied during the six summer months to London contains practically identically the same amount of organic carbon and nitrogen as that present in the unfiltered Welsh waters, *viz.*, 0·128 part per 100·000 against 0·123, whilst during the six winter months it is only half as much again, *viz.*, 0·199.

The oxygen absorbed from permanganate is a little more, *viz.*, 0·057 during the summer instead of 0·048, but only a little more than half as much again, *viz.*, 0·079 grain per gallon during the winter. In like manner the "albuminoid ammonia" in the Welsh water averages 0·0033 against 0·0045 and 0·0055 in the respective summer and winter London supplies excluding the Kent. The colour of the water when viewed through a 2-ft. tube was on an

Average Results of Analyses of Welsh and London Waters.

	Welsh Water, Unfiltered.	London Supply. Filtered River Water. Summer. May to October.	London Supply. Filtered River Water. Winter. November to April.	Kent Company.
Appearance (on examination in a 2-ft. tube)	Generally clear and bright.	Generally clear and bright.	Occasionally turbid	Clear and bright
Colour by Lovibond's tintometer scale { units of red	0·4	0·3	0·3	0·0
" yellow	1·0	0·7	0·9	0·0
Odour at 100° F.	None	None	None	None
Phosphoric acid	Very slight trace	Very slight trace	Very slight trace	Very slight trace
Free ammonia	0·0003	0·0002	0·0004	0·0000
Albuminoid ammonia	0·0033	0·0045	0·0055	0·0012
Chlorine	0·68	1·34	1·32	1·64
Oxygen absorbed in 15 minutes	0·018	0·035	0·044	0·010
" " 4 hours	0·048	0·057	0·079	0·018
Nitrogen as nitrates, &c.	0·022	0·128	0·258	0·401
Organic carbon (parts per 100,000)	0·091	0·090	0·163	0·057
" nitrogen " "	0·032	0·038	0·036	0·017
Total organic elements (carbon + nitrogen)	0·123	0·128	0·199	0·074
" solids	4·3	19·3	23·9	28·6
" hardness	3·1	12·5	17·0	19·5
Permanent hardness	1·6	4·2	4·2	5·4
Cultivation tests by gelatine plates, number of colonies per cubic centimetre.	26	167	298	99
Microscopical examination of matters in suspension	Vegetable *débris*, diatoms, organisms.	Vegetable *débris*, diatoms, organisms, &c., in comparatively large quantities.	Vegetable *débris*, diatoms, organisms, &c., in comparatively large quantities.	Few fibres and occasional organisms.

average even more in the case of the Welsh water, *viz.*, 1·4 against 1·0 and 1·2 in the case of the London supplies.

From this comparison of the average results of nearly three thousand analyses, it is evident that the organic impurity of the unfiltered Welsh water is a little less than that in the average filtered London water, and the inquiry naturally follows as to the specific differences which exist. This is undoubtedly due to two things: first, the quantity of the solid matter in solution, principally salts of lime, giving the London water its greater hardening properties; and second, the comparatively large quantity of floating particles, on which, as has been shown, the number of microbes present to a great extent depends.

The unfiltered Welsh water examined on the spot contained 26 microbes per cubic centimetre, whilst the average London filtered waters contained in summer 167 and in winter 298, the Kent supply containing 90. Of course, it will be noted that these numbers refer solely to the quantity and not to the character of the microbes.

The Effect of Softening the present Supply.

The suggestion has been repeatedly made that the excessive quantity of lime in the London water should be removed by the adoption of the well-known Clarke's process, and it is only natural to inquire as to what would be its effect upon the present filtered river water, not only in removing the excess of carbonate of lime, and thus effecting an enormous improvement in the commercial value of the London water, but also in assisting in the process of purification. With the view of testing this question certain experiments have been made on one of the best London waters, *viz.*, that of the New River Company, in order that the percentage reduction, if any, which might be brought about in the quantity of dissolved as well as undissolved impurity might be ascertained.

The results of these, which are stated in the table below, are perfectly definite, and show that even in the case of the New River supply the reduction of dissolved organic impurity was equal to 25 per cent., as shown by the oxygen absorbed, whilst 64 per cent. of the total hardness was removed and 87 per cent. of the bacteria, and with these the whole of the matters in suspension. If one of the less pure waters had been taken for this experiment, such as the Lambeth or Southwark, doubtless the percentage reduction of impurities would have been larger.

On applying these results to the average analytical figures given in the table, it will be found that the "albuminoid ammonia" in the summer supply would fall by the adoption of the process of softening from 0·0045 to 0·0034, or identically the same quantity as that in the unfiltered Welsh supply, whilst the winter supply would be improved to the quality of the present summer supply; that the oxygen absorbed in four hours would fall from 0·057 to 0·043 in summer, and from 0·079 to 0·059 in in winter, against 0·048 in the Welsh water, or a yearly average of only 0·003 grain per gallon in excess; that the organic carbon and nitrogen would be less in proportion, whilst practically the whole of the "suspended matters" would be removed, and with them 87 per cent. of the bacteria, thus reducing the summer *number* (apart from their character) of microbes from 167 to 21, and the winter from 298 to 38, against 26 in the Welsh water, or an annual

Experiments to ascertain the Effect produced on the Impurities of the New River Supply by the Process of Softening.

Date.	Description.	Examination in a 2-ft. Tube. Appearance.	Colour by Lovibond's Tintometer Scale. Red.	Yellow.	Blue.	Odour at 100° F.	Phosphoric Acid.	Ammonia. Free.	Albuminoid.	Chlorine.
1895. Dec. 16	Untreated water.....................	Clear	0·1	0·5	..	None	V.S.T.	0·0003	0·0059	1·30
	Treated with 9·4 per cent. of saturated lime water.	"	..	0·3	..	"	"	0·0003	0·0047	1·30
" 18	Untreated water.....................	"	0·1	0·6	..	"	None	0·0003	0·0082	1·30
	Treated with 9·4 per cent. of saturated lime water.	"	0·0	0·4	..	"	"	0·0003	0·0069	1·30
" 20	Untreated water.....................	"	0·1	0·6	..	"	"	0·0002	0·0102	1·25
	Treated with 9·4 per cent. of saturated lime water.	"	0·0	0·4	..	"	"	0·0002	0·0068	1·30
	Average per cent. reduction...		100	35	..			Nil	23	

Date.	Description.	Oxygen absorbed in 15 Minutes.	4 Hours.	Total Solids.	Appearance on Ignition.	Hardness. Total.	Permanent.	Cultivation Tests by Gelatine Plates. Number of Colonies.	Microscopical Examination of Matters in Suspension.
1895. Dec. 16	Untreated water	0·029	0·045	23·4	S.B.	17·4	4·2	96	0·3 mm. Tangled mass of fibres, diatoms, bacteria, &c.
	Treated with 9·4 per cent. of saturated lime water.	0·024	0·037	10·6	"	5·4	4·0	12	Trace. Few vegetable fibres, organic *débris*.
" 18	Untreated water	0·035	0·051	25·2	"	17·4	3·8	110	Slight trace. Few cotton and other fibres.
	Treated with 9·4 per cent. of saturated lime water.	0·025	0·049	11·0	"	5·0	4·2	6	Very slight trace. One or two fibres.
" 20	Untreated water	0·030	0·064	24·4	"	17·2	4·5	60	Very slight trace. Few fibres and little *débris*.
	Treated with 9·4 per cent. of saturated lime water.	0·020	0·028	13·8	"	8·5	4·5	16 After 15 days gelatine still solid. 3 moulds and few colonies.	Very slight trace. Little *débris*.
	Average per cent. reduction...		25			64		87	

average difference of + 3. In addition to these enormous improvements the hardness would be reduced in summer from 12·5° to 4·1°, and in winter from 17·0 to 6·1°, or an annual average of 5·2°, against 3·1°, which was the average hardness of the Welsh waters as examined in May 1894.

It would therefore seem that by the adoption of the system of softening, the present supply, in respect of its chemical quality and number of bacteria, would be improved to a degree comparable with that of the Welsh sources.

The discussion on this paper stands adjourned until February 15th.

Manchester Section.

ROOMS OF THE CHEMICAL CLUB, VICTORIA HOTEL.

Chairman: George E. Davis.
Vice-Chairman: Peter Hart.
Committee:

F. H. Bowman.	J. M. Irving.
R. Forbes Carpenter.	M. J. Langdon.
R. S. Dale.	J. Lewkowitsch.
R. Le Neve Foster.	E. Schunck.
B. W. Gerland.	W. Thomson.
H. Grimshaw.	D. Watson.
J. Grossmann.	

Hon. Local Secretary:
J. Carter Bell,
Bank House, The Cliff, Higher Broughton, Manchester.

SESSION 1896-97.

Feb. 5th, 1897:—
Dr. B. W. Gerland. "Further Notes on Indigo."
Mr. R. J. Flintoff. "The Fixing of Pigments with Albumen."

Meeting held on Friday, January 8th, 1897.

MR. GEORGE E. DAVIS IN THE CHAIR.

ON A NEW METHOD FOR THE RAPID VALUATION OF ZINC DUST.

BY ANDRÉ R. WAHL.

ZINC dust is a body in very extensive use as a reducing agent, especially in vat indigo dyeing, and in the manufacture of certain colouring matters. For these purposes, especially in the latter, where the reductions performed are very delicate operations, requiring theoretical proportions of the materials, it is very important to know the actual value in metallic zinc of the zinc dust employed. The methods at present in use are either unreliable or require much time to perform, and I have therefore been led to seek a method at the same time both exact and rapid, and consequently especially suitable for commercial application. The principle which I have employed, and which entirely fulfils these conditions, is the reduction of a ferric salt and subsequent titration of the quantity of ferrous salt produced.

I have found that when solid ferric sulphate is added to zinc dust suspended in a little cold water with exclusion of free acid, a reaction occurs with evolution of heat, and the zinc quickly and totally dissolves with formation of a clear greenish solution. A small residue remains consisting of lead and other impurities. The solution of the zinc takes place without any evolution of hydrogen, and the reaction is therefore represented by the equation—

$$Fe_2(SO_4)_3 + Zn = ZnSO_4 + 2FeSO_4$$

When all has dissolved, it is only necessary to acidify the solution with sulphuric acid and titrate with permanganate to find the quantity of ferrous salt obtained, and hence the quantity of metallic zinc in the sample under examination.

Procedure.—(*a.*) *Preparation of Pure Ferric Sulphate.*—500 grms. of pure ferrous sulphate are dissolved in as little water as possible, and to it are added 100 grms. of sulphuric acid and 210 grms. of nitric acid (60 per cent.). On adding the nitric acid, torrents of nitrous gas are evolved, the solution acquiring a nearly black colour, which disappears again when the whole of the acid is added. The solution is evaporated on the water-bath until it becomes solid, when it is ground with alcohol in a mortar, put on a filter, and washed with alcohol until the filtrate is no longer acid. The product is then dried thoroughly on the water-bath, and the salt, which is a perfectly white powder, is kept in stoppered bottles for use.

(*b.*) *Analysis.*—About ½ grm. of zinc dust is put into a stoppered 250 c.c. flask and to it are added 25 c.c. of cold water. The mixture is agitated, and when the zinc is thoroughly suspended, 7 grms. of ferric sulphate are added. There is a gentle evolution of heat, and after shaking for a quarter of an hour, the zinc will have completely dissolved, with exception of a slight residue of impurities. 25 c.c. of strong sulphuric acid are then added, and the flask is filled with water to 250 c.c. 50 c.c. of this solution, after dilution with 50 c.c. of water, are titrated with standard permanganate.

From the quantity of the latter employed, the percentage of metallic zinc is at once found.

The new method gives results which agree with those obtained by Drewson's method, *viz.*, reduction of chromic acid. Thus for a certain sample of commercial zinc dust the following numbers were obtained:—

——	New Method.	Drewson's Method.
I.	85·5	84·9
II.	85·4	84·5
III.	85·5	..

The new method has, however, the advantage of being extremely simple and rapid; it only requires a standard solution of permanganate, a reagent found in every commercial laboratory, and lastly, since the end point is indicated by the pink colour of the permanganate, the titration is sharper than in the method of Drewson, in which a spotting test is employed. To obtain accurate results it should be borne in mind that the ferric sulphate, if not well dried, may sometimes retain a trace of alcohol, which would reduce a little permanganate. It is therefore better to perform a blank experiment with the same quantity of ferric sulphate as used in the analyses and deduct the amount of permanganate reduced from that found.

DISCUSSION.

Dr. GROSSMANN said that the method described in the paper might be more convenient than the method of titrating with bichromate as generally used, but it suffered from the same defects as the chromate and other methods. First of all, they did not consider the fineness of the zinc powder, which had a great deal to do with its commercial value. Secondly, they did not show any quantity of iron present as such. There was nothing to prevent anyone from grinding iron filings up finely and mixing them with zinc dust, and none of these methods would show that. This method, as well as the older ones, therefore, would open a field for adulteration.

Dr. LEWKOWITSCH said he had used this method some 16 years ago in an analytical laboratory, but he did not publish it as he thought it was not new.

Mr. BARNES (Borough Analyst, Accrington) said the writer of the paper described a method which he (Mr. Barnes) published about 11 years ago* as a rather tedious process. He did not find it so, as several tests could be made at once by having a number of graduated tubes, in which the

* This Journal, 1886, 145.

hydrogen was collected, and by using one with a standard zinc all corrections for temperature and pressure were avoided. It was possible that iron might be measured as zinc in this process, as in titration by permanganate, but he did not think that was of much consequence, because metallic iron could be determined in a separate test by permanganate, or might even be removed by the magnet.

Mr. Harry Grimshaw said, from his own experience, he was of opinion that Mr. Barnes's method was decidedly quicker than the one set forth in the paper. He did not think any chemical method could very well give the difference between the respective fineness, which, of course, was an item of commercial value. A few years ago he had tried a kind of ground zinc, but found it was not suitable for work solely on account of not being in a sufficiently fine state of division.

Mr. Green, in replying on behalf of Mr. Wahl, said that all the methods of estimation of zinc dust at present in use were open to the objection of showing iron present as if it were zinc, but he did not consider this of much importance. A large quantity of iron was not of common occurrence, and if present could readily be estimated. No process could be expected to give satisfactory results with a zinc dust containing coarse lumps, and such a product would be useless for the purposes for which zinc dust was employed. The method of evolution and measurement of hydrogen employed by Mr. Barnes, besides being somewhat troublesome, was open to the objection that the lead was shown as zinc, whereas in Mr. Wahl's method lead remains undissolved. What was required in a commercial method of estimation was a process, readily and quickly performed, which would accurately indicate the reducing power of a given sample of zinc dust when used in alkaline or neutral solution; and Mr. Wahl's method fulfilled these conditions.

ON THE PROPOSED NEW SCHEME FOR DEALING WITH THE MANCHESTER SEWAGE.

BY WILLIAM THOMSON, F.R.S. ED., F.I.C.

The treatment of town sewage is at the present moment a question of supreme importance to the people of Manchester, and I have undertaken to commence a discussion upon the definite scheme which has now all but received the sanction of the Corporation.

The scheme is this: that the Manchester sewage be purified by precipitation, and that the effluent be conveyed by a tunnel some 10 miles in length to Latchford, and there poured into the River Irwell, *i.e.*, the Ship Canal. The estimated cost of this conduit is somewhere about 200,000*l.*, and it must not be forgotten that, if the experience gained in the construction of the Manchester Ship Canal goes for anything, it is probable that a great increase on the estimated expenditure may be anticipated before the work is completed.

To one who has not followed the arguments put before the City Council the scheme appears an extraordinary one. The river has been receiving up to the present time a large portion of the untreated sewage of Manchester and other townships on its banks. Now Manchester has resolved to purify all its sewage by precipitation, which will give a tolerably pure effluent. The further purification of this effluent will, however, entail an enormous cost on the inhabitants, and it is suggested that a conduit be constructed to run this purified sewage into the river 10 miles below Manchester. This looks as if the sewage, after being greatly purified by precipitation, were more objectionable than the raw sewage. This development of the sewage question is consequent upon the Joint Committee refusing to be satisfied in the first instance with an effluent produced from the sewage by precipitation alone, although, so far as they know, it may prove satisfactory as regards freedom from sediment and putrid odours during warm weather.

It has hitherto been considered by the supremely practical people of Manchester that great and costly undertakings should be carried out by processes of natural development, and that no step be taken which is not absolutely necessary; but in this scheme we are asked to make two steps at once, each of them involving large expenditure. The Corporation has given its provisional vote for both steps to be taken at the same time, and one may assume therefore that a strong case has been put before it. We must therefore look to the origin of the scheme to find how it came to be presented, because there can be no doubt that, from the premises set forth, the conclusions at which the Corporation arrived were correct.

Sir Henry Roscoe propounded the statement, which has been taken by the Mersey and Irwell Joint Committee, that it is impossible to obtain a satisfactory effluent from the Manchester sewage by precipitating the suspended matter and a large quantity of the soluble organic matter from it by means of sulphate of iron and lime; and with a view of making it satisfactory he advised the further treatment of the effluent by one of two methods: (*a*) By pouring it into filters composed of ashes, coke breeze, or sand, and then leaving it for a time sufficient to allow bacteria which were there cultivated to eat up or further oxidise a great portion of the organic matters left in solution; or (*b*) that the effluent (obtained by precipitation) should be filtered through land.

At this stage it came under the consideration of the City Surveyor, who treated the question from this point of view in a extremely able and practical manner. He calculated the cost of cinder or other similar filters and the working of them, and took into account their liability to become choked and the probable cost of cleansing and repairing for the one scheme; and he calculated the area of land required for filtration, and the cost of working, &c.; and after taking into consideration the liability of the land to become inoperative, the difficulty of finding land, and the cost of pumping which would be required for some of the land which was available, he finally himself propounded a third scheme, which would be much less costly than either of the others, *viz.*, to construct a conduit to take the whole effluent into the river 10 miles below Manchester.

Assuming one of the three above-mentioned alternatives to be absolutely necessary, the last scheme is undoubtedly the best, if the Mersey and Irwell Joint Committee will be satisfied with it; but, so far as I can understand, it is not by any means certain that they will; and it has power to prevent pollution at this point as well as at any other on the stream or canal.

The crux of the whole matter lies in the premise which led to the serious consideration by the Council of these three schemes, *viz.*, that the sewage, after being treated by precipitation and simple filtration, is unfit to pass into the River Irwell or the Ship Canal.

Now from a common-sense standpoint in deciding what kind of effluent is satisfactory for passing into any river, the nature of the river must be considered. The River Irwell ceased to be a fishing stream probably 50 years ago, and since then it has practically become an open sewer for Manchester and Salford, and the townships higher up on its banks. It must be conceded that to reconvert the Irwell into a fishing stream would be to make those who live near it expend probably 100*l.* for every penny which could be obtained through the value of the fish which could be taken from it, and I think no one would seriously argue that this should be done. Then for what reason are we called upon to purify the river? It cannot be shown that those who live along its banks suffer from zymotic diseases to a greater extent than those who don't. The answer to this question is obvious: the river must be purified to the extent, first, that it will not become silted up to any extent, and secondly, that it will not be a nuisance to those who live near it or who are in boats sailing upon it. In my opinion both of these reasons may be taken as one, because the putrid odour which emanates from the canal in warm weather comes chiefly from the fermentation, decay, or decomposition of the organic matters of the raw sewage which settles to the bottom.

The problem should therefore be this: Is it possible to treat sewage simply by precipitation, or by precipitation and simple filtration, so as to prevent it from throwing down a sediment capable of putrefaction, and so that the organic matters left in it will not themselves emit a putrid or objectionable odour on being kept for, say, two or three weeks in warm weather, or, more accurately, for such

time as the water remains stagnant in the Ship Canal? I believe it is possible to do this by one or other of several precipitation processes, and I think this should be practically tried during one or more years before Manchester is called upon to expend immense sums of money for the further treatment of its sewage. This, however, is not the criterion of the Mersey and Irwell Joint Committee. They have taken the working chemical standard laid down by Sir Henry Roscoe more seriously, I think, than he intended, *viz.*:—

(1.) That the effluent should not contain more than $\frac{1}{10}$ths of a grain per gallon of suspended organic matter;

(2.) That it should not yield more than 0·1 grain of albuminoid ammonia per gallon; and

(3.) That it should not require more than 1·0 grain per gallon of oxygen derived from potassium permanganate to oxidise the matters contained in solution.

It might be argued that town sewage effluent containing more than the quantities suggested by the Joint Committee would decompose and emit objectionable odours; but the sewage of different towns differ from each other very materially, and I have seen no record of any test made with any effluents obtained from Manchester sewage by precipitation, which proved that none of them would keep for two or three weeks in warm weather without emitting a putrid or objectionable smell. On the contrary, I have myself kept samples of effluents obtained from the sewage of both Manchester and Salford for many weeks without their emitting any putrid or unpleasant odour. Here is a sample produced by one precipitation process from Manchester sewage which I have kept in a warm room for two months, and it has remained during that time free from objectionable smell; and I think Mr. Carter Bell will bear me out as regards the keeping qualities of most of the effluents produced by the precipitation processes worked in connection with the Salford sewage. I have some samples of the effluents obtained by perhaps the simplest of all, *viz.*, the addition of about 16 grains of lime per gallon of sewage; this gives a copious precipitation. The precipitate settles rapidly, and the effluent, without filtration, was comparatively clear. The effect of the lime on the effluent was to produce a faint characteristic smell, not unpleasant, resembling to some extent herringbrine, but this smell did not increase nor alter during many months, and the effluent on being kept warm did not undergo decomposition. It has frequently been stated that effluents from the lime process of purification do undergo putrefaction, but I think this takes place when a much smaller quantity of lime is employed. It has been suggested that although the effluent may not emit objectionable odours when kept by itself, that it will putrefy when mixed with the waters of the Ship Canal; but it is difficult to understand this statement, because the waters of the Ship Canal will emit a putrid odour, whether or not you mix it with a sewage effluent, if samples of it be kept in a warm place for some time.

If we assume that effluents produced by precipitation are capable of entering into decomposition and evolving putrid odours when kept in a bottle or vessel in a warm room for some days, weeks, or months, it does not follow that if the same effluent were poured into the Ship Canal that this putrid decomposition would go on under those conditions. The point upon which I wish to be specially emphatic is this: that part of the scheme which is now before the Corporation is to purify the waters of the Ship Canal in the best possible manner by chemical means, *i.e.*, by precipitation and subsidence or filtration; and it seems extraordinary that an experiment should not be made to observe whether the smell during warm weather is not greatly reduced or entirely removed, and thus determine directly by that means whether such an effluent is or is not fit for passing into the Ship Canal before expending further large sums of money.

The result must certainly be a distinct improvement, and it can certainly not be worse than it was previously.

It has been suggested that Manchester is bound to produce a purer effluent than that which can be done by precipitation, as an example to manufacturers and smaller townships up the stream. Let us look at the nature of the example which Manchester proposes to set.

The Act of Parliament says that "every person who causes to fall or flow into any stream solid or liquid sewage shall be deemed to have committed an offence under the Act," but it goes on to say the person causing the sewage so to fall or flow shall *not* be deemed to have committed an offence against this Act if he shows to the satisfaction of the court having cognisance of the case that he is using the best practicable and available means of rendering harmless the sewage matter so falling or flowing into the stream.

The Manchester Corporation might improve the effluent obtained from precipitation by means of filtration through coke-breeze or cinders, or by the process which was regarded by the Joint Committee as the best of all, *viz.*, by filtration through land. The latter would involve, according to the City Surveyor's calculations, the purchase of about a ninth part of the whole area of Manchester for the purpose of treating the 26,000,000 gallons of effluent with which it has to deal. If the proposed scheme be carried out, the example which will be shown to manufacturers and to the smaller townships higher up the stream is that they have abandoned the idea of further treating their effluent as impracticable, and as an alternative have carried it by means of a conduit nearer to the tidal waters of the river. If this example means anything it means that the best thing which the manufacturers and townships higher up the stream can do is to follow it and to make for themselves other conduits for the same purpose. This method, however, can hardly be called a practicable one, and I am afraid that if all the manufacturers and townships up the stream were to follow this example there would be very little water left for the Ship Canal, and it is probable that the owners of the Ship Canal would much prefer to be supplied with the crude sewage than have the effluent removed by conduits in the manner proposed by Manchester.

It seems to me in carrying out such Acts of Parliament that the method employed by the late Dr. Angus Smith under the Alkali Act is the one which should be employed by the Joint Committee for dealing with the river, *i.e.*, to allow the purification methods to develop slowly. We know that the treatment of sewage with iron or alumina and lime or some similar process very materially improves the quality of the effluent. Why should the authorities not insist upon all those on the watershed employing an efficient precipitation process? If the whole of the water passed into the Ship Canal reached this standard of purification, I think a most remarkable diminution would be observed in the putrid odours evolved during warm weather. Experiments might be carried on by different manufacturers on the watershed, or by the chemists and engineers connected with the Mersey and Irwell Joint Committee, or by the Corporations of Manchester and Salford, to find a better method of dealing with the question.

The matter stands at present in a most unsatisfactory position, because the Joint Committee are not satisfied with precipitation processes, and their dissatisfaction is, I believe, based chiefly upon laboratory experiments. It must be taken into account that the greater the pressure put upon manufacturers to purify their sewage, the greater will be the loss to Manchester, and it seems obvious that such pressure should be applied in the most careful manner possible.

I understand that manufacturers and the smaller townships have been required to treat their effluents by land filtration, and have had to buy land at exhorbitant prices for the purpose of complying with the standard. It appears to me that, if such conditions are to be made, Acts of Parliament should be framed with a view to compel landowners to sell the necessary land at something like agricultural or twice agricultural value.

Discussion.

Dr. Pankhurst expressed surprise that the Manchester Corporation had not taken the advice of chemical experts on the subject of the sewage effluent, and contended that

had they done so they would never have been placed in the difficult position in which they now found themselves. It was to be deplored that when a great public body was embarking upon a large expenditure it did not supply itself in the first instance with scientific aid. He was satisfied that if crude sewage were prevented from entering the Ship Canal and a good system of chemical precipitation were to be applied in every instance and worked with reasonable efficiency, the practical difficulty would be absolutely removed, and a steady course of improvement set in motion. If that were so, what a terrible thing it was to put upon local bodies the enormous burden of purchasing land for filtration purposes. Mr. Thomson had focussed for them the conditions which would solve the difficulty. Through this and similar societies, and by public opinion, pressure should be brought to bear upon the Joint Committee, upon the Local Government Board, and the Manchester Corporation and other local bodies to adopt the proposed solution.

Dr. D. B. Hewitt (speaking as a member of the Mersey and Irwell Joint Committee) said, that while he fully appreciated Mr. Thomson's desire to deal fairly with the question before them, he thought he had dealt rather severely with the Joint Committee, in so far that he had made a statement which he (Dr. Hewitt) would be able to show, upon authority, had no foundation whatever. He held in his hand a report presented at a recent meeting of the Joint Committee, which contained the following:—

"As there seems to be some misapprehension with regard to the principles upon which the Mersey and Irwell Joint Committee are acting and their methods of applying those principles, the Sub-Committee consider it advisable to make a short statement.

"First, it is desirable to call attention to the fact that no definite standard of purity has been adopted, and the members of the Joint Committee, on reference to the proceedings, will find that so far back as December 1891, on the recommendation of Sir Henry Roscoe, a resolution was passed stating that it was undesirable to define the maximum amount of pollution to be allowed, but to insist upon the best practicable and available means being used in each case, whether a sanitary authority or a manufactory."

Therefore he took it that the Joint Committee were certainly in accord with at least one portion of Mr. Thomson's paper, but while that one sentence stood correct, there were other sentences in which he attempted to prove that a definite standard had been adopted, and it was in order that he might correct that statement that he had read the extract from the report. He had no doubt that the samples which Mr. Thomson had handed round were fair, but he (Dr. Hewitt) had seen samples laid before the Joint Committee which did not agree with the results arrived at by Mr. Thomson. He had seen hundreds of samples of sewage treated by land irrigation, precipitation, and filtration, and precipitation only, and therefore he was perfectly satisfied that it was quite possible for any local authority to produce a very good effluent by various methods of precipitation together with filtration that would not undergo secondary decomposition. The Joint Committee had, nevertheless, not yet met with an effluent that would not undergo secondary decomposition, and give off bad odours after being kept for a time. The Joint Committee were trying to induce people to adopt satisfactory methods. They had not adopted hard and fast lines. They knew there were places in the watershed producing satisfactory effluents, which if not carefully watched were apt to go wrong. With respect to Mr. Thomson's views on the purchase of land for filtration, he thought it was a great hardship that large communities should have to pay enormous prices for land under compulsion by Act of Parliament in order to protect their neighbours from river pollution. The whole object of the Joint Committee was to prevent this pollution by a similar method to that established by Dr. Angus Smith in connection with the Noxious Vapours Acts and approved by Sir Henry Roscoe. This mode of procedure was one in which the Committee had great hope for a solution of the problem. If the Corporation could make a satisfactory effluent, they could make it just as well at Davyhulme as they could at Thelwall, and if they could not, then the natural deduction was that they wished to evade the law by the construction of a tunnel at an expenditure of 200,000*l*. From the latest information at his disposal he did not think the Corporation intended to turn the effluent into tidal waters, but above them, in which case they would be well within the jurisdiction of the Joint Committee.

Mr. Harry Grimshaw said that when the Joint Committee was first instituted and got to work, it commanded their entire approval and admiration, and it certainly raised their hopes to a very high point; but now their hopes were dashed, and he feared their chemical confidence was somewhat shaken. He was of opinion that the Committee had made the mistake of forcing people to run before they were able to walk, and before ascertaining the nature of the ground on which they were asked to walk or run. With regard to the health question, neither Mr. Thomson nor himself would attempt to argue that the pollution of streams had no effect upon health, but river pollution was not mainly responsible for a heavy death rate. In many cases, taking Manchester for instance, the idea had been exploded that the areas showing the highest death rate lay near these sewage-laden streams. The next point was that all they knew at the present time, gathered from laboratory experiments and from general observation and accumulation of facts, with regard to the condition of the sewers, tended to prove that most of the offensive odour really originated primarily in the solid matter contained in the sewage and river water; and he though that he would not be interpreting chemical opinion wrongly if he asserted that the removal of the suspended matters would render them comparatively innocuous. Dr. Hewitt's arguments practically admitted the fact that the offence which arose from the Ship Canal could not be laid at the door of the sewage effluents which passed into it, so long as there was such a large amount of actual raw sewage from many places continually passing into the canal. He should like to suggest through Dr. Hewitt that the Joint Committee should really make a good study of what had been done in Glasgow by a system of precipitation, which appeared to produce an effluent which could not be charged with so-called secondary decomposition. There was a great danger of harassing the trades and industries of this district, of which they were the centre, and the Joint Committee could not be too careful in making progress step by step rather than endeavouring to do too much by leaps and bounds. It was said that before long the question of the Manchester sewage scheme would come before the highest court in the land. He was inclined (though it might be considered somewhat egotistical) to think that the Society of Chemical Industry was the highest court before which it could be brought.

Mr. J. Carter Bell was surprised that Dr. Hewitt admitted that the Joint Committee had no definite standard. A number of experiments had been carried out by various companies at the Salford Sewage Works, but they could not get a definite answer from the Joint Committee as to whether the results were good enough or not. The reason why lime did not answer in many cases was because sufficient was not used. Ten to fifteen grains per gallon of free lime were necessary. The drawback was the volume of sludge, and he was experimenting with another precipitant in the hope of reducing the bulk of the sludge. He preferred to use lime water, but the engineering difficulties in the way were apparently insurmountable, as they would want 200,000,000 galls. of it per day at Salford. He still had faith in lime as a good precipitant, and contended that, with a sufficient quantity in the state of lime water, he could produce an effluent that would satisfy the Ship Canal Company and the Joint Committee.

Mr. A. E. Powell asked Dr. Hewitt if he was aware that the horrible stench about which he complained was caused by the Ship Canal Company filling the old bed of the river at Mode Wheel with the abominable sludge which they dredged from the bottom of the canal. This was worst between Salford Docks and Barton Locks, and no sanitary authority had yet interfered with them, though they had compelled corporations to adopt sanitary regulations and carry the sewage out to sea.

The CHAIRMAN said he believed the Local Government Board were mainly responsible for the present deadlock in regard to the sewage question. At the time of the Local Government Board enquiry in Manchester in 1888 it was pointed out that the area of land applied for was far too small for filtration purposes, so that the Manchester scheme ought never to have been sanctioned. The term "secondary decomposition" had been introduced in the course of the discussion, and as he had never been able to understand how this condition was usually ascertained, perhaps someone would be able to inform the meeting on this point; one thing, however, was certain—that the samples submitted by prosecutors in the law courts generally were sealed up in bottles and stank badly, while others, kept in open bottles by the defendants, were clear and sweet; and it should be remembered that the Irwell river is not covered in during its flow, but was ever free to the atmosphere. In this way he thought the prosecutions conducted on behalf of the Joint Committee were tainted a little bit too much with the desire to win their case by any means in their power. He would like to add that when the Mersey and Irwell Bill was before Parliament, a clause should have been inserted similar to that in the Alkali Act, which forbids inspectors acting as feed advisers to those they were called upon to inspect; and the sooner that some amendment of the Joint Committee's Act was made in this direction the better.

Dr. HEWITT, in reply to the Chairman, assured the meeting that the greatest pressure had been brought upon the Joint Committee by manufacturers and authorities of the local districts for permission to allow their adviser to give as much information as possible; and the reason the Committee had placed the information obtained by Sir Henry Roscoe at the service of the public was simply with the view of bringing about a change for the better as soon as possible. Whatever the members of the Society might think about this, it had been done for the benefit and at the request of the public.

The CHAIRMAN: The explanation given by Dr. Hewitt might be satisfactory to the Committee themselves, but it was not satisfactory to outsiders. The alkali inspectors were often asked to give advice to manufacturers whose works they inspected, but they were not allowed to take fees for doing so.

Mr. THOMSON, in reply, said he was quite aware, as Dr. Hewitt had admitted, that the Joint Committee claimed to have no definite standard, and the Rivers Pollution Act recognised no standard. At the same time, they had a working standard which had been expressly stated as equivalent to 1 grain of oxygen from permanganate per gallon and 0·1 grain of albuminoid ammonia. They did not take samples and ascertain whether they would undergo "secondary decomposition" (he should call it primary decomposition). He had seen many of Mr. Carter Bell's effluents from various processes, some of which had been corked up and others kept open, but very few of them had any objectionable smell. One third of the Manchester sewage received no treatment, and the whole of that filth was sent into the river, there to undergo decomposition. Was it not better to take all the obnoxious material from it, and, instead of trying to get certain places to produce the highest possible standard of effluent, purify the sewage by precipitation and send the sludge to sea by steamship, and then pass the effluent into the river or canal at Davyhulme? The point before the public was whether it was desirable to spend 200,000*l.* in the construction of a conduit to run this effluent to a point lower down the Canal. They were all agreed upon one point, however — that the sewage must be purified to such an extent that the effluent would not cause bad odours to be emitted from the Canal, and the question was, How could this be achieved? He was not certain that the Joint Committee would not interfere even then, as the river at Latchford was also within their jurisdiction. He had recently examined some samples of water from the River Irk, and he found some 50 grains per gallon of solid matter in suspension, whereas in cases in which he had been engaged 2 or 3 grains were considered highly objectionable. He thought it very important that the Committee should not insist upon such high standards of purification till all the refuse waters and sewage had been treated by precipitation.

Newcastle Section.

Chairman: F. S. Newall.
Vice-Chairman: G. T. France.
Committee:

A. Allhusen.	W. Martyn.
P. P. Bedson.	John Morrison.
T. W. Hogg.	John Pattinson.
H. Louis.	W. W. Proctor.
T. W. Lovibond.	W. L. Rennoldson.
N. H. Martin.	T. W. Stuart.

Hon. Local Secretary and Treasurer:
Saville Shaw, Durham College of Science, Newcastle-on-Tyne.

SESSION 1896-97.
Dates of Meetings.—Feb. 25th, March 25th, April 29th.

Meeting held at the Durham College of Science, Newcastle-on-Tyne, Thursday, December 17th, 1896.

MR. F. S. NEWALL IN THE CHAIR.

ON SOME POINTS CONNECTED WITH TURNIP MANURING.

BY WILLIAM SOMERVILLE, D.SC., AND R. GREIG SMITH, M.SC.

WHEN a farmer manures his fields he expects that the most, if not all, of the costly constituents which he applies will be returned to him in the increased crop. He may manure for a particular crop or for a series of crops; in the former case artificial fertilisers are chiefly employed, and in the latter farmyard manure. Very frequently both ideas are carried out together, and dung with fertilisers is applied for the root crop, which is considered the beginning of the rotation. A root crop is necessary, in order to allow the land to be cleaned. To grow turnips well, a manure containing phosphate is required, and since this constituent can only be appropriated by plants when it is present in an easily available form, manure containing soluble or easily available phosphate must be used. It is found that increasing quantities of this manure give an increase of crop, but this increase does not correspond to the amount of manure used, and consequently a very pertinent question arises: "What becomes of the excess?" Is it taken up by the plant to produce a richer bulb? Is it in the top? Is it washed into the drains or the subsoil or does it remain in the soil? Where the excess remains in the soil it will doubtless influence the growth of the succeeding crops of the rotation, but it will gradually, as time goes on, become more and more insoluble, changing to the condition of the phosphates which occur naturally in soils. It appears to become converted into the phosphates of iron and alumina, which undergo dehydration, and become akin, so far as solubility is concerned, to crystalline or ignited phosphate, and as such it is practically lost to the farmer. Should, however, the phosphate be taken up by the plant, either in the bulbs or in the tops, it is returned to the land again as dung and as green manure, and is in this way prevented from undergoing degradation.

Of no less importance is the nitrogen, which, when applied in soluble forms, will, unless absorbed by the crop, become lost to the soil, since it is only fixed by the growing plant.

In order to throw some light upon the destination of these two constituents, their amounts were estimated in the bulbs and tops of turnips manured in different ways, during 1895. The samples were received from eight experimental stations, four of which had been manured in one way and four in

another. One set received dung along with artificial fertilisers, while the other received artificial fertilisers only.

On the field five roots were selected as average or typical specimens from each plot; these were put up into bags and, together with the tops, were sent to the laboratory of the Durham College of Science for analysis; but as the tops would have lost too much water in transit, special samples for moisture determination were taken from three stations, two of which belonged to the artificially manured series, and one to the dung set. For these samples five average tops were chosen, and each was quartered, care being taken that the dividing was uniformly done, so far as the old and the young leaves and the neck of the turnip were concerned. The quarters were put into tight tin boxes, and despatched to the laboratory, where they were cut up and tested for moisture. The turnips, on arrival, were washed and dried, the tops and tails pared, and the bulbs cut into vertical segments like orange liths. One segment from each turnip was many times cut transversely, by means of a machine bread knife, to within an eighth of an inch of one of the vertical sides, a proceeding which caused the portion, as it dried, to spread out fanwise, and so expose a maximum amount of surface. These were threaded upon thin wire, and hung over tin boxes similar to those used for baking bread. The samples were at first dried slowly in the air, then, after a time, were put into a warm cupboard, and when all danger of "stewing" was past, they were dried in the steam-bath until they became sufficiently brittle to grind. For the moisture determinations, tapering wedge-shaped portions were cut from the centre of a section of each root. These were sliced, after the manner of the larger portions, weighed, and dried, at first slowly, then for 24 hours at 103° C. In the powdered portions of the large samples, the moisture was estimated by exposure to 100° C. in the air-bath for three hours.

The well-known Kjeldahl method was used for the estimation of the nitrogen, and the phosphoric acid was determined in the ash by precipitation with magnesium chloride in the presence of citrate and ammonia. In the case of the tops, the silica was eliminated previous to precipitation, but this preparation was unnecessary with the roots.

The greatest care was observed that every determination of each constituent was performed in precisely the same way, so that the results could be compared one with the other.

In the set getting artificial manure, swedes were sent from two of the farms and yellow turnips from the others; with the lot receiving dung and artificial fertilisers, three farms sent swedes and one yellows. The names of the farmers and the kind of turnip grown in each trial are given below. Further details as to the trials will be found in the *Fourth Annual Report on Manurial Trials (Dr. Somerville), Season 1895.*

Artificial Fertiliser Set.

Mr. T. Newby,	Muncaster Head,	Cumberland	Yellows.
Mr. T. B. Maughan,	Middlefield,	Durham	„
Mr. Thompson,	Shittlehope Burn,	„	Swedes.
Mr. J. T. Armstrong,	Hardriding,*	Northumberland.	„

Farmyard Manure and Artificial Fertiliser Set.

Mr. J. Wallace,	Skelton Pastures,	Cumberland..........	Yellows.
Mr. G. Liddell,	Kimblesworth,	Durham	Swedes.
Lord Armstrong,	Cragside,	Northumberland	„
Mr. H. H. Scott,	Hipsburn,*	„	„

Manuring with Artificial Fertilisers alone.

It is noticed that when turnips are grown upon unmanured soil, the individual bulbs are smaller than those raised under more favourable circumstances, and as a consequence the yield is also smaller. The smaller roots are, however, firmer, and one is led to expect that this firmness means less water—that is more solids—an expectation which is realised by the percentage of solid matter in the bulbs of plot 1, being higher than in any other plot.

The addition of phosphoric acid and potash, however, or these with a small quantity (½ cwt.) of nitrate of soda, does not materially alter the amount of solids in the roots; but from the diminution in the tops, as will be emphasized later, mineral manures alone, or with the addition of small quantities of nitrate, appear to make the plants mature earlier.

As the nitrate is gradually increased no regular variation in the bulbs is obtained, those receiving the medium application (1 cwt.) have least dry matter. On the other hand, the tops show a steady decrease in solids with increase of nitrate of soda in the manure. This, however, only holds good to a certain extent. Very soon a limit is reached, and we see from plot 8, receiving 2 cwt. of nitrate, the solids in the tops are greater than on plot 7, getting 1½ cwt. The nitrate on both these plots was applied at the same time, *viz.*, half with the seed and half at thinning.

As the application of nitrate is delayed, the period of growth is prolonged, a state of things indicated, not only by the luxuriant appearance of the tops when the turnips were lifted, but also by the analysis. Plots 5, 7, and 6 each received a phospho-potassic manure with 1½ cwt. of nitrate of soda, the latter being sown with the seed on plot 5, spread broadcast at singling time on plot 6, while on plot 7 it was applied half the one way and half the other. The later sowing has produced a less percentage of solids in the bulbs and a greater percentage in the tops than the earlier, while the half-and-half mode of application of plot 7 lies between the two. Evidently the leaf cells of the plants are actively manufacturing organic matter, and as the plant matures, or as the stimulating effect of the nitrate declines, the percentage of solids in the leaf falls whilst that of the bulb increases.

The action of phosphoric acid in forcing the crop is shown by plot 10, which received dissolved bones in addition to nitrate, kainit, and superphosphate. There is about as much total nitrogen in this manuring as in the case of plot 4, so that these two may be compared and the action of the extra phosphate observed, but a general survey of all the analyses shows that in the dissolved bone plot the solids of the bulbs are at a maximum and of the tops at a minimum, which can only be accounted for by the extra phosphate.

Potash appears to be necessary for quick ripening, and in this respect plays much the same part as phosphoric acid. The incomplete manure (plot 9) has less solids in the bulbs and more in the tops than plot 10; evidently the lack of potash causes the solids to remain largely in the tops when the crop is pulled.

It is a general rule that when any single manurial constituent is sown, the amount in the crop is increased; and phosphate shows itself to be no exception, as both bulbs and tops hold increased percentages of phosphoric acid through the action of the superphosphate. The absence of nitrogen in the manure of plot 2 has been reflected in the contents of the bulbs. Most of the plots in this experiment have had the same quantity of minerals, with varying amounts of nitrate, or with the same quantity of nitrate applied at different times, and on that account it is with the nitrogen that differences are to be expected.

Nitrate in small quantity, *viz.*, ½ cwt. per acre, does not alter the quantity of nitrogen in the bulb, but when given in larger doses, a considerable increase in nitrogen is obtained. Speaking broadly, there does not appear to be a constant rise in percentage of nitrogen with a continual increase of nitrogenous manure; either nothing happens, or the percentage is raised to what may be considered the normal amount, since it is the same as is found in the unmanured crop. But a closer examination of the figures shows that the percentage varies slightly with the manure, there being a rise in the case of the bulb nitrogen and a

* Tops from these farms were examined for moisture. Another set of samples of tops was received from Mr. R. H. Cleasby, Broomside, Durham, on whose farm an experiment of the artificial fertiliser set was carried out, and moisture averages were struck between these and the tops from Mr. J. T. Armstrong.

Table I.

Plot.	Manuring per Acre.				Percentage of Constituents in						Plot.
					Fresh Roots.			Fresh Tops.			
	Superphosphate.	Nitrate of Soda.	Kainit.	Dissolved Bones.	Solids.	Nitrogen.	Phosphoric Acid.	Solids.	Nitrogen.	Phosphoric Acid.	
	Cwt.	Cwt.	Cwt.	Cwt.							
1	0	0	0	0	9·34	0·110	0·0510	15·26	0·479	0·089	1
2	5	0	2	0	9·29	0·115	0·0687	14·88	0·497	0·111	2
3	5	½*	2	0	9·29	0·114	0·0640	14·76	0·477	0·106	3
4	5	1 *	2	0	9·00	0·143	0·0635	14·63	0·468	0·104	4
5	5	1½*	2	0	9·18	0·119	0·0648	14·33	0·461	0·104	5
6	5	1½†	2	0	8·70	0·128	0·0614	16·81	0·627	0·140	6
7	5	1½‡	2	0	8·93	0·145	0·0566	14·78	0·524	0·117	7
8	5	2 ‡	2	0	8·79	0·143	0·0573	16·20	0·570	0·130	8
9	5	½	0	2½	9·07	0·121	0·0624	15·89	0·492	0·129	9
10	5	½	2	2½	9·25	0·135	0·0597	14·13	0·428	0·109	10

* Nitrate all applied before sowing. † Nitrate all applied after thinning.
‡ Nitrate applied half before sowing and half after thinning.

constant fall with the nitrogen of the tops, from which it would appear that the more nitrate we apply at one time, the greater is the percentage of nitrogen found in the bulbs and the less in the leaves. This, however, only holds good to a limit expressed by the ratio of 5 cwt. of superphosphate to 1½ cwt. of nitrate of soda, for when the last manure is raised beyond this, as shown by plots 7 and 8, the bulb nitrogen is constant, while the top nitrogen is increased. The phosphoric acid is constant when the same weight of superphosphate is applied and when the nitrate is all put on at the same time, but when put on at different times more is found in the tops and less in the bulbs with the later manurings.

Dissolved bones have had practically no effect upon the phosphoric acid, probably because the plant has had as much as it required in the superphosphate; but the nitrogen has been raised in the bulbs and lowered in the tops, as is seen on comparing plot 10 with plot 3, both of which received the same amount of nitrate of soda.

Potash decreases the phosphoric acid all over the plant; and its action is very marked on the nitrogen, that in the bulb being increased and that in the top decreased.

Table II.

Plot.	Manuring per Acre.				Average Yield.						Solids per Acre.			Ratio of Roots to Tops as 100 to	Plot.
	Superphosphate.	Nitrate of Soda.	Kainit.	Dissolved Bones.	Roots.			Tops.			Roots.	Tops.	Total.		
	Cwt.	Cwt.	Cwt.	Cwt.	Tons	Cwt.	Qrs.	Tons.	Cwt.	Qrs.	Lb.	Lb.	Lb.		
1	0	0	0	0	10	6	1	2	3	3	2,157	748	2,905	34·7	1
2	5	0	2	0	14	18	2	3	2	2	3,105	1,042	4,147	33·5	2
3	5	½*	2	0	15	4	1	3	8	3	3,164	1,140	4,304	36·0	3
4	5	1*	2	0	15	15	1	3	8	2	3,179	1,123	4,302	35·3	4
5	5	1½*	2	0	17	6	1	4	0	1	3,560	1,288	4,848	36·2	5
6	5	1½†	2	0	17	5	3	4	6	3	3,369	1,633	5,002	48·5	6
7	5	1½‡	2	0	17	10	1	4	11	0	3,505	1,506	5,011	43·0	7
8	5	2‡	2	0	16	13	1	4	3	2	3,280	1,515	4,795	46·2	8
9	5	½	0	2½	14	6	0	3	10	1	2,906	1,250	4,156	43·0	9
10	5	½	2	2½	15	16	3	3	2	3	3,282	993	4,275	30·2	10

* Nitrate all applied before sowing. † Nitrate all applied after thinning.
‡ Nitrate applied half before sowing and half after thinning.

The amount of solid matter produced per acre in the bulbs should be of more importance than the weight of crop, since turnips are grown primarily for food. Closely connected with the question of quantity is that of quality, which is indicated by the nitrogen contained in the solids. Looking over Table II. we see that the roots receiving superphosphate, kainit, and 1½ cwt. of nitrate of soda contain most solids, and further, that, with this group, the best result is obtained when the nitrate has been all applied at sowing. With the early application of 1½ cwt. of nitrate, the plants are given a start which enables them to keep well ahead of those receiving less manure, or the same quantity applied at later periods. What the later sowings want in root solids they make up for in top solids, and as a consequence the total organic matter per acre is practically the same in all three cases.

The effect of the time of application is shown better here than in the total crop, which shows a difference of 4 cwt. of roots to the credit of the intermediate application, while the solids are actually lower by 55 lb. Assuming these roots to contain an average of 9 per cent. of solids, the 55 lb. of dry matter would be equivalent to 5½ cwt.; so that, while the weight of crop per acre of the roots receiving the nitrate half at sowing and half at singling is 4 cwt. more than when the nitrate is applied all at sowing, in reality, so far as the solids are concerned, the bulbs are 5½ cwt. less.

An increase of nitrate over the 1½ cwt. has depressed the solids very considerably. As a rule an increase of nitrate produces an increase of crop,* but with the four stations of the set the crops have been in three cases much diminished: with the diminution in the crop there has been no corresponding rise in the percentage of solids, and we are therefore compelled to conclude that with the applied amounts of superphosphate and kainit, 1½ cwt. of nitrate of soda is the optimum quantity.

* Manurial Trials, season 1895, page 70.

Just as 2 cwt. show over-manuring, quantities less than 1½ cwt. show under-manuring, and so long as the manure contains an insufficiency of nitrate it does not appear to matter much whether a whole or a half cwt. is applied. Again, this is at variance with conclusions regarding the action of nitrate based upon the weight of crop.

It would seem that the ratio of nitrogen to phosphate in a turnip manure should be as 1 : 5½, or one part of ammonia to five of phosphate when the manure contains potash and when no dung is applied.

Increasing the amount of phosphate by means of dissolved bones, which contributes combined phosphoric acid and nitrogen in many different conditions as regards solubility and availability, has increased the solids (compare plot 4), but perhaps its best point is that the extra phosphate has caused an earlier ripening.

When no farmyard manure is applied to the turnip break, potash should on no account be omitted, as has been demonstrated in the North of England field trials.* Theoretically potash is more important than phosphoric acid as a manurial constituent for the crop; but practically, owing to the turnip's facility for taking up this constituent from the soil, potash becomes of relatively less importance; still it should not be ignored if a good crop of bulbs is desired. A comparison of plots 9 and 10 shows that through the action of the kainit about 300 lb. of solid matter have been transferred from the top to the root.

Table III.

Plot.	Manurial Constituents applied: Lb. per Acre.		Crop Constituents in Lb. per Acre.										Plot.
			Phosphoric Acid.					Nitrogen.					
	Phosphoric Acid.§	Nitrogen.	Roots.	Tops.	Roots and Tops.	Roots and Tops, less Plot 1.	Remaining in Soil.	Roots.	Tops.	Roots and Tops.	Roots and Tops, less Plot 2.	− Loss or + Gain.	
1	0	0	11·8	4·3	16·1	..	..	32·4	23·5	55·9	..	..	1
2	75·2	0	23·0	7·7	30·7	14·6	60·6	38·4	34·8	73·2	..	..	2
3	75·2	8·9*	21·8	8·1	29·9	13·8	61·4	38·8	36·6	75·4	2·2	− 6·7	3
4	75·2	17·8*	22·4	8·0	30·4	14·3	60·9	50·5	35·8	86·3	13·1	− 4·7	4
5	75·2	26·7*	25·1	9·4	34·5	18·4	56·8	57·8	41·4	99·2	26·0	− 0·7	5
6	75·2	26·7†	23·8	13·6	37·4	21·3	53·9	49·6	60·9	110·5	37·3	+10·6	6
7	75·2	26·7‡	22·2	11·9	34·1	18·0	57·2	56·9	53·3	110·2	37·0	+10·3	7
8	75·2	35·6‡	21·4	12·1	33·5	17·4	57·8	53·3	53·3	106·6	33·4	− 2·2	8
9	121·8	16·5	20·0	10·1	30·1	14·0	107·8	38·8	38·7	77·5	4·3	−12·2	9
10	121·8	16·5	21·3	7·6	28·9	12·8	109·0	47·9	30·1	78·0	4·8	−11·7	10

* Applied before sowing. † Applied after thinning. ‡ Applied half before sowing and half after thinning.
§ Soluble phosphoric acid only of superphosphate taken.

The Destination of the Phosphate and the Nitrogen.

Having considered the solids, the nitrogen, and the phosphoric acid, there remains the very important question of the amount of the last two constituents abstracted by the plant from the soil. From this the quantity of manure remaining in the soil can be calculated. To effect this, the number of pounds per acre of the constituents in root and top are added together and, in the case of phosphoric acid, subtracted from the amount in plot 1; in the case of nitrogen, subtracted from that in plot 2. The unmanured plot must be credited with having taken up all the available phosphate. The plots manured with nitrate received also superphosphate and kainit, both of which have, as may be seen by comparing plots 1 and 2, enabled the plant to take up more nitrogen from the soil. That this could be done infers that in the unmanured plot the soil nitrogen was not deficient, and there was enough for the available phosphate. In the phosphoric acid question, therefore, the subtraction of plot 1 from the others is justified. A glance at the table shows that only about a quarter of the phosphate applied in the manure has been taken up by the crop; the rest remains in the soil. The nitrogen varies considerably according to the amount used and the time of application. In some cases what is applied is not abstracted by the crop, and in others there is much more. It will, however, be better to discuss these results in detail.

In the plots 2 up to 8, each received 5 cwt. of superphosphate containing 75·2 lb. of soluble phosphoric acid, while plots 9 and 10 got the same quantity of superphosphate, together with 2½ cwt. of dissolved bones, in all 121·8 lb. of phosphoric acid. Without nitrate, 14·6 lb. are absorbed by the plant from the manure, and 60·6 lb. remain in the soil, part of which will benefit the next and a few succeeding crops, while the rest will become, as shown in the Rothamsted experiments, an integral portion of the soil and will be taken up with extreme slowness.

With the application of ½ cwt. of nitrate, rather more (0·8 lb.) is left behind, but with the nitrate still increasing; less and less phosphoric acid remains in the soil. This is in all probability due to the nitrate increasing the crop, and with it the root fibres, which ramify throughout the soil and absorb the manure from the phosphate-coated particles. As the manuring with nitrate is deferred, more phosphate is taken up, since plots 5 and 7 are very similar and plot 6 much greater. Increasing the quantity of nitrate over 1½ cwt. rather prevents the assimilation of the phosphate. Dissolved bones have left all their phosphoric acid in the soil, and kainit has caused the crop to leave more than would have been the case had the manure been withheld.

On the whole, the phosphoric acid story is a very sad one; so much must be applied to grow a respectable crop, and at the same time the most of it is left in the soil to become so insoluble that succeeding crops can only take it up with difficulty. This, however, is precisely what has been found to be the case by the Rothamsted and other experimenters.

The nitrogen question is more interesting. As shown in the column "Roots and Tops, less plot 2," the application of 1½ cwt. of nitrate of soda has resulted in least loss. Of all the manurial constituents, nitrogen is the one which we desire to have in the crop; for, should it be left in the soil, excepting as root residue, we do not expect to find it there in the succeeding season.

Plots 3, 4, and 5 show that as the application of nitrate increases, the quantity left in the soil gets less and less. With 1½ cwt., the quantity left in the soil is practically *nil*; and when we consider that this plot grew the largest crop, we must conclude that the nitrate has been all saved, for much more than 0·7 lb. would be found in the root residue.

* Manurial Trials, season, 1895, page 73.

As the time of manuring is deferred, the nitrogen abstracted and found in the crop increases to such an extent that there is a very material gain of this constituent.

Plot.	Minerals with 1½ cwt. Nitrate of Soda.	Nitrogen.
6	Applied at sowing........	0·7 lb. left in soil.
7	Applied half at sowing, half at thinning.	10·3 lb. excess in plant.
8	Applied at thinning.......	10·6 lb. .. ..

From loss that must occur through drainage, one expects that a considerable portion of the nitrate applied in the early season will be lost to the soil and to the crop; but these results show a very small loss under conditions of rational manuring, that is to say, with a perfectly balanced manure. At first sight, the increase of nitrogen would seem to indicate what is not justified by our present knowledge, *viz.*, a fixation of nitrogen by the turnip crop; but it must be remembered that in all probability the nitrogen in the crop of plot 2, which we have assumed to be the soil nitrogen which has nitrified and been absorbed by the plant, is in reality only a portion of it, the rest having been lost by drainage. This loss is the less the greater the crop, and with the heavier and later doses of nitrate the loss has been minimised to such an extent as to appear a gain. Moreover, the later application of nitrate occurs at a time when the plant is in a more forward state; it is growing more freely, and is more capable of absorbing the manure. The crops of plots 6 and 7 were lifted along with the others at a time which, though correct for the plots as a whole, would have been too early for these two taken individually. They have evidently not reached maturity, and with a mild season they would have continued to grow and mature, with the result that the excess of nitrogen which is in the tops would in all probability have been transferred to the bulbs with undoubted advantage.

The application of 2 cwt. of nitrate has evidently disturbed the balance of the manurial constituents, and what was a gain (plot 7) has become an evident loss.

Kainit produced a material increase in the crop and in the solids, but it has practically no effect on the phosphates and the nitrogen withdrawn from the soil. The dissolved bone plot produced about the same crop as plot 4, both of which were manured with about the same quantity of nitrogen; it produced 12½ cwt. of roots more than plot 3, but has abstracted a little less phosphate and a little more nitrogen from the soil. All the phosphate and two-thirds of the nitrogen of the bones have practically been left in the soil.

Manuring with Dung and Artificial Fertilisers.

It is the usual agricultural custom to spread farmyard manure either over the stubble or along the drills previous to sowing the turnip seed, and most farmers declare that it is impossible to grow a crop of swedes—that is, a good one —without dung; consequently a "mucking" forms an essential feature of turnip manuring. On this account any information regarding the constituents taken up by the root crop manured in the customary way should be of some value. At the outset, however, the variable nature of farmyard manure must be considered. It is always applied in cartloads, the contents of which may weigh 12, 14, 16, or even 20 cwt. As a rule they are assumed to be 16 cwt. In composition, this natural manure is most variable, and depends upon a number of factors, such as the "ripeness" of the heap, the feeding of the stock, the kind of animal producing it, the method of storage and treatment, the kind and amount of litter used, and so on. Lawes and Gilbert, recognising the difficulty, not only of sampling the manure, but also of pitching upon any one heap as being typical, preferred to calculate rather than analyse its composition. Their result agrees closely with that obtained by the late Dr. Voelcker from manure sampled with the greatest care. The tabulated percentages of water-soluble and insoluble nitrogen and phosphoric acid are taken from Dr. Voelcker's figures, and the amounts in 16 loads are from them calculated. The nitrogen soluble in water,

	Composition of Dung.		In 16 Loads.	
	Soluble.	Insoluble.	Soluble.	Insoluble.
	Per Cent.	Per Cent.	Lb.	Lb.
Nitrogen	0·297	0·309	85	88
Phosphoric acid	0·175	0·274	50	78

and on that account readily available as plant food, is about equal to that contained in a little over 5 cwt. of nitrate of soda, while the soluble phosphoric acid in the 16 loads and in 3⅝ cwt. of superphosphate is about the same. The insoluble nitrogen and phosphoric acid will probably not contribute much towards the crop to which they are applied. A farmer would never think of manuring his turnips with these quantities of artificials, and yet long practice and experience has shown him that 16 loads of dung is only a medium dressing.

The scheme of the experiment, consisting of dung with different amounts of artificial fertilisers, may be gathered from Table IV.

The effect of dung alone, as seen by comparing plots 2 and 1, has shown itself chiefly in the production of solids, which are materially increased so far as percentage is concerned and nearly doubled in amount. An increase in the nitrogen content of the roots and tops ought to be expected from the quantity of this constituent in the dung, but the two plots give the same analysis showing that the manure has directed itself to increasing the crop and not to improving the quality. The phosphoric acid, however, is decidedly increased not only in the bulbs but also in the tops, a circumstance which probably indicates a more mature crop, and this opinion is endorsed by the lower ratio of the top solids to the roots.

The addition of varying amounts of nitrate tells practically nothing beyond the fact that 1 cwt. to 16 loads of dung has done least harm; the single cwt. has given a greater percentage and amount of solids than the lower and the higher quantity.

Manuring with increasing quantities of superphosphate causes a rise in the figures all round; the phosphoric acid, the nitrogen, and the solids both in bulb and top, and the amount of solids per acre gradually increase with the manure. One is prepared to expect good things of phosphate since the dung is so essentially nitrogenous and the regularity of increase in the percentages confirms the belief that there is enough nitrogen in the dung for almost any quantity of superphosphate and certainly for 7½ cwt. The ratio of tops to roots is very small, the energies of the plant under the action of the added fertilisers being apparently devoted to forming the bulb.

Kainit and dung together have a very disastrous effect; the percentage of solids and of nitrogen are much decreased. Potash has had no influence upon the phosphoric acid of the roots nor on the composition of the tops, but it has prevented, as shown by the lessened ratio, that luxuriant growth of leaf so characteristic of nitrogen. The weight of roots grown per acre was not increased by the potash, and since they were much more watery than those grown with dung alone, the solids came out much less.

Dissolved bones and kainit have done exceedingly well, especially with the smaller quantity of the former manure. The bulb constituents are high, and there are more solids than in any other plot. Doubling the quantity of the bones has not altered the per cent. of nitrogen, but has decreased the phosphoric acid in the bulbs, and has increased that in the tops.

Bone-meal has practically been inoperative and bears out what is generally shown in field experiments, that it is decomposed in the soil with exceeding slowness. Along

Table IV.

Plot.	Manuring per Acre.						Percentage of Constituents in						Plot.
							Fresh Roots.			Fresh Tops.			
	Dung.	Nitrate of Soda.	Super-phos-phate.	Kainit.	Dissolved Bones.	Bone Meal.	Solids.	Nitrogen.	Phos-phoric Acid.	Solids.	Nitrogen.	Phos-phoric Acid.	
	Loads.	Lb.	Lb.	Lb.	Lb.	Lb.							
1	..	..	..	..	..	..	8·81	0·147	0·0480	13·23	0·394	0·080	1
2	16	..	..	..	..	..	9·06	0·146	0·0655	12·72	0·404	0·099	2
3	16	56	..	..	..	..	8·92	0·137	0 0583	13·74	0·413	0·097	3
4	16	112	..	..	..	..	9·17	0·134	0·0592	12·08	0·357	0·084	4
5	16	168	..	..	..	..	8·68	0·141	0·0618	12·66	0·421	0·099	5
6	16	..	280	..	..	..	8·84	0·126	0·0655	12·11	0·386	0·099	6
7	16	..	560	..	..	..	8·95	0·131	0·0727	13·32	0·428	0·120	7
8	16	..	840	..	..	..	9·34	0·135	0·0748	13·73	0·445	0·133	8
9	16	..	..	224	..	..	8·52	0·128	0·0648	12·62	0·411	0·100	9
14	16	..	..	224	246	..	9·29	0·145	0·0729	13·90	0·452	0·117	14
15	16	..	..	224	492	..	8·81	0·146	0·0630	13·22	0·459	0·132	15
16	16	..	..	224	..	371	9·02	0·134	0·0608	12·82	0·412	0·107	16
17	16	18⅔	93⅓	224	82	123⅔	8·72	0·145	0·0677	12·55	0·403	0·113	17

with dung it should be seen to best advantage, because the liquid of the manure would supply a capital medium for fostering the bacteria which change the organic matter of the bone into simpler bodies. Undoubtedly some phosphate has been dissolved, and as a result the solid matter per acre has been increased.

The addition of the complex manure has brought about no great change. The solids are lessened, though by increasing the crop the total solids per acre have benefited, and since the root composition is unaltered and the ratio diminished, one may be justified in concluding that the period of growth has been shortened.

Table V.

Plot.	Manuring per Acre.	Average Crop.						Solids in Lbs. per Acre.			Ratio of Roots to Tops as 100 to	Plot.
		Roots.			Tops.			Roots.	Tops.	Total.		
		Tons.	Cwts	Qrs.	Tons.	Cwts.	Qrs.					
1	No manure	13	13	1	3	10	3	2,696	1,048	3,744	38·9	1
2	Farmyard manure 16 loads	22	13	3	4	16	2	4,606	1,378	5,984	29·9	2
3	Do. with 56 lb. nitrate	22	10	1	4	15	2	4,499	1,470	5,969	32·7	3
4	" " 112 lb. "	22	7	0	5	1	2	4,591	1,373	5,964	29·9	4
5	" " 168 lb. "	22	15	1	5	1	2	4,126	1,440	5,866	32·5	5
6	" " 280 lb. super.	23	3	3	4	4	1	4,592	1,129	5,721	24·6	6
7	" " 560 lb. "	23	16	0	4	4	0	4,772	1,253	6,025	26·2	7
8	" " 840 lb. "	24	6	3	4	9	0	5,091	1,369	6,460	26·9	8
9	" " 224 lb. kainit	22	13	0	4	3	1	4,323	1,177	5,500	27·2	9
14	No. 9 " 246 lb. dissolved bones	24	13	3	4	12	3	5,137	1,443	6,580	28·1	14
15	" " 492 lb. " "	23	17	1	4	7	0	4,710	1,288	5,998	27·3	15
16	" " 371 lb. bone-meal	24	7	2	4	19	2	4,926	1,429	6,355	29·0	16
17	No. 2 " mixed manure	24	14	3	4	9	2	4,831	1,258	6.089	26·0	17

Table VI.

Plot.	Manuring per Acre.	Lb. Manure Constituents applied per Acre in Artificial Fertilisers.		Pounds of Phosphoric Acid per Acre, extracted by Crop.				Pounds of Nitrogen per Acre, extracted by Crop.				Plot.
		Phosphoric Acid.*	Nitrogen.	Roots.	Tops.	Total.	Excess over Plot 2.	Roots.	Tops.	Total.	Excess over Plot 2.	
1	No manure	..	..	14·7	6·3	21·0	..	45·0	31·2	76·2	..	1
2	Farmyard manure, 16 loads	..	..	33·3	10·7	44·0	..	74·2	43·8	118·0	..	2
3	Do. with 56 lb. nitrate	..	8·9	29·4	10·4	39·8	..	69·1	44·2	113·3	..	3
4	" " 112 lb. "	..	17·8	29·6	9·5	39·1	..	67·1	40·6	107·7	..	4
5	" " 168 lb. "	..	26·7	31·5	11·2	42·7	..	71·9	47·9	119·8	1·8	5
6	" " 280 lb. super.	35·5	..	34·0	9·2	43·2	..	65·5	36·0	101·5	..	6
7	" " 560 lb. "	70·9	..	38·8	11·3	50·1	6·1	69·8	40·3	110·1	..	7
8	" " 840 lb. "	106·4	..	46·8	13·2	54·0	10·0	73·5	44·4	117·9	..	8
9	" " 224 lb. kainit	..	..	32·9	9·4	42·3	..	64·9	38·3	103·2	..	9
14	No. 9 " 246 lb. dissolved bones	39·4	6·1	40·3	12·2	52·5	8·5	80·2	46·9	127·1	9·1	14
15	" " 492 lb. "	78·8	12·2	33·7	12·8	46·5	2·5	78·0	44·7	122·7	4·7	15
16	" " 371 lb. bone-meal	78·8	14·1	33·2	11·9	45·1	1·1	73·2	45·9	119·1	1·1	16
17	No. 2 " mixed manure	51·2	9·7	37·5	11·3	48·8	4·8	80·3	40·4	120·7	2·7	17

* Soluble phosphoric acid only of superphosphate taken.

The Destination of the Phosphate and the Nitrogen.

What strikes one most on looking over Table VI. is the general uselessness of attempting to assist the action of dung as a manure for turnips. So far as the solids are concerned, only 6 out of 11 plots give an excess over the farmyard-manure plot, and these same plots take more phosphoric acid out of the soil, four of these and another give more nitrogen in the total plant, but only three contribute more nitrogen in the bulbs.

The total crop of plot 2 has taken from the dung about half of the soluble nitrogen and phosphoric acid, about one quarter of the total nitrogen, and one-fifth of the total phosphoric acid.

Increasing quantities of nitrate of soda do not cause proportionate amounts of nitrogen to be taken up. The medium quantity has taken out least not only in bulbs but also in tops. That with $1\frac{1}{2}$ cwt. is the only one of the nitrate plots that contains more nitrogen than the dung plot, and the excess (about one-fifteenth of that applied in the manure) is not in the bulbs but in the tops. The object of adding nitrate to dung would be, if anything, to increase the nitrogen of the crop, and especially of the bulbs, a thing which it has signally failed to do, consequently we must look upon the nitrate applied with dung as remaining in the soil, and therefore being practically lost.

Adding increasing doses of phosphate, regularly, augments the crop, and causes the plant as a whole to take up proportionate amounts of this constituent. The smallest quantity is lost, for although the bulbs are richer than those on plot 2, the tops are poorer, and as a net result, there is less phosphoric acid recovered. With the double and treble quantities, about an eleventh of the acid is returned in the crop. Though the recovery of nitrogen is proportional to the phosphoric acid, in no case does it exceed that of the plot manured with dung alone.

When kainit was added to the farmyard manure, it had very little effect upon the amount of crop, and it has shown itself to be markedly detrimental to the assimilation of nitrogen, and, to a less degree, the phosphoric acid.

Questions of loss are always very depressing, and accordingly it is a pleasure to consider the action of dissolved bones with kainit, which together form a capital manure With this combination there is virtually a gain all round. The larger quantity has been less successful than the smaller, in which from a fourth to a fifth of the phosphate has been recovered. Why the larger quantity of manure has failed while the smaller has succeeded is a matter of conjecture, and with our present knowledge it would be perhaps unwise to hazard any speculation, except the very broad one that, given certain climatic conditions there is a definite proportion between the food constituents which contribute to the nourishment of particular crops, and this ratio is disturbed with detriment to the plant.

Bone meal has returned a little of its nitrogen and phosphoric acid in the tops, the roots are practically unaffected.

The mixed manure has comparatively speaking done exceedingly well, a tenth of the phosphate applied is nearly all in the bulb, and though more nitrogen has been recovered by some of the other plots, yet the albuminoids of the bulbs are second to none.

On the whole, the addition of the one-sided artificial fertilisers to the multiform farmyard manure has resulted in a loss, nitrate and potash causing a loss of both constituents and phosphate a loss of nitrogen. It is only when a general manure, such as the mixed fertilisers or as kainit with dissolved bones, which contains various forms of phosphate and combined nitrogen, is used, that some of all the constituents are recovered in the crop.

Nottingham Section.

UNIVERSITY COLLEGE, NOTTINGHAM.

SESSION 1896-97.

Dates of Meetings.—Feb. 24th (Nottingham), March 24th (Nottingham), April 28th (Derby), May 26th (Nottingham).

Meeting held in Derby, Wednesday, December 16th, 1896.

MR. F. J. R. CARULLA IN THE CHAIR.

THE FILTRATION OF SEWAGE THROUGH COKE BREEZE.

BY S. F. BURFORD, F.C.S., AND THOS. READER SMITH, ASSOC. M. INST. C.E.

The use of coke as a filtering medium for sewage effluents, though often tried, has not hitherto proved sufficiently effective to be generally adopted. This may have arisen from the fact that the filters have frequently been too small, whilst the pieces of coke have been too large, leaving considerable spaces between, and also that the rate of passage through the filters has been too rapid, so that the so-called filters have simply acted as screens or strainers.

The results obtained by Mr. Dibdin in his experiments on London sewage, in which coke-breeze was employed, but with important modifications of the usual method, have attracted considerable attention.

The following is a record of work with an experimental filter at the Kettering Sewage Disposal Works, on the lines recommended by Mr. Dibdin. The sewage is that of a manufacturing town in which leather works constitute the chief industry; there are also dyeworks and tanneries. The water supply is less than 20 galls. per head, and a portion of the storm water is diverted.

It will be seen that the crude sewage is very foul, it is fairly represented by the sample below.

—	Chlorine.	Ammonia.		Oxygen consumed at 80° F.	
		Free.	Albumenoid.	In 15 M.	In 4 Hours.
Grains per gallon.	10·29	7·945	2·205	4·6961	5·5416

On arrival at the works the sewage is treated with lime and sulphate of alumina, in the form of aluminoferric, in the proportion of 7·8 grains of lime per gallon, and 4·7 grains per gallon of aluminoferric, reckoned on an average daily flow of 500,000 gallons. The sewage, after treatment, flows continuously through the precipitation tanks.

The filter measures 21 ft. by 10 ft. by 3 ft. deep; there are 2-in. drain pipes at the bottom; it is filled to a depth of 2 ft. 9 ins. with small coke, the size of walnuts, and covered at the top with 3 ins. of gravel. The discharge is controlled by a valve. Reckoning the water space as half the total contents of the filters it would contain about 2,000 gallons of effluent.

The experiment was commenced on the 26th Feb. 1896 by charging the filter with tank effluent, after precipitation with lime and aluminoferric. The process of filling occupied 30 minutes, and portions were taken every five minutes and mixed for analysis. The filter was left at rest for one hour and then slowly emptied during two hours; portions being taken every 15 minutes and mixed for analysis. It was worked in this way once a day, Sundays excepted, up to the 19th of March, being filled between noon and 1 p.m.

Samples were taken daily for the first five days, and afterwards on Tuesdays and Thursdays, as being representative days.

The following are the analyses for this period, all parts being expressed in grains per gallon:—

Analyses.

Date.	Tank Effluents. Chlorine.	Tank Effluents. Ammonia. Free.	Tank Effluents. Ammonia. Albumenoid.	Tank Effluents. Oxygen consumed at 80° F. In 15 mins.	Tank Effluents. Oxygen consumed at 80° F. In 4 hours.	Tank Effluents. Nitrogen as Nitrates.	Filter Effluents. Chlorine.	Filter Effluents. Ammonia. Free.	Filter Effluents. Ammonia. Albumenoid.	Filter Effluents. Oxygen consumed at 80° F. In 15 mins.	Filter Effluents. Oxygen consumed at 80° F. In 4 hours.	Filter Effluents. Nitrogen as Nitrates.
1896.												
Feb. 25	6·58	4·683	0·525	1·9236	2·9540	0·220	7·19	3·871	0·532	0·8335	1·3540	0·320
„ 26	5·83	2·898	0·350	1·0318	1·6469	0·220	6·11	3·283	0·462	0·5283	0·8598	0·384
„ 27	5·97	2·583	0·658	0·8878	1·7774	0·368	5·97	2·583	0·413	0·4620	0·7168	0·280
„ 28	6·11	2·639	0·490	0·8176	1·3745	0·288	6·11	2·604	0·511	0·4046	0·7084	0·352
„ 29	6·24	3·143	0·581	0·8120	1·3916	0·064	6·24	3·115	0·532	0·5661	0·8951	0·192
Mar. 3	4·61	2·100	0·392	1·0827	2·0188	0·288	4·75	2·296	0·378	0·5723	0·9520	0·320
„ 5	6·04	3·661	0·483	1·2540	1·7589	0·416	5·97	3·962	0·441	0·5955	0·9601	0·432
„ 10	4·61	1·967	0·462	0·6652	1·1004	0·352	4·61	2·572	0·301	0·3197	0·6042	0·352
„ 12	5,28	3·353	0·420	0·8069	1·2880	0·128	5·21	2·583	0·434	0·3458	0·5725	0·400
„ 17	5·48	2·296	0·350	0·8022	1·3235	0·448	5·21	2·821	0·308	0·3500	0·6361	0·512
„ 19	5·22	2·835	0·539	0·8680	1·4396	0·416	5·09	2·751	0·431	0·4382	0·6650	0·416

Percentages of Improvement in Filter Effluents as compared with Tank Effluents.

Date.	Ammonia. Free. Increase.	Ammonia. Free. Decrease.	Ammonia. Albumenoid. Increase.	Ammonia. Albumenoid. Decrease.	Oxygen consumed at 80° F. 15 mins. Decrease.	Oxygen consumed at 80° F. 4 hours. Decrease.	Remarks.
1896.							
Feb. 25	..	17·33	1·33	..	56·66	54·16	One hour at rest in filter:— Per Cent. Free NH_3, increase .... 3·54 Albumenoid NH_3, decrease ..... 7·62 Oxygen in 15 minutes, decrease.. 49·87 Oxygen in 4 hours, decrease ..... 50·03
„ 26	13·28	..	32·0	..	48·70	47·81	
„ 27	..	..	..	37·23	47·90	59·70	
„ 28	..	1·32	4·28	..	50·51	48·47	
„ 29	..	0·89	..	8·43	30·28	35·65	
Mar. 3	9·33	..	..	3·57	47·14	52·82	
„ 5	8·22	..	..	8·69	52·51	45·41	
„ 10	30·75	..	..	34·84	51·93	45·10	
„ 12	..	22·96	3·33	..	57·13	55·59	
„ 17	22·86	..	..	12·00	56·33	51·92	
„ 19	..	2·96	..	20·03	49·51	53·78	

From March 24 to April 23 the experiment was varied, an earlier sewage, 10.30—11.0 a.m., being used, and the tank effluent allowed to remain at rest in the filter for 3 hours. After April 1, carbferalum, 3·13 grains per gall. was substituted for the aluminoferric.

The analyses for this period are as follows:—

Date.	Tank Effluents. Chlorine.	Tank Effluents. Ammonia. Free.	Tank Effluents. Ammonia. Albumenoid.	Tank Effluents. Oxygen consumed at 80° F. In 15 mins.	Tank Effluents. Oxygen consumed at 80° F. In 4 hours.	Tank Effluents. Nitrogen as Nitrates.	Filter Effluents. Chlorine.	Filter Effluents. Ammonia. Free.	Filter Effluents. Ammonia. Albumenoid.	Filter Effluents. Oxygen consumed at 80° F. In 15 mins.	Filter Effluents. Oxygen consumed at 80° F. In 4 hours.	Filter Effluents. Nitrogen as Nitrates.
1896,												
Mar. 24	4·34	1·652	0·371	0·4121	0·8710	0·640	4·34	1·260	0·273	0·1030	0·3468	0·768
„ 26	4·61	1·715	0·560	0·7516	1·1284	0·576	4·61	1·400	0·294	0·2312	0·4095	0·704
„ 31	4·61	1·736	0·413	0·6804	1·0348	0·480	4·48	1·491	0·322	0·2289	0·4340	0·704
April 1	5·02	2·205	0·455	0·7240	1·2292	0·384	4·75	1·351	0·413	0·2031	0·3794	0·768
„ 7	4·76	2·352	0·476	0·4420	0·7548	0·064	4·76	1·848	0·483	0·1907	0·3159	0.912
„ 9	5·46	2·555	0·609	0·8148	1·3955	0·064	5·46	1·435	0·483	0·3207	0·6230	0·480
„ 14	5·46	2·786	0·553	0·7700	1·2488	0·032	5·46	1·827	0·343	0·2884	0·4928	0·544
„ 16	5·04	2·684	0·511	1·3795	2·6107	0·048	5·04	1·582	0·364	0·4057	0·7338	0·512
„ 21	6·02	3·024	0·640	1·0068	1·6833	0·128	5·88	2·222	0·409	0·3707	0·6300	0·288
„ 23	6·44	2·842	0·798	1·0116	1·6091	0·160	6·16	1·844	0·336	0·3794	0·5767	0·256

Percentages of Improvement.

Date.	Ammonia. Free. Increase.	Ammonia. Free. Decrease.	Ammonia. Albumenoid. Increase.	Ammonia. Albumenoid. Decrease.	Oxygen consumed at 80° F. In 15 mins. Decrease.	Oxygen consumed at 80° F. In 4 hours. Decrease.
1896.						
March 24	..	23·72	..	26·41	75·02	60·18
„ 26	..	18·36	..	47·50	69·23	61·74
„ 31	..	14·11	..	22·03	66·35	58·02
April 1	..	38·73	..	9·23	71·94	69·16
„ 7	..	21·42	1·47	..	56·85	58·14
„ 9	..	48·83	..	20·68	60·64	55·34
„ 14	..	34·42	..	37·97	62·54	60·53
„ 16	..	40·99	..	28·76	70·59	71·73
„ 21	..	26·52	..	36·09	63·18	62·57
„ 23	..	35·11	..	57·89	62·49	67·88

Remarks. Three hours at rest in filter:—

	Per Cent. Decrease.
Free NH_3	29·72
Albumenoid NH_3	27·18
Oxygen in 15 minutes	65·88
Oxygen in 4 hours	62·72

From April 24 to May 21 the effluent remained at rest in the filter for five hours, the filter being filled from 10.30 to 11 a.m. as before.

Analyses.

Date.	Tank Effluents. Chlorine.	Tank. Ammonia. Free.	Tank. Ammonia. Albumenoid.	Tank. Oxygen consumed at 80° F. In 15 mins.	Tank. Oxygen consumed at 80° F. In 4 hours.	Tank. Nitrogen as Nitrates.	Filter Effluents. Chlorine.	Filter. Ammonia. Free.	Filter. Ammonia. Albumenoid.	Filter. Oxygen consumed at 80° F. In 15 mins.	Filter. Oxygen consumed at 80° F. In 4 hours.	Filter. Nitrogen as Nitrates.
1896.												
April 28	5·32	2·646	0·693	0·8948	1·9678	..	5·60	2·030	0·455	0·2923	0·6619	0·256
„ 30	6·02	2·943	0·763	1·0774	1·8219	..	5·95	2·310	0·273	0·3796	0·6843	0·160
May 7	6·16	2·968	0·742	0·9240	1·8012	0·288	6·16	1·827	0·420	0·3623	0·6330	0·128
„ 12	6·30	3·605	0·805	1·0981	1·9962	0·16	6·16	2·394	0·518	0·3856	0·7066	0·160
„ 14	6·86	3·801	0·728	1·1586	2·0370	0·16	6·30	2·387	0·490	0·4037	0·7179	0·220
„ 19	6·16	3·773	0·903	1·1687	1·8818	..	6·16	2·401	0·574	0·4168	0·7324	0·160
„ 21	7·84	5·187	1·036	1·6363	2·9568	..	7·42	3·451	0·882	0·4378	1·0659	0·256

Percentages of Improvement.

Date.	Ammonia. Free. Increase.	Ammonia. Free. Decrease.	Ammonia. Albumenoid. Increase.	Ammonia. Albumenoid. Decrease.	Oxygen consumed at 80° F. In 15 mins. Decrease.	Oxygen consumed at 80° F. In 4 hours. Decrease.
1896.						
April 28	..	23·28	..	34·34	67·33	66·36
„ 30	..	21·52	..	64·22	64·76	62·44
May 7	..	38·44	..	43·39	60·79	64·86
„ 12	..	33·59	..	35·65	64·88	64·60
„ 14	..	37·20	..	32·69	65·15	64·75
„ 19	..	36·36	..	36·43	64·33	61·08
„ 21	..	33·46	..	14·86	73·24	64·07

Remarks. 5 hours at rest in filter:—

	Per Cent. Decrease.
Free NH_3	31·98
Albumenoid NH_3	37·37
Oxygen in 15 minutes	65·78
Oxygen in 4 hours	64·02

From May 22 the filter was worked twice daily, being filled from 10.30 to 11 a.m., and from 6.0 to 6.30 p.m., and left at rest for three hours each time.

After June 2 the amount of carbferalum was increased to 4·7 grains per gallon.

Analyses.

Date.	Tank Effluents. Chlorine.	Tank. Ammonia. Free.	Tank. Ammonia. Albumenoid.	Tank. Oxygen consumed at 80° F. In 15 mins.	Tank. Oxygen consumed at 80° F. In 4 hours.	Tank. Nitrogen as Nitrates.	Filter Effluents. Chlorine.	Filter. Ammonia. Free.	Filter. Ammonia. Albumenoid.	Filter. Oxygen consumed at 80° F. In 15 mins.	Filter. Oxygen consumed at 80° F. In 4 hours.	Filter. Nitrogen as Nitrates.
1896.												
June 2	6·92	4·585	0·518	1·3142	2·2340	0·06	7·19	2·394	0·336	0·5333	0·9324	0·448
„ 4	7·19	3·983	0·700	1·3311	2·3408	..	7·19	2·611	0·301	0·6334	1·0192	0·240
„ 9	6·79	4·501	1·001	1·2320	2·1667	0·12	6·79	2·807	0·609	0·4894	0·7381	0·480
„ 11	3·80	1·365	0·385	0·5600	1·0922	..	4·88	0·868	0·364	0·2036	0·4485	1·000
„ 16	6·86	4·109	0·553	1·2135	2·0554	..	6·99	2·597	0·308	0·5751	0·9824	0·350

PERCENTAGES OF IMPROVEMENT.

Date.	Ammonia.				Oxygen consumed at 80° F.		Remarks.
	Free.		Albumenoid.		In 15 mins.	In 4 hours.	
	Increase.	Decrease.	Increase.	Decrease.	Decrease.	Decrease.	
1896.							Twice filling in 24 hours. 3 hours at rest in filter :—
June 2	..	47·78	..	35·32	59·42	58·26	Per Cent. Decrease.
„ 4	..	34·44	..	57·00	52·49	56·45	Free NH_3 .... 38·61
„ 9	..	37·63	..	39·16	60·27	65·92	Albumenoid NH_3 .... 36·24
„ 11	..	36·41	..	5·45	63·64	58·93	Oxygen in 15 minutes .... 57·69
„ 16	..	36·79	..	44·30	52·60	51·72	Oxygen in 4 hours .... 58·25

During the entire course of these experiments there was never any indication of putridity, and when the experiments were stopped on June 16th, and the filter broken into, the coke was found to be absolutely free from any putrid odour. In all cases the filter effluent was clear and comparatively free from odour.

The results of these experiments indicate that the process is worthy of trial on a larger scale, and that if the filter be carefully constructed, the effluent allowed to remain at rest for some hours, and thorough aeration induced, the process is a valuable help in the treatment of sewage effluent.

Further experiments will be made as steps are being taken to construct large filters.

It will be noticed that in Mr. Dibdin's experiments the degree of purification, whether judged by ammonias or oxygen consumed was about the same, being 75 to 80 per cent. as compared with the unfiltered effluent. In our experiments the purification was not so considerable, and as judged by the oxygen consumed was much higher than in the case of the ammonias, this probably arising from the different character of the crude sewage.

DISCUSSION.

Mr. JNO. WHITE (County Analyst, Derbyshire) said that he was impressed with the very irregular manner in which the improvement was brought about. If they took the tank effluent of April 7th and looked at the amount of albuminoid ammonia, 0·476, they would see that it had increased to 0·483 on the same day in the filter effluent, while if they referred to the next sample, two days later, they found a reduction from 0·609 to 0·483. There were very many striking examples of that kind to be found, but the figures wanted careful study.

Mr. F. E. LOTT said that five or six years ago he had, in conjunction with his partner Mr. Matthews, carried out several experiments upon a pretty bad sewage, both with and without chemicals, and they noted one point which, as far as they then knew, was not previously commented on, namely, that the amount of deposit, when sewage was allowed to settle without any chemical treatment at all, was very considerable; and when treated with the small amount of chemicals frequently recommended, the result hardly supported the strong claim in favour of such treatment, that was to say, simply as regarded precipitation. In respect to the actual precipitation effect of chemicals upon sewage, there was a great divergence of opinion in the various papers read on this subject. In an experiment lasting four or five days they found that a million gallons of sewage passing slowly through the experimental tanks deposited 8·8 tons of sludge by simple subsidence, whereas treated with 10 grains of lime per gallon a similar quantity of sewage yielded only 15·7 tons of sludge—a considerable increase, but not so great when the actual amount of lime added and precipitated is deducted—a point they particularly noted was that when air-drying the sludge, that obtained by simple subsidence was much less offensive than that obtained by precipitation with lime or with other chemicals tried. He would like the opinion of the authors of this paper upon the question of precipitation apart from the chemical changes. He would further like to ask if the sewage they experimented with was allowed to partially settle before treatment. That, he thought, was an important point, as his opinion was that sewage allowed to settle in tanks before the addition of chemicals gave much better results when subsequently treated than when the chemicals were added originally to the raw sewage. There were chemicals which assisted as separators of suspended and other matter, and chemicals which only made the effluent less offensive; fortunately the two sets of chemicals were not always identical, and might therefore be used at different times.

Mr. J. T. WOOD said Mr. Reader Smith had told them that the sewage of the town of Kettering was to a large extent from tannery and leather works, and it was a well-known fact that sewage from places of that character containing lime liquors and waste tan liquors mutually precipitated one another. Tanneries dealing with their own sewage could very often get a clear effluent without any other treatment than simply mixing the different liquors in the works. It was known in bacteriological work that small quantities of antiseptics seemed to have a vivifying effect on many micro-organisms. Very small quantities actually seemed to increase the activity of the bacteria. It occurred to him that the same was the case with lime, though it may have increased the putrefaction going on in the sewage merely by rendering it alkaline.

Mr. L. ARCHBUTT said that there were several figures he found it difficult to reconcile with each other. Take, for instance, the sewage on April 7th. They found free ammonia 2·352, and in the filter effluent 1·848—a decrease of half a grain per gallon. There was a slight increase in the albuminoid ammonia, and an increase in the nitrogen as nitrates of 0·912 grain. But on April 9th, two days later, they found a decrease in the free ammonia of 1·12 grains, a decrease also in the albuminoid ammonia of 0·226 grain, and yet the nitrates only showed an increase of 0·48 grain. On May 7th, there was a still more extraordinary result, the free ammonia disappearing to the extent of 1·14 grains per gallon, the albuminoid ammonia 0·322 grain, and yet the nitrates had, according to the analyses, diminished also from 0·288 to 0·128 grain. These figures, to his mind, appeared to indicate that putrefaction was going on, but he would be glad to hear what explanation the readers of the paper had to offer.

Mr. S. F. BURFORD, in reply, said certain variations and irregularities were due to the fact that one day the sewage would be in its normal state and then the next it would be abnormal. With regard to the matter of the amount of lime to be used, it would first be necessary to ascertain the nature of the sewage.

Mr. T. READER SMITH also replied, and said he found that a much clearer effluent could be got by the use of chemicals than without, and they also got a much more rapid precipitation. With reference to the amount of sludge precipitated, they had had 80 tons per day, representing 8 tons of pressed cake.

The CHAIRMAN said the sewage question was one of the greatest importance to them in Derby. He was only sorry that they had not had any Town Councillors there

that evening, as they might have asked some questions to elucidate the points in dispute between the town and county.

SOME PROPERTIES OF CERTAIN METALLIC PHOSPHATES.

BY R. M. CAVEN B.SC., F.I.C., AND A. HILL.

In extension of the observations made by one of us on some properties of ferric phosphate (this Journal, January 31st, 1896), we have made experiments on the hydrolysis of the phosphates of certain other metals, by water and aqueous potash and ammonia. It has already been shown that $FePO_4$ is completely hydrolysed by NH_4HO aq. and therefore, *a fortiori* by KHO solution. Experiments were next made on *aluminium phosphate*; and since this compound is readily soluble in KHO aq., and NH_4HO aq., behaviour analogous to that of $FePO_4$ with these reagents could not be looked for. On account of this fact the precipitation of $Al(OH)_3$ by the decomposition of $AlPO_4$, in presence of excess of KHO aq. or NH_4HO aq., could not be achieved, nor the hydrolysis demonstrated.

It is well known that $AlPO_4$ is soluble in solutions of Al salts. If Na_2HPO_4 aq. is added drop by drop to a solution of pure $Al_2(SO_4)_3$ in water, at first no precipitate appears, and then a cloudiness which disappears on shaking. When as much Na_2HPO_4 aq. has been added as the clear solution can contain, slight warming causes a cloudiness, and, on boiling the liquid, $AlPO_4$ is gradually reprecipitated, but upwards of an hour's boiling is necessary for complete precipitation. The clear filtrate is then found to contain both Al and PO_4; so that $AlPO_4$ is somewhat soluble in $Al_2(SO_4)_3$ aq., even at the boiling point of the latter. Slight precipitation of $AlPO_4$ from its saturated solution obtained by shaking with $Al_2(SO_4)_3$ aq. occurs, on standing, even in the cold. The addition of NH_4Cl aq. to this solution does not cause any precipitation.

If $AlPO_4$ is dissolved in NH_4HO aq., KHO aq., or NaHO aq., it is partially reprecipitated from these solutions by the addition of NH_4Cl aq.; the completeness of the reprecipitation depending on the relative masses of chloride and hydroxide present. NH_4HO aq. added after some NH_4Cl aq. to $AlPO_4$ dissolved some of the precipitate, and the $AlPO_4$ was again precipitated from solution on adding more NH_4Cl aq. to the filtrate.

$AlPO_4$ is soluble in a large quantity of $H\bar{A}$ aq. in the cold, and is reprecipitated from this solution on boiling, but not by $NH_4\bar{A}$ aq. in the cold. If some $AlPO_4$ is dissolved in the least possible quantity of HCl aq., $H\bar{A}$ aq. added in excess and then $NH_4\bar{A}$ aq., no precipitation takes place. If only a few drops of $H\bar{A}$ aq. have been added, $AlPO_4$ comes down on the addition of $NH_4\bar{A}$ aq., but this precipitate is redissolved completely on the addition of excess of $H\bar{A}$ aq. A solution of $Al_2(SO_4)_3$ containing a little $AlPO_4$ was not precipitated by $H\bar{A}$ aq. and $NH_4\bar{A}$ aq., but the addition of a little Na_2HPO_4 aq. caused precipitation. Also a solution of $AlPO_4$ in $Al_2(SO_4)_3$ aq., containing almost a saturating amount of $AlPO_4$ yielded a precipitate with $H\bar{A}$ aq. and $NH_4\bar{A}$ aq.

The slow hydrolysis of $AlPO_4$ by water (as well as that of $FePO_4$) was shown by R. T. Thomson (this Journal, March 29, 1886).

The properties of *chromic phosphate* are very similar to those of $AlPO_4$. $CrPO_4$ was found to be soluble in $Cr_2(SO_4)_3$ aq. though not to such an extent as in the case of $AlPO_4$ in $Al_2(SO_4)_3$ aq.

When a saturated solution was made by adding Na_2HPO_4 aq. to $Cr_2(SO_4)_3$ aq., until a permanent precipitate remained after shaking, the clear filtrate deposited $CrPO_4$ on being warmed. The complete precipitation was less tedious than in the case of the solution of $AlPO_4$ in $Al_2(SO_4)_3$ aq., but, after boiling produced no further turbidity, the clear liquid was found to contain PO_4. NH_4Cl aq. did not cause the saturated solution to precipitate.

$CrPO_4$ is soluble in a moderate quantity of NH_4HO aq. in the cold, though much less readily soluble than $AlPO_4$. A reddish-violet liquid is formed, from which the addition of NH_4Cl aq. causes a partial reprecipitation of $CrPO_4$. $CrPO_4$ is also precipitated from its solution in KOH aq. by a moderate quantity of NH_4Cl aq., as is the case with $AlPO_4$.

$CrPO_4$ is readily soluble in $H\bar{A}$ aq., and when this reagent is added in moderate quantity after the precipitation of $CrPO_4$ from a nearly neutral solution by $NH_4\bar{A}$ aq., it redissolves much, or even all, of the precipitate.

Hot water hydrolyses $CrPO_4$. The precipitate obtained by adding Na_2HPO_4 aq. to $Cr_2(SO_4)_3$ aq. was washed with repeated quantities of hot water, and after all the sulphate had been washed away, much PO_4 appeared in each lot of wash water. The precipitate, which settled with difficulty at first, became much denser during the process of washing, and then settled quickly. The colour of the precipitate also changed from greyish-green to dark green.

For the detection of Fe, Al, and Cr, in a mixed precipitate of their phosphates, the following method has proved very satisfactory. Dry the precipitate and heat it in a Pt. box—made by folding a piece of foil 2 ins. square over a block $1\frac{1}{4}$ ins. square—together with enough solid KHO, free from Al., to enable the whole to fuse together.

Maintain the mass in a state of fusion over the Bunsen flame, and freely exposed to the air for a minute or two, and when cold extract with water and filter. The Fe will be present as Fe_2O_3, which will remain on the filter, and the Al and Cr will be in the alkaline filtrate. The addition of excess of NH_4Cl aq. will precipitate the Al, probably as a mixture of hydroxide and phosphate; and the yellow colour of the filtrate from this precipitate will indicate the presence of Cr. This may be confirmed by acidifying and adding H_2SO_3 aq. to reduce the chromate to green chromium salt. Traces of Cr and Al may be detected by this means in presence of large quantities of PO_4 and Fe; and the damage to the Pt. foil is so small that the same piece may be used many times without perceptible injury.

For the investigation of the behaviour of *cupric phosphate* with various reagents, the substance was precipitated as a sky-blue powder by adding Na_2HPO_4 aq. to $CuCl_2$ aq. According to Steinschneider (Journ. Chem. Soc. **60**, 1423), the precipitate thus obtained may vary in composition under different circumstances, but is likely to consist of a double salt of $Cu_3(PO_4)_2$ and NaCl. We found the precipitate to be soluble in $CuCl_2$ aq. and also in $CuSO_4$ aq. to a perceptible extent. This was proved by adding a little Na_2HPO_4 aq. to excess of $CuCl_2$ aq., and also of $CuSO_4$ aq., filtering and testing the filtrate for PO_4; and also by adding only a drop or two of Na_2HPO_4 aq. to $CuSO_4$ aq., and shaking when the precipitate first formed was redissolved.

Cupric phosphate is said to be decomposed by hot water under pressure (Watt's Dict. 1894, Art. Phosphates). In order to investigate this property, the precipitate, obtained as above, was filtered through calico and stirred with boiling water repeatedly in a large beaker. The precipitate was allowed to settle after each treatment, and the supernatant liquid syphoned off and tested for Cl and PO_4, the process being repeated eight times.

The following observations on the relative quantities of Cl and PO_4 present in the wash water after each treatment, show that the cupric phosphate precipitate was hydrolysed by hot water under ordinary atmospheric pressure:—

1st treatment with hot water ..	Cl more than PO_4.
2nd „ „ „ ..	„ „
3rd „ „ „ ..	Cl getting less, PO_4 increasing.
4th „ „ „ ..	Cl nearly absent, PO_4 still present in quantity.
5th „ „ „ ..	Trace of Cl, PO_4 present in large amount.
6th „ „ „ ..	Trace of Cl. PO_4 about the same.
7th „ „ „ ..	Faint trace of Cl, PO_4 about the same.
8th „ „ „ ..	Free from Cl, PO_4 still present in large amount.

During the process of washing the precipitate altered greatly in appearance. It became much denser, settled quickly, and gradually changed in colour from sky blue to light grass-green. On boiling the precipitate, left after the above washings, with water, much PO_4 was dissolved therefrom.

Steinschneider (*ibid*) states that on washing normal cupric phosphate with water, a basic phosphate of the composition $4CuO, P_2O_5, H_2O$ is obtained.

In order to try and obtain this compound, we boiled the precipitate with water for about 9 hours under an inverted condenser, the wash water being changed three times during this period. After this treatment further washing did not remove any PO_4 or other soluble matter, and the precipitate had therefore arrived at a state of constant composition.

It was dried at 100° C. and then 1 grm. was taken for analysis and was further dried at 100° C. until the weight was constant. The resulting powder—0·9952 grm. yielded 0·6620 grm. of CuO by hydrolysis with KHO aq. (see below) = 66·52 per cent. CuO; and the filtrate, acidified with HCl aq. and treated with NH_4HO aq. and magnesia mixture, gave 0·4630 grm. $Mg_2P_2O_7$ = 29·67 per cent. P_2O_5.

These results would indicate the presence of—

$$100 - (66·52 + 29·67) = 3·81 \text{ per cent. } H_2O.$$

The percentage of combined water was estimated directly by the loss of weight which the powder, dried at 100° C., suffered on further heating.

0·4980 grm. lost 0·0020 grm. after being heated for two hours at 130° C., 0·0010 grm. after an hour at 170° C.; and a further loss of 0·0150 grm. took place during ignition at a dull red heat over the luminous Argand flame. After this the weight remained constant.

The percentage of combined water thus estimated = 3·62. The actual percentage composition of the precipitate found and that corresponding to the formula $4CuO, P_2O_5, H_2O$, are as follows :—

——	Found.	Theory.
CuO	66·52	66·46
P_2O_5	29·67	29·77
H_2O	3·62	3·77
	99·81	100·00

These results, therefore, support the statement of Steinschneider as to the composition of the precipitate after washing and drying at 100° C. till constant.

The tenacity with which the single molecule of water remains in combination with the basic phosphate is remarkable, since a dull red heat is necessary for its complete elimination. The ignited precipitate, consisting of—

$$Cu_3(PO_4)_2, CuO\ (= 4CuO, P_2O_5),$$

was of an olive-green colour.

Action of KHO aq. on Precipitated Cupric Phosphate.—In the cold, KHO aq. did not appear to produce any change; but on boiling, the precipitate became dark coloured and seemed to consist of CuO only.

The action of KHO aq. was investigated quantitatively. 1 grm. of $CuSO_4 5H_2O$ was dissolved in hot water, Na_2HPO_4 aq. added in excess, and the precipitate filtered off. This precipitate was transferred to a porcelain dish, excess of KHO aq. added, and the mixture boiled for some time. The dark-coloured precipitate was filtered off, washed free from alkali, dried, ignited, and weighed.

Weight of precipitate obtained	0·3179 grm.
Theoretical weight of CuO obtainable from 1 grm. $CuSO_4 5H_2O$	0·3189 „

Therefore cupric phosphate is completely hydrolysed by KHO, aq.

Properties of Bismuth Phosphate, $BiPO_4$.—Bismuth carbonate was added in excess to HCl aq., so that the solution contained no free acid. The solution was filtered, CO_2 boiled off from the filtrate, and Na_2HPO_4 aq. added in excess.

Solubility of $BiPO_4$ in $BiCl_3$ aq.—$BiCl_3$ aq., prepared as above by filtration from excess of carbonate, possessed an acid reaction. Na_2HPO_4 aq., added drop by drop, produced a precipitate which dissolved in the excess of $BiCl_3$ aq. Also precipitated $BiPO_4$ was added to excess of $BiCl_3$ aq., and the solution filtered. The filtrate contained PO_4, showing that $BiPO_4$ is somewhat soluble in $BiCl_3$ aq.

The precipitated $BiPO_4$ was washed in a similar manner to the cupric phosphate precipitate, and the wash water tested.

1st treatment	Cl and PO_4 about the same.
2nd „	Cl and PO_4 both diminished.
3rd „	Traces only of Cl and PO_4.
4th „	Neither Cl nor PO_4 present.

So that $BiPO_4$ was not hydrolysed by hot water; and when boiled with water for some time, no PO_4 was abstracted.

By boiling with KHO aq. and filtering, a fair amount of PO_4 was obtained, showing hydrolysis to be considerable. A similar treatment with NH_4HO aq. extracted some PO_4, though the action was not so pronounced as in the case of KHO aq.

Quantitative Investigation of the Action of KHO aq. on $BiPO_4$.—A weighed quantity of pure Bi_2O_3 was dissolved in the *least possible quantity* of HCl aq.; excess of Na_2HPO_4 aq. was then added to the solution, and the precipitate filtered off and transferred to a large porcelain dish. KHO aq. was added, the liquid and precipitate boiled for some time, the latter allowed to settle, and the supernatant liquid carefully decanted through a filter. The residue in the dish was boiled with fresh KHO aq., the liquid again decanted, and the precipitate washed free from alkali by decantation and transferred to the filter. The residue was straw-coloured. It was dried on the filter and ignited in a porcelain crucible until the weight was constant. If $BiPO_4$ is completely hydrolysed by KHO aq., then the precipitate obtained should weigh the same as the Bi_2O_3 originally taken, since it has the same composition :—

Weight of Bi_2O_3 taken	0·8396 grm.
„ precipitate obtained	0·8378 „

Therefore the hydrolysis of $BiPO_4$ by KHO aq. is complete.

Properties of Lead Phosphate, $Pb_3(PO_4)_2$.—This substance was obtained as a heavy white precipitate by adding excess of Na_2HPO_4 aq. to $Pb(NO_3)_2$ aq.

To try if $Pb_3(PO_4)_2$ is soluble in $Pb(NO_3)_2$ aq., some of the well-washed precipitate was boiled with $Pb(NO_3)_2$ aq. and filtered. The filtrate did not give any yellow precipitate or colour with ammonium molybdate solution, proving that $Pb_3(PO_4)_2$ is insoluble in $Pb(NO_3)_2$.

The behaviour of $Pb_3(PO_4)_2$ with hot water was examined as in other cases. The precipitate was washed four times by decantation, and then the wash water from the fifth washing was tested for nitrate and phosphate, neither of which was found. Another treatment yielded not a trace of either nitrate or phosphate. Hence $Pb_3(PO_4)_2$ is not hydrolysed by hot water. The precipitate was also boiled with water for some time, and the filtrate yielded only a slight yellow coloration with the molybdate solution. So that $Pb_3(PO_4)_2$ is scarcely attacked by continued boiling with water.

Action of NH_4HO aq.—Some lead phosphate was digested with dilute ammonia in the cold. The filtrate showed small quantities of PO_4 and of Pb. Since $Pb(OH)_2$ is soluble in NH_4HO aq., the presence of lead was to be expected. On boiling with NH_4HO aq. for some time, large quantities of Pb and of PO_4 were found in the filtrate.

Action of KHO aq.—Only a slight action took place in the cold, but on boiling with aqueous potash the precipitate was entirely dissolved. As in the case of $AlPO_4$, no hydrolysis could be demonstrated, and we cannot say whether $Pb_3(PO_4)_2$ dissolves in KHO aq. as such, or whether it suffers hydrolysis previous to solution.

Discussion.

The Chairman said it would be obvious to everyone present that the observations made by Mr. Caven were of special interest to analytical investigators. They had a paper to follow, which was an extremely important one to the borough of Derby, on the purification of sewage, and he thought it would be best to have the paper just read in print before they began to discuss it. In that way they possibly would also gain a little by others, who were not then present, joining in the discussion.

Scottish Section.

Chairman: John Clark.
Vice-Chairman: J. B. Readman.
Committee:

A. C. J. Charlier.	A. Macdonald.
R. Cox.	R. C. Menzies.
C. A. Fawsitt.	E. Ostlere.
J. Gray.	Sir R. Pullar.
D. Harris.	H. Rose.
G. G. Henderson.	P. Rottenburg.
R. A. Inglis.	D. R. Steuart.
W. I. Macadam.	R. T. Thomson.

Hon. Secretary and Treasurer:
Thomas Gray, Technical College, 204, George Street, Glasgow.

SESSION 1896-97.

Meeting held in Edinburgh on Tuesday, Dec. 22nd, 1896.

MR. JOHN LAING, F.I.C., IN THE CHAIR.

THE REMOVAL OF ORGANIC MATTER FROM PEATY WATER.

BY J. B. READMAN, D.SC., F.R.S.E.

The treatment of water by the method about to be described is not new. What may probably be novel in it was the attempt to purify water, highly coloured by peat, in a large service reservoir, from which, while the treatment was going on, a considerable area was being served with water.

In May 1895, a large reservoir of about 113 acres in extent, constructed by direction of the County Council of Linlithgow, was opened at Forrestfield, near Bathgate. Its greatest depth is about 24 ft., and its holding capacity is about 200 million gallons. Nearly one million gallons are drawn off every day.

It was found before completion that the capacity of the lake was not sufficiently large, and a considerable quantity of peat, which forms part of the bottom, had to be excavated. The result of this was to expose the freshly-cut and puddled-up surface to the solvent action of the water when the lake was filled with water. Owing to the urgent necessity for assessment purposes of having the reservoir filled and the water turned on by a certain date, little or no time could be spared to cover up or otherwise deal with this peaty bed, and the consequence was the lake was filled soon after it was deepened.

It was not long before the people in the distributing area discovered that the new supply was highly discoloured and strongly impregnated with a peaty flavour and odour, and, besides, was most unpalatable, and a general complaint arose that the water was unfit to use for potable purposes.

As Public Analyst for Linlithgowshire, where the reservoir is situated, samples of water both from the feeder and from the lake were, for the first time, sent to me for examination. Previous to this, and as far as I am aware, no chemical examination, either of the stream or of the springs had been supplied to, or demanded by, the Local Authority. I found that the Forrest Burn water was of fairly good quality and practically free from peat, *at that time, i.e.*, in the spring of the year, but that the water of the reservoir was highly coloured and practically uniformly so in this respect all over its area.

On the 4th of June of last year (1895), just about one month after the water was formally turned on, I reported on the water as follows:—

June 1895.

"Total solids	7·63	grains per gallon.
Volatile residue	2·10	
Saline "	5·53	
Free ammonia	0·0042	" "
Albumenoid ammonia	0·0210	" "
Nitrates and nitrites	None.	
Combined chlorine	1·86	grains per gallon.
Total hardness	2·10	
Temporary	2·10	
Permanent	None.	
Iron	0·098	" "
Phosphates	Very faint traces."	

The water had a faint alkaline reaction, was of a brown peaty colour, and contained a small quantity of suspended matter. The water was soft, and free from animal organic pollution, but it contained a large quantity of dissolved vegetable matter (peat), which rendered it dark brown in colour and gave the water, when heated, the odour of decayed leaves. In our opinion, the presence of so much peaty matter made the water in its then condition unfit as a supply for general domestic purposes and specially so for potable purposes.

The excessive amount of peat is indicated, not only by the colour of the water, but also by the high figure for albumenoid ammonia. Waters of this class (containing even less dissolved vegetable matter than this one under examination) have been known to give rise to outbreaks of diarrhœa.

We had made experiments in the laboratory from time to time on the water, and ascertained that the addition of about $3\frac{1}{2}$ grains per gallon, or 1 lb. of alum for every 2,000 gallons of water, threw out practically all the colouring matter from the water, and that this result was accomplished during the course of one night or, say, after about 12 hours' standing.

As economy had to be considered, my first endeavour was to get a cheap source of alumina, and this was readily obtained from alum cake, which was guaranteed to contain 14 per cent. of soluble alumina and not over $\frac{1}{2}$ per cent. of uncombined sulphuric acid. As the behaviour of the precipitate when present in quantity was uncertain, I thought it prudent to apply the alum in the initial stage as far from the outflow as possible, *i.e.*, at the extreme west end of the lake, where the Forrest Burn enters. It was thus added nearly one mile away from the pipes which supply the district from the reservoir.

To carry this out a kind of rack made of spars of wood was constructed across the Forrest Burn. In reviewing this procedure one must keep in mind that the water was uniformly coloured all over the lake, but it was certainly very much less coloured than when I first reported on it. The treatment was begun on 30th March last by adding to the rack 30 cakes per day, *i.e.*, about 20 cwt. The effect was most rapid: the hydrated alumina precipitate, as expected, formed a lake with the colouring matter, and within a few yards of the spot the precipitate had subsided and the water above and around was clear, sparkling, and colourless. The addition of alum at this place was continued for a week, and then we divided off the lake into five divisions by placing posts on opposite banks about equal distances apart. Thus Nos. I. were driven into the ground about 300 yards from the rack, Nos. II. were placed about 200 yards from Nos. I., Nos. III. about 200 yards from Nos. II., and so forth.

Success, so far, having emboldened us, we made, on the 7th April, an alteration of the place where the cake was added. In place of putting it all in the rack 18 cakes only were placed there, and the remainder placed just below the water on the bank at No. I. post. The practice was likewise started of drawing five samples from the reservoir every day, midway between each set of posts.

Up to now the alum was added entirely by the rack or by placing the cakes on the shore. Now a raft was constructed, in the form of a square, with a spar bottom, always under water, upon which the cakes were placed. This raft was anchored between posts Nos. II., and was, after an interval, moved eastwards, and finally came to rest between posts Nos. IV.

A month after the first addition of alum at the rack the water had immensely improved in colour and odour, and when the experiments were stopped two months from the commencement, the water was in every respect equal to that supplied to this city. The experiments consumed about 60 tons of alum cake, but it must be remembered

that much of this was expended on the over-cautious addition at the west end of the lake.

Analysis of the Treated Water, June 1896.

Total solids		6·79 grains per gallon.
Volatile residue	1·61	
Saline „	5·18	
Free ammonia		0·0063 „ „
Albuminoid ammonia		0·0077 „ „
Nitrites		None.
Nitrates		Faint trace.
Combined chlorine		0·74 grains per gallon.
Total hardness	3·6°	
Temporary	0·5	
Permanent	3·1	
Iron		None.
Phosphates		None.

The water had a neutral reaction, but when boiled was faintly alkaline. It was free from odour when heated to 100° F. Viewed through a 2-ft. tube, it showed but a trace of yellow colour, and had a bright, clear, and colourless appearance when seen in bulk.

Now, it may be asked: Has the water gone back in colour? or is it still as colourless as when the experiments were considered at a end, about the end of May last? In reply, I must say that the water has not maintained its colour in the reservoir. It will be remembered that the spring of this year was a remarkably dry one, it was then the treatment began, and during its continuance scarcely any rain fell; the consequence was the Forrest Burn flowed into the lake nearly colourless, and there was no washing of the peaty banks of the gathering ground by heavy downpours.

I was not then aware that the Forrest Burn passed through a district accessible to drainage from peat. When the rain set in the burn came down highly charged with peat, and naturally had a serious effect on the colour of the whole reservoir.

The remedy, however, lies with the local authorities. If they are content with the water as it now is, which is undoubtedly much improved in colour, it is needless to go to any further expense: if, however, they want a clear and colourless supply, it is only necessary to resume and continue the alum treatment by the raft system.

As to the cost of the purification process, it was ascertained by experiment that 3½ grains per 1,000 galls. threw all the peaty matter out of solution—this is equivalent to 1 lb. for every 2,000 galls. treated,—and as one million galls. are daily drawn off, the quantity of alum cake required would be 500 lb. or, say, 5 cwt. per day. This quantity would cost about 7*s*. 6*d*., or, with occasional attendance, say, 10*s*. a day. In summer weather, or rather at periods when there is a low rainfall, the treatment could be suspended altogether.

I estimate that a cost of about 150*l*. a year would suffice to effect this result.

In regard to the action of the water on lead, no experiments were made at the time: this was because of the fact of water being almost entirely supplied to the villages by means of stand-wells in the streets. Very few houses had the water introduced; hence no lead pipes. Since then, however, we have made a number of experiments on the point, and have found that the reservoir water has certainly more action on lead than Edinburgh water; but what is satisfactory is that the treated water has only about half the solvent power of the untreated water.

Discussion.

Mr. Laing remarked that the simplicity and the economy of the process which Dr. Readman had applied in his experiments should lead to its general adoption, as a glance at the specimens of the water and the results of analysis, before and after treatment, showed that the author had successfully solved the problem on a large scale. He expressed his regret at hearing that the water had not remained colourless, due to the influx of peaty water; and he suggested that the water might be subjected to a purifying process before reaching the reservoir. A spent shale bing would form a capital filter, and would supply the alum perhaps, but he feared the water would dissolve out other salts, such as iron, &c., which it would not be advisable to have present.

Mr. Gray asked the author whether the purified water contained alum, and if not, how this was avoided. He further asked if Dr. Readman was of the opinion that the water would have remained clear in the absence of any influx of peaty matter, and whether the object he had in view in carrying out the process was not only to purify the water in the reservoir, but also to protect the underlying peat, by means of the alumina-lake deposited in it, from the solvent action of the water. He also asked if the colour of the water after treatment had developed slowly, or if there had been any sudden accession of colour after heavy rainfalls, as an answer to these questions might give some information as to the efficiency of the alumina-lake in retarding or preventing further contamination.

Dr. Readman stated that the water was free from alum, and that the treatment was stopped as soon as the organic matter was carried down; excess of alum was thus avoided. His object in applying the process was not only to purify the water in the reservoir, but also, by the aid of subsequent treatment, to provide a protecting layer to prevent solution of colouring matter from the peat. He was misled at the time of the experiments by his understanding that the Forrest Burn was not accessible to peaty drainage; this view was apparently confirmed by the appearance of the water, which was then clear and colourless. He was not able to say whether the colour of the present water had developed slowly or quickly, as he had not visited the reservoir since the treatment was stopped.

Obituary.

JOSEPH D. WEEKS.

Member of the Society of Chemical Industry.
Of the United States Geological Survey.
Formerly President of the American Institute of Mining Engineers and Metallurgists.
Editor of the *American Manufacturer*, &c.

Those members of the Society of Chemical Industry who attended the Annual General Meeting of 1895, held in Leeds, and were present at the annual dinner of the Society, will doubtless still retain in memory a more or less vivid impression of the eloquent words of him, now the subject of this brief notice, though then the welcomed guest of the Society. It seems truly but a brief space which intervenes between that welcome and this adieu. Mr. Joseph D. Weeks was stricken with apoplexy on December 26th, 1896, and died within a few hours after the seizure at Pittsburg, Pennsylvania. He was born in Lowell, Mass., in 1840, and, after completing a scientific course of study at Wesleyan University, Middletown, Conn., he entered the ministry. After the war he proceeded to Cincinnati, and subsequently became a newspaper writer. In 1872 he went to Pittsburg, and was connected with the *American Manufacturer*, with which the *Iron World* was soon afterwards united. In 1875, becoming associate editor of the *Iron Age*, during the 10 succeeding years he had charge of the Western department of that journal. In 1883 he became secretary of the Western Pig Iron Association, which office he continued to hold up to the time of his decease. In 1886 he assumed the control of the *American Manufacturer*. For the United States Census of 1880, Mr. Weeks prepared the details and figures entitled "The Manufacture of Coke" and "The Manufacture of Glass." In addition to this he prepared the entire Volume XX. of the United States Census of 1880, entitled "Statistics of Wages in Manufacturing Industries, with Supplemental Report on Strikes, Lock-outs, and Trade Societies."

In 1885 he began his work for the United States Geological Survey, making reports yearly on the manufacture of coke, petroleum, natural gas, and the production of manganese. For the Census of 1890 he prepared three reports—Crude Petroleum, Natural Gas, and

Manganese; and in the Department of Manufactures three reports—Coke, Glass, and a work on Petroleum Refining, the latter being the first of its kind ever published by the Government. For many years Mr. Weeks was a member of the American Institute of Mining Engineers, and was President of that body during 1895.

It is with much regret we have within two months to bid adieu to two distinguished members of our Society in the United States, Mr. Alfred H. Mason and Mr. Joseph D. Weeks: and in stretching out the hand of fraternal sympathy to our American Section and fellow-members, probably no more fitting words can be found with which to close this notice, than those with which Mr. Weeks opened his speech at Leeds on the occasion already referred to:—

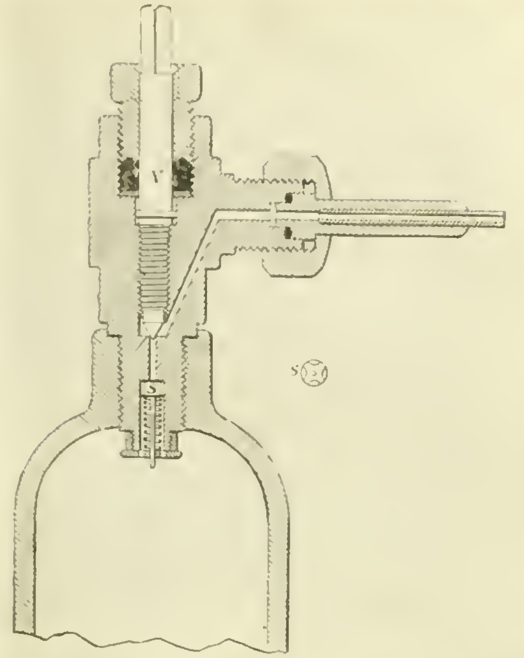
"We speak the same language, we have the same thoughts, we have in many respects the same history, we have the same hopes, we have the same aspirations, and I think that in the future, as humanity marches onward and seeks higher aims and cherishes loftier aspirations, the Americans and the English will be found side by side through all the centuries."—W. S.

Journal and Patent* Literature.

I.—PLANT, APPARATUS, AND MACHINERY.

Valve for Cylinders containing Compressed or Liquefied Gases. E. Ducretet and L. Lejeune. Comptes rend. 1896, **123**, 810—811.

The valve recommended by the authors is shown in the accompanying figure. The function of the new valve is to prevent the rapid and explosive exit of the compressed gas when the plunger V is suddenly unscrewed. This is accomplished by the introduction of a valve S, which has an adjustable permanent opening, thus preventing the violent escape of gas, whilst it does not interfere with the rapid charging of the cylinder.



—J. S.

PATENTS.

Centrifugal Machines, Impts. in, for Drying Sugar and other Granular Substances. W. P. Abell, Essequibo. Eng. Pat. 22,900, Nov. 29, 1895.

See under XVI., *page* 56.

Meters [Distillery] for Measuring Spirits and Distillates, Impts. in. C. F. Chapp, Basse Terre, Guadeloupe. Eng. Pat. 23,849, Dec. 13, 1895.

An apparatus for recording the total weight and average alcoholic strength of the produce from a still. It comprises a scoop-shaped scale-pan, overhanging and forming part of which, is a trough-shaped chute, into which the spirit from the still is discharged by a pipe ending in a ⊤ head, thence overflowing into the scale-pan. The latter, being suitably counterpoised, oversets when loaded to a predetermined degree (with 1—10 litres) and discharges its contents; and in order that at this stage the overflow from the chute may cease, the trough is made shallow at the discharge end, but deep elsewhere, and is thus able to hold and retain the liquid coming from the still-pipe until the pan has resumed its normal position. The liquid discharged from the pan flows into and completely fills a small tank fitted with two siphons—a large one, which transfers the bulk of the spirit to the storage vessel, and a smaller one, which serves to take a "sample" of the liquid. The consecutive samples are, of course, collected in a separate vessel, and, united, form the *average* sample. The number of fillings of the scale-pan is recorded by a counting machine actuated by a float situated in the above-mentioned tank. Two testing chambers are provided at the inlet and outlet ends, respectively, of the meter, fitted with draw-off cocks, thermometers, alcoholometers, &c.—H. T. P.

Filtering Liquids and Impregnating the same with Gases, Impts. in Apparatus for. [Impregnating Filter.] W. Haydt, Stuttgart, Germany. Eng. Pat. 24,835, Dec. 27, 1895.

"An impregnating filter, such, that during the forcing through of the liquid, a continuous admission of the gas under pressure takes place into an ante-chamber. This is in direct communication with the filter chamber, and is traversed by the liquid in a finely divided state, on its way to the filtering receptacle. The purpose of this is to effect both the highest possible degree of impregnation as well as a rapid and thorough filtration."

* Any of these specifications may be obtained by post by remitting 8d.—the price now fixed for all specifications, postage included—to Sir Henry Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

Apparatus for bringing to a desired Temperature Articles of all kinds, more particularly Wines; An Improved. C. Fischer, Hamburg. Eng. Pat. 20,869, Sept. 21, 1896.

A DOUBLE-WALLED, bucket-shaped vessel fitted with a lid (also jacketed) and provided with a false bottom carrying a central tube of perforated metal. The bottles of wine, &c. are placed in the annular space round the central tube, and the latter is charged with ice, or a flame is maintained in it, according to requirements.—H. T. P.

Filters for Wines, Spirits, and other Liquids; Impts. in. W. Elze, Köstritz Thüringen, Germany. Eng. Pat. 21,010, Sept. 22, 1896.

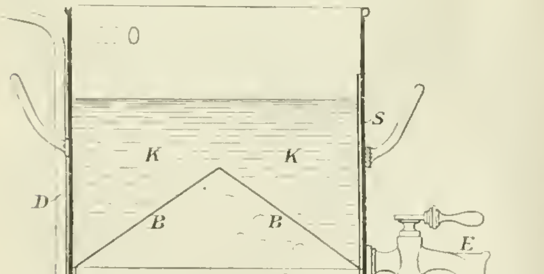
THE filter consists of a cylinder mounted on trunnions, and the filtering material, which consists of cellulose pulp, is placed between two perforated plates fixed at one end of the cylinder.—A. L. S.

Apparatus for Heating [Sterilising] and Cooling Liquids, Impts. in. W. F. E. Casse, Copenhagen. Eng. Pat. 22,060, Oct. 5, 1896.

THE sterilising apparatus claimed is intended for continuous work, and consists of a system of parallel jacketed tubes, connected in series. The outer jackets are divided into two sets, traversed by hot and cold water respectively, whilst the liquid to be sterilised flows continuously through the inner tubes, but in an opposite direction. The inner tubes are so connected as to be readily accessible for cleaning purposes.—H. T. P.

Distilling Apparatus, An Improved. S. S. Bromhead, London. From C. Dicker, Brussels. Eng. Pat. 20,292, Oct. 28, 1895.

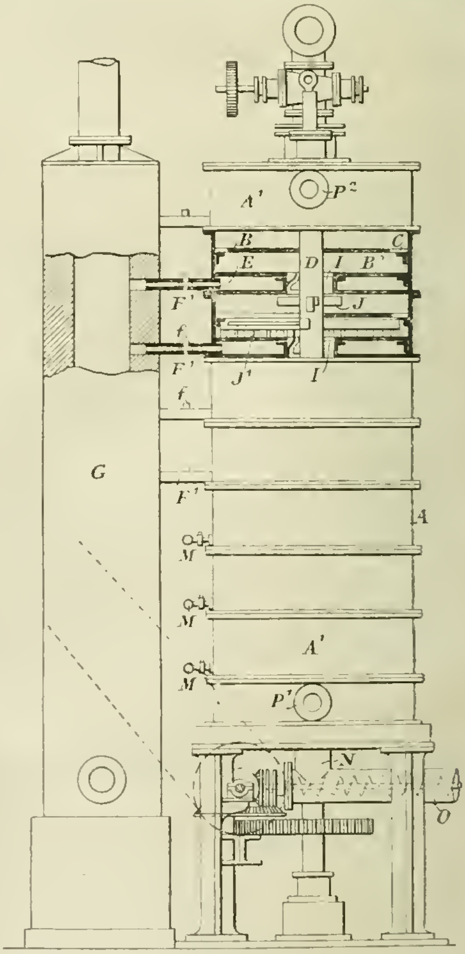
THE distilling apparatus consists of two cylindrical casings fitting one above the other, the lower of which, A, contains the liquid to be distilled, and is heated by a fire, while the upper, K, acts as a cooler. The latter is provided with a conical bottom, B, and the condensed water flowing down on the inner side of this is caught in an annular hollow dripping channel, R, and is led away through a tube, C.



The water for use in the cooler is led by a pipe, D, to the interior of the dripping channel, and thence passes upwards through an opening (not shown) to the mass of water above the conical bottom, finally overflowing through the vertical outlet channel, S, to the cock E. This water may be caught in a funnel, E', and be led to the lower vessel, thus acting as an automatic feed thereto. For obtaining alcohol, ether, essences, extracts, and the like, the upper and lower vessels are screwed together, and the lower vessel has suspended therein a water-bath, G, containing the alcohol or ether, upon which may be placed a sieve, H. By the use of this arrangement, "any desired extracts and essences can be prepared by means of alcohol, ether, &c." In a modification, the upper cooling vessel may be formed as a double cone, and the cooling liquid therefrom removed by means of a pipe from the apex, and led to a special device for regulating the level of the liquid in the lower or distilling vessel.—R. S.

Solid and Pasty Substances, Apparatus for Treating with Gases at Elevated Temperatures. L. Mond, Regent's Park, London. Eng. Pat. 23,665A, Dec. 10, 1895.

THE apparatus consists of an upright chamber, A, divided by trays or partitions, B B', into a number of sets of superposed compartments, A', each set consisting of a heating compartment, E, and two reacting compartments. Each heating compartment is connected by a flue, F', having a valve, *f*, to a combustion chamber, G, for gaseous fuel, and is provided with a vertical partition, so that the said gases must pass completely around the compartment before passing away through a second flue, similar to and placed behind F', to the chimney shaft. By means of the valves *f* each compartment may be independently heated. The



reacting compartments are chambers down which the solid material is caused to pass by rotary stirrers or scrapers, J J', of which the stirrers J, acting on the trays B, tend to move the material from the centre to the periphery, where it falls through the openings C to the tray B' below. The stirrers J' on this tray move the material from the periphery towards the centre, so that it falls through the opening I on to the next tray below. The gases enter at P' and pass

upwards through all the reacting compartments, finally escaping through the outlet P². The treated product is discharged through a pipe, N, into a conveying worm, O, or directly into receptacles. The lower reacting compartments may be cooled instead of heated, by passing cold air or water through the branch pipes and valves M into the compartments E.—R. S.

Wet Steam, Method of Highly Superheating. [*Drying and Superheating.*] W. Schmidt, Ballenstedt-on-Harz, German Empire. Eng. Pat. 1520, Jan. 22, 1896.

The method consists in first letting the wet steam pass through a coil, wherein it is dried, or dried and slightly superheated, by the coolest portion of the waste gases of a furnace, whilst flowing in a direction opposite to that of the said gases, and then conducting the dried steam into a second coil, wherein it is properly superheated by the hottest portion of the waste gases whilst flowing in the same direction. In the apparatus described, the two coils encircle a flue through which the waste gases pass, the first coil being arranged above the second, and connected at its lower end to the lower end of the latter, so that steam passed downward through the first coil will pass upward through the second.—R. A.

II.—FUEL, GAS, AND LIGHT.

Coal, The Supposed Ontario. A. McCharles. Eng. and Mining J. 1896, **62**, [22], 512.

It was at first supposed that a discovery of coal had been made in Ontario. The mineral now proves to be merely "anthraxolite," which is of little economic value, except for use on the spot as fuel. An analysis of the mineral given in the Government Report by Prof. Coleman shows the following results:—Hygroscopic moisture, 2·67 per cent.; volatile organic matter, 4·78 per cent.; fixed carbon, 55·85 per cent.; sulphur 1·06 per cent.; ash, 36·5 per cent.; total, 100·86 per cent. Other assays show varying amounts of ash, but none less than 20 per cent. The substance in its unexposed state is very black and lustrous, and readily breaks up by hand into thin layers and small cubes. It burns well with a short reddish flame when started with wood, and gives a good heat, leaving an ash resembling reddish sand. If the draught is not very strong, the lumps burn only on the outside. It is stated to have been successfully used in forging iron. The above report refers to the deposit 17 miles west of Sudbury, and there is now stated to be another large deposit of the same material 30 miles east of that town.
—A. W.

Thiocyanides from Spent Gas-purifying Materials. V. Hölbling. Mitt. tech. Gewerbe-Museums in Wien, 1896, 245—253.

Marasse proposed (Ger. Pat. 28,137, 1882) to obtain calcium thiocyanide by heating the residue, after extraction of the spent material with water to remove the soluble ammonium salts,—with lime and water in closed vessels at 100° C. Marasse believed that calcium ferro-cyanide and calcium sulphide were first formed, and that these then reacted on one another, forming calcium thiocyanide and iron sulphide.

The author finds that higher temperatures are necessary for the formation of calcium thiocyanide, and that the reaction is not so simple as Marasse believed. The reaction is probably most nearly expressed by the following equations, the formula $Fe(CN)_2$ usually assumed for the cyanide present in the residues in question being adopted:—

1. $3Fe(CN)_2 + 3S + 3CaO = Ca_2Fe(CN)_6 + CaSO_3 + 2FeS.$

2. $Ca_2Fe(CN)_6 + CaSO_3 + CaO + 7S = 3Ca(CNS)_2 + CaSO_4 + FeS.$

According to these equations, the proportions of the Prussian blue and sulphur entering into reaction are $3Fe(CN)_2:10S$, or, practically, equal weights. But in the spent material the blue is only about 5 per cent. or less, whilst the sulphur is about 25 per cent.; and the excess of sulphur is lost as iron sulphide. Some, at least, of this loss can be obviated by adding to the spent material the waste blue ("Hell-blau," or mixture of Prussian blue and gypsum), generally thrown away as a waste product in the preparation of Prussian blue and yellow prussiate from spent gas-purifying materials.

Attempts to obtain the cyanide in the more commercially useful form of alkaline thiocyanide by replacing the lime by caustic alkali (as suggested by Marasse), or by alkaline carbonates, were not successful enough to be commercially valuable. With the caustic alkalis the cost would be prohibitive, and it was also found very difficult to filter off the solution containing potassium sulphides from the residual iron sulphide. With alkaline carbonates (potash was used) heating for 12 hours under 3 atmospheres pressure was necessary, and even then about 30 per cent. of the blue remained unacted on. Complete decomposition of the blue could not be effected. It is just possible that this carbonate process might be used in works where yellow prussiate is made, the undecomposed blue being worked up for the prussiate.

Attempts to employ ammonia led to no results likely to prove commercially applicable.—L. T. T.

Ethylene and Benzene, Estimation of. F. Haber and H. Oechelhäuser. Ber. 1896, **29**, 2700.

See under XXIII., *page* 67.

Acetylene Regulations in Paris. J. Gas Lighting, Dec. 22, 1896, 1230.

See under Trade Rep., *page* 73.

PATENTS.

[*Illuminating Gas.*] *Production of Gases from Hydrocarbon Oils, Impts. in the.* R. E. Middleton, London. Eng. Pat. 15,254, Aug. 14, 1895.

The oils are placed in a closed vessel, which contains carbons suitable for producing one or more electric arcs on sending a current through. The oils are thereby vaporised; they are then washed, and led to a gas-holder. The gases may be mixed with ordinary coal-gas or other gases. The residuum of the oils may be used as fuel.—R. S.

Products of Combustion, A New or Improved Apparatus [*Combination with Two Gas-meters*] *for Estimating the Carbonic Acid or other Constituents in Gases or.* G. Craig, Cumnock, Ayrshire, N.B. Eng. Pat. 22,303, Nov. 22, 1895.

See under XXIII., *page* 65.

Earth Metals, Impts. in the Manufacture or Production Electrolytically of Coatings composed of Oxides of the. [*Mantles.*] R. Langhans, Berlin, Germany. Eng. Pat. 23,137, Dec. 3, 1895.

In this process an electrolytic coating of a hydroxide of the earth metals is formed on a conducting foundation, and is subsequently converted into the oxide by calcination. The deposition is preferably effected in a cylindrical vessel containing a diaphragm forming or being lined with an insoluble electrode, within which is a saturated solution formed by dissolving in a concentrated solution of a neutral earth salt, such as the sulphate or nitrate, earthy hydrates precipitated in the cold, and well washed, while between the diaphragm and the outer vessel is a solution of the hydrated oxide or carbonate of the same earth metal. A thin platinum cylinder forms the anode, and the cathode is the body to be coated—say, an incandescent mantle formed of a gauze cone of platinum, gold, carbon, or other suitable conductor. When sufficiently coated, the mantle is removed from the bath, washed, dried in air free from carbonic acid, and calcined by gradually raising the temperature. The minimum current density per square centimetre for 20 per cent. solutions of the salts of zirconium and thorium is 1·66 ampères, of yttrium 1·25 ampères, and of aluminium 0·75 ampère, but double this current density is preferably employed. To render the deposit less dense, the solution of the basic salt may be prepared by adding ammonia to the aqueous solution of the neutral salt until

the hydroxide which separates out is redissolved; and this is continued as long as re-solution occurs. Instead of ammonia, basic derivatives, such as amines and the like, as also sodium and potassium hydroxides, may be used. Where a thick coating is required, an alcoholic solution of the basic earth salt—say, nitrate of thorium—is preferably employed, in conjunction with an alcoholic solution of ammonia. To prevent cracking, the foundation may be covered with a netting or winding of combustible fibres, such as cotton, silk, or the like, which will be destroyed during calcination, leaving the oxide coating porous; or the hydroxide deposit may be converted, previous to calcining, into organic or inorganic salts, preferably the latter, which will yield oxides on heating.—G. H. R.

Earthy Oxides, Impts. in the Manufacture or Production Electrolytically of Coatings composed of. [*Incandescent Mantles.*] R. Langhans, Berlin, Germany. Eng. Pat. 24,546, Dec. 21, 1895.

The mantle constructed of an electro-conductive material, such as platinum, gold, or carbon, forms the cathode in a bath composed of a dilute solution of, say, 3 per cent. of an earthy hydroxide, soluble in water, such as thorium. An insoluble anode is employed, and the current density is about 3 ampères per 100 sq. cm. Directions are given for the preparation of the hydroxides of thorium, and in the term earthy hydroxide are included the hydroxides and the oxides of both the earth metals proper and the earth alkali metals. When the mantle is sufficiently impregnated, it is removed from the bath and calcined, and in order to prevent the coating cracking during calcination, and to render it porous, there is dissolved in the bath an organic base or alkaloid, which will be deposited jointly with the hydroxides of the earth metal on the negative electrode, and be subsequently destroyed on the mantle being calcined. Instead of dissolving opium, morphine, bases of the veratrine class, &c., in the bath, the mantle may be covered with combustible fibres previous to electrolysing, as in Eng. Pat. 23,137, 1895 (see previous abstract); or the hydroxide deposited may be transformed before drying and calcining into a salt capable of parting with its acid under the action of heat.—G. H. R.

Gas, Combustible: Impts. in the Manufacture of. [*Use of Refuse.*] L. Denayrouze, Neuilly (Seine), France. Eng. Pat. 23,856, Dec. 12, 1895.

A cylindrical vessel or retort is used for making combustible gas by the distillation of waste material, such as waste fatty matters, residues from petroleum distillation, tar, sewage matters, fæces, &c.—L. T. T.

Incandescent Lamp for the Combustion of Paraffin Oil and other Liquids. A. M. Clark, London. From A. Meyenberg, M. Wendorf, and S. Henlein, Frankfort-on-the-Maine. Eng. Pat. 23,863, Dec. 12, 1895.

The reservoir of the lamp consists of two chambers, the inner one containing benzoline, and the outer one, which completely surrounds the inner chamber, containing paraffin oil. Air under pressure can be supplied to both chambers from an india-rubber ball (situated in the hollow base of the lamp), which is inflated as often as required by means of a compressible india-rubber bulb situated on the outside of the lamp. The vaporising chamber to which the paraffin oil is raised from the reservoir by means of the compressed air is situated within the burner mouth-piece and the mantle support, and by leading the vapour produced down a small pipe which is bent upwards at its lower extremity, the upward rush of gas induces an indraught of air through holes provided round the surrounding burner-tube, in the usual way. The mixture of vapour and air then burns at the burner mouth-piece, which is provided with a conical cap of wire gauze as usual.

The benzoline is used merely to start the lamp. Air from the india-rubber ball is saturated with the benzoline, ascends by a small pipe to the burner mouth-piece, and is there allowed to burn long enough to so far heat the vaporising chamber that the paraffin oil can give a regular supply of gas. By turning a two-way cock, the paraffin vapour reaches the burner just before the air saturated with benzoline, is turned off, so that the benzoline flame ignites the paraffin vapour, and, on the other hand, when extinguishing the lamp, the benzoline conduit remains open for a brief interval after the closing of the paraffin conduit, so that the paraffin vapour generated after the stoppage of the paraffin oil supply, may be consumed. To facilitate the use of the two-way cock, a pointer is fastened to its outer end at right angles to its length and moves over a graduated scale.—H. B.

Incandescent Gas Lighting, Impts. in the Preparation and Production of Bodies ["*Lucium*"] *for Use in what is known as.* P. Barrière, Paris. Eng. Pat. 24,008, Dec. 14, 1895.

The patentee claims to have discovered in monazite sand a new body which he calls "Lucium," to be used in making mantles. The sand is fused with sodium carbonate to convert the metallic oxides into insoluble carbonates, lixiviated with water, the insoluble matter slowly calcined with sulphuric acid, the sulphates dissolved in cold water and precipitated by ammonia. The hydrates are dissolved in HCl, the solution neutralised, and precipitated with oxalic acid. The precipitated oxalates are treated with sulphuric acid, of which the excess is expelled, the sulphates are dissolved in cold water and again precipitated by ammonia. The gelatinous precipitate is filtered off, washed, and dissolved with sulphuric acid, the solution being allowed to stand from five to six hours, after addition of saturated sodium sulphate solution to precipitate Ce, La, and Di. After filtering, the solution is again precipitated by ammonia and the precipitate converted into sulphate, when the thorium is precipitated by potassium sulphate. The solution is filtered, and the "Lucium" precipitated by heating to boiling after adding concentrated sodium hyposulphite (thiosulphate) solution. The precipitate is washed, dissolved in hydrochloric acid, and the new body precipitated by means of ammonia, filtered off, and washed. It is then converted into nitrate and mixed with some nitrate of zinc solution. The patentee claims for use in incandescent gas lighting the new body, either alone or in combination with oxide of zinc or other body "capable of imparting power of radiation," and also claims the above-described process of purification of the body. (See Chem. News, **74**, 212, 259, and 269.)—H. B.

Charging Air with Combustible Matter [*Carburetting Air*] *and applying it for Illuminating, Impts. in the Method of and Apparatus for.* L. Denayrouze, Neuilly, France. Eng. Pat. 24,381, Dec. 19, 1895.

Air under a pressure represented by 30 to 40 ins. of water is sent through the mains and mixed, at the burner itself, with (*a*) a volatile liquid hydrocarbon, (*b*) a solid hydrocarbon, or (*c*) combustible dust.

(*a*.) The liquid is withdrawn by the outlet of the air passing over the outlet of a tube leading from the liquid receiver beneath the burner, and the spray and air are mixed by a fan before passing to the burner. (*b*.) The solid, such as naphthalene, is contained in a receptacle above the burner, and the air blown over its surface. The mixture passes to a chamber containing a rotating fan, where it is mixed with more air, and thence passes to the burner. (*c*.) The powder receptacle has a small feed roller at the bottom, which delivers a small regular quantity of powder, which is driven by the air jet to the mixing chamber and thence to the burner.—R. S.

Mantles for Incandescence Gas Lighting, Impts. in. A. and W. S. Taylor, Glasgow. Eng. Pat. 24,505, Dec. 21, 1895.

The cotton mantle is washed in dilute solution of nitric acid or ammonium nitrate and then immersed in a "strong solution in 'water of oxide,' nitrate, or sulphate of aluminium, ammonium bichromate, and zinc nitrate," in the proportion of about 70, 20, and 10 per cent. respectively. The aluminium salts may be replaced by beryllium salts, the bichromate by titanium salts, and the zinc nitrate by manganese nitrate. The mantle may then be dusted over with powdered sulphur or gunpowder and burned.—H. B.

Glow Bodies for Incandescent Lamps [*Rhodium Oxide*]. *Composition to be used in the Manufacture of.* G. Lowenberg, Berlin. Eng. Pat. 21,267, Sept. 25, 1896.

In thoria-ceria mantles the introduction of a little "oxide of rhodium" permits a considerable reduction of the amount of ceria required. Thus a mantle containing thoria, 98·8 per cent.; ceria, 1 per cent.; and "oxide of rhodium," 0·2 per cent., emits as much light as one composed of thoria, 98 per cent., and ceria, 2 per cent. The claim is for a composition containing oxides of thorium and cerium and "one or more of the oxides of the gold group, as palladium, iridium, osmium, ruthenium, rhodium, and the like."

—H. B.

Incandescent Gas or Oil Lighting [*Incandescent Glass Chimneys*], *Impts. in.* N. F. M. Wilsmore, Hampstead. From D. Little, Melbourne, Australia. Eng. Pat. 1521, Jan. 22, 1896.

Any earths, minerals, &c., capable of incandescing under the action of heat are to be brought into minute subdivision and then incorporated with the substance of ordinary glass chimneys, so as to become incandescent when heated by an ordinary oil lamp or burner.—H. B.

Lamps for Burning Hydrocarbon Oil or Spirit, Impts. in or connected with. R. Kiesow, London. From A. Kiesow, Berlin. Eng. Pat. 1507, Jan. 21, 1896.

In order to avert the danger of the accumulation in the lamp reservoir of an explosive vapour which might be ignited by the flame of the lamp, the patentee traps off the vapour by employing as a receptacle for the wick a descending tube, which at its upper end is hermetically fixed into the reservoir collar into which the burner is screwed. The tube passes downwards to within a short distance of the bottom of the reservoir, where it is spread out in the form of an inverted saucer, to meet the walls or bottom of the reservoir. The tubular wick chamber thus formed is provided at its lowest part with holes which allow the oil to flow into it from the reservoir; as these holes are always sealed by the oil in the lamp, no vapours from the outside reservoir can pass into the tube and so reach the burner. The supply of air to the reservoir, necessary to permit the proper flow of oil to the wick, enters through a hole in the filler-cap, and to prevent the outflow of the oil in the event of the lamp being upset, the cap is formed like a cylindrical box divided into two compartments communicating with each other, with the outside air, and with the reservoir by means of minute apertures.

—H. B.

Obtaining Light [*Liquid or Gaseous Hydrocarbon Lamps*], *Impts. in Means for.* F. Tatham, Lewisham Hill. Eng. Pat. 14,009, June 24, 1896.

Consists in supplying oxygen under pressure at the point where the hydrocarbon is to be ignited and burned. Three forms are shown: (*a*) where ordinary paraffin or like oil is used, (*b*) where a heavy liquid hydrocarbon is used, and (*c*) where a gas is employed. The flame in each case may impinge upon a pencil, pencils, or a plate of refractory incandescent material. In *a* the oil is drawn up by a wick, in *b* by the oxygen used with a sprayer nozzle. In the case of *a*, the wick tubes are cooled by water, and a water-circulating arrangement is shown and described.—R. S.

Gas, Appliances for increasing the Illuminating Power of, under Combustion. R. Lavender and W. Tice, both of London. Eng. Pat. 5336, March 10, 1896.

These consist in placing just over or on the burner a disc or cap (or both) of metal or other suitable material in such a position as to direct the current of air into the blue portion at the base of the flame. With Argand burners an inner disc is used, and also an outer case turned inwards, so that the inner air column is diverted against the inner surface of the blue zone, the outer air column against the outer surface of the same zone.—L. T. T.

Air Carburetter, A New System of. A. Peugeot, Valentigney, France. Eng. Pat. 14,213, June 26, 1896. Date claimed under International Convention, April 13, 1896.

Several forms of an air carburetter intended for supplying hydrocarbon gas engines are shown, each consisting essentially of one or more capillary diaphragms dipping to a constant depth in the liquid hydrocarbon, and through, round, or over which the air to be carburetted is caused to pass. The diaphragms may be cylindrical, vertical, spiral, or of other forms, and are made of such materials as hair wrapped upon wire cloth, ramie, silk, cotton, tampico, piassava, hemp, wadding, felt, ground wood, or fabrics.

—R. S.

Acetylene, Impts. in the Manufacture of, and in Apparatus therefor. R. P. Pictet, Geneva. Eng. Pat. 18,207, Aug. 17, 1896.

Many of the impurities present in acetylene made from calcium carbide are due to the rise of temperature produced by the violent reaction which takes place when the carbide comes in contact with the water, some of the acetylene being partially decomposed. The presence of air in the acetylene is also prejudicial.

The patentee proposes to keep the water used for decomposing the carbide at a temperature of about 20° C. (the limits of − 45° C. and + 60° C. must not be exceeded) by means of a water jacket outside, and coils for circulating water, brine, &c. at any suitable temperature inside the vessel containing the said water. A large bulk of water is used. The carbide of calcium is employed in small pieces, and is so fed into the water by means of hoppers or otherwise, that the adhering film of air is detached by the water (and can escape by the hopper) before the carbide falls into the enclosed portion of the water vessel.—L. T. T.

Acetylene, Impts. in the Purification of, and in Apparatus therefor. R. P. Pictet, Geneva. Eng. Pat. 18,208, Aug. 17, 1896.

Acetylene made from calcium carbide contains more or less of the following impurities: ammonia, hydrogen, sulphuretted, arseniuretted, and carburetted hydrogen, and carbonic oxide. These are the cause of the acetylene attacking copper and other metals and forming compounds which are dangerous and sometimes detonating, and they also render the acetylene flame less luminous. The present process for removing the detrimental impurities consists in: (1) Bubbling the gas through a concentrated solution of calcium chloride cooled to a temperature between − 20° and − 40° C. (2) Passing the gas through 40 per cent. sulphuric acid at a temperature between − 20° and − 60° C. (3) Washing the gas with a solution of lead salts at the ordinary temperature. (4) Drying the gas over chloride of calcium. "As is well known chemical reactions entirely cease at low temperatures and there is a selective order in which the reactions progressively diminish." The low temperatures are employed to prevent the action of the reagent on the acetylene itself, and are chosen so as to utilise that modifying action of temperature on the reactions. Other reagents may also be used. They are placed in vessels so arranged as to allow of suitable cooling.—L. T. T.

Heating the Air to be burned in Lamps, Contrivance for. [*Concentric Chimneys.*] H. Niemeyer, Cassel, Germany. Eng. Pat. 20,552, Sept. 17, 1896.

The lamps are provided with a concentric arrangement of chimneys, entirely or partly of glass, so that the air for combustion passes first upwards from the outer air, then downwards through an annular channel, and lastly upwards in the central or combustion chamber. By this arrangement the air supply is heated before use. The amount of air can be regulated by the vertical adjustment of the glasses to each other by means of a screw.—R. S.

Calcium Carbide or Analogous Compounds, Impts. in and relating to, and the Treatment of the same to form a Uniform Composition. [Tablets.] J. A. Deuther, Boston, U.S.A. Eng. Pat. 20,598, Sept. 17, 1896.

In practical operation it is found, that the product resulting from the processes now used to produce calcium carbide is

not uniform, and therefore not capable of yielding the same determined amount of gas. In the present invention the calcium carbide is pulverised and thoroughly mixed. It is then treated with a binding material, which may be either a solid or a liquid, and compressed into tablets of a certain weight, so that each tablet represents by decomposition a definite amount of gas.—D. B.

Carbide of Calcium [Protective Coating], An Improved Process for the Treatment of. M. P. E. Létang, Paris. Eng. Pat. 21,572. Sept. 29, 1896.

To preserve calcium carbide from the influence of moisture or of steam, and to dissolve the lime (resulting from the decomposition of the carbide) or to render the same at least "liquid" and thereby to promote its automatic elimination, it is proposed to cover the pieces of carbide with a coating consisting of 55 parts of glucose, 5 parts of petroleum, and 40 parts of carbonate of lime in powder, or similar and effective materials.—D. B.

Acetylene Gas, Improved Apparatus for the Automatic Production of. R. Haddan, London. Eng. Pat. 23,812, Oct. 26, 1896.

In this apparatus for the production of acetylene, the crushed calcium carbide falls in small portions into a vessel of water, this fall being regulated by the rising and falling of the bell of the gas holder, by means of a rod which carries two cross rods actuating a lever which operates a spring valve in the bottom of the receiver containing the crushed carbide.—L. T. T.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Mesitylene from Acetone. V. Meyer and W. Molz. Ber. 1896, **29**, 2831—2833.

It has recently been shown by Meyer and Pavia that trialkylbenzenes containing the substituents in symmetrical position, take up two acetyl residues when subjected to the Friedel-Crafts reaction (acetylation in presence of aluminium chloride), whilst benzene and its alkyl homologues only take up one acetyl residue when similarly treated. This observation affords a means of ascertaining the constitution of hydrocarbons when only small quantities of material are at disposal. The authors have used it for the purpose of solving the pending question as to the constitution of mesitylene obtained from pure acetone, and confirm their former statement that this "mesitylene" is symmetrical trimethylbenzene. They also disprove the presence of hemellitene in mesitylene prepared from pure acetone, as suggested by Hantzsch.—D. B.

Naphthas (Natural), Heavy, and their Treatment. W. Charitschkow. Chem. Rev. Fett- u. Harz-Ind. **3**, 267—269, 286—287.

Large quantities of heavy natural naphthas ("goudron naphta") are found in the North and South Caucasus. As a rule they are rich in bituminous matter, besides containing a large proportion of heavy liquid hydrocarbons. The sample analysed by the author consisted of 85·06 C, 10·54 H, 4·30 O, and 0·01 per cent. of ash. In physical properties, samples from this region agree pretty closely, the sp. gr. ranging from 0·952 to 0·960; viscosity, over 8 minutes (Engler) at 50° C.; flashing point, 135° to 200° C. Very little paraffin is present.

From the fact that the springs are traceable to considerable depths—60 to 200 fathoms—it is believed that we have in these naphthas a separate variety of fluid bitumen, and not an oxidation product of the ordinary light naphthas.

At Geran a spring of heavy naphtha is worked, the refined products comprising a solar oil (sp. gr. 0·88), a valuable cylinder oil (sp. gr. 0·960; viscosity, 900 seconds (Engler) at 50° C.), and various lubricating oils; and the solid pitch residuum (sp. gr. 1·01—1·03) is used for manifold purposes.

The chief difficulty in the manipulation of this naphtha is caused by its considerable hygroscopicity, by the difficulty of separating water from it, as well as mechanical impurities in suspension. At Geran the crude oil is warmed by the condenser coil from the stills to remove part of the water, the remainder being driven off by heating to 100°—110° C. for 48 hours in the still. Ostrejko's steaming apparatus (this Journal, 1896, 26) should be useful for this class of oils.

In testing such oils in the laboratory, freedom from water and solid impurities is an essential preliminary, and this the author secures by dilution with an equal quantity of petroleum spirit (free from water and completely volatile below 100° C.). After 48 hours' contact, the water and solids are removed by filtration through cotton wool or a dry filter, and the solvent driven off at 100° C. This process is also considered highly suitable for the refinery, the diluent being recoverable, as well as for examining all heavy petroleum products; the flashing-point remains unaffected, provided the petroleum spirit is sufficiently pure.

When some only of the physical properties are to be ascertained, the operation may be shortened, the water being driven off—for flashing-point determinations—by heat at 110° to 120° C. in the Pensky-Martens cup. Specific gravity may be found after solution in a lighter oil of known density, or by direct measurement at a temperature of 50° C., at which the naphtha is sufficiently fluid to allow the instrument to sink freely.

Regarding the question of fractional-distillation testing, the author is not in accord with Wischin as to the necessity for assimilating laboratory practice to that of the refinery, the objects in view being different. Instead of the process of measuring off the distillate by a pipette, which is difficult when steam distillation is performed, he prefers to provide the still with a float and index, thus allowing the volume to be read off direct. It is advisable to terminate the operation as soon as the residue has become solid and of a density greater than 1, and as many equal volumes of fractions as possible should be obtained.—C. S.

Wood, Treatment of, with Antiseptics prepared from Petroleum. A. W. Adiasiewietsch. Trudy. bak. otd. imp. russk. Tech. obschtsch. 1896, **11**, 243.

See under IX., *page* 45.

PATENTS.

Petroleum, Crude, and Analogous Oils; Impts. in and Apparatus for Refining, by means of Fuming Sulphuric Acid. J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen. Eng. Pat. 23,436, Dec. 6, 1895.

Recently unsuccessful attempts have been made to use fuming sulphuric acid for the purification of petroleum. One cause of failure is due, with some oils, to the difficulty of securing an intimate admixture with the oil, of the fuming sulphuric acid, as the latter, immediately after contact, rapidly forms pitch-like bodies possessing an inner nucleus of unaltered acid, which either takes no part in the purification, or only acts after prolonged agitation. The principle of the process for overcoming this difficulty consists in mixing in the fuming sulphuric acid at the point of contact of streams of the crude oil (or streams of air and oil), the streams possessing a different velocity, so that considerable friction occurs, whereby the acid is in a sense "rubbed or ground" into a fine state of division and becomes most intimately admixed with the entire body of the oil. Another cause of failure is the fact that when treating some crude oils, especially the heavier fractions, with sulphuric anhydride, besides the ordinary pitch-like compound, which separates out, other similar combinations are formed in some cases, which do not separate from the oil. On washing such oils in the usual way with water and alkali, the impurities are regenerated and impart a dark colour and objectionable odour to the product. The said combinations can by this invention be readily removed by treatment of the oil with a small percentage of ordinary concentrated sulphuric acid. The refined oil obtained by working according to this invention is said to be lighter in colour than that obtained when using either concentrated sulphuric acid alone, or sulphuric anhydride alone, and on exposure to light it continuously becomes

lighter in colour, whilst the product purified with ordinary concentrated acid alone, bleaches a little at first, but afterwards re-assumes a yellow colour. The loss of oil is also said to be less. About half the quantity of acid hitherto employed is used. Sulphuric acid containing the following percentages of anhydride or SO_3 have been found to give good results:—With benzene 20 to 60 per cent. of SO_3, with petroleum 10 to 20 per cent., with lubricating oil 5 to 20 per cent., and with vaseline 25 to 30 per cent. of SO_3.—D. B.

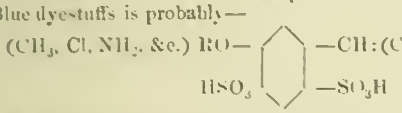
Meters [Distillery] for Measuring Spirits and Distillates, Impts. in. C. F. Chapp, Basse Terre, Guadaloupe. Eng. Pat. 23,849, Dec. 12, 1895.

See under I., *page* 33.

IV.—COLOURING MATTERS AND DYES.

Patent Blue Dyestuffs, Researches on. Part I. Derivatives of Benzylsultone, Benzylsultame, and Benzalsultime. P. Fritsch. Ber. 1896, **29**, 2290—2301.

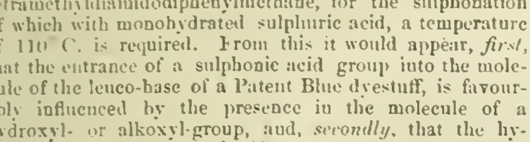
The constitution of the leuco-sulphonic acids of the Patent Blue dyestuffs is probably—



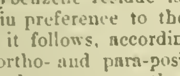
As evidence in support of this, in regard to the positions occupied by the sulphonic acid groups, may be adduced the ease with which *m*-hydroxy- and alkoxy-tetra-alkyldiamidotriphenylmethane compounds are sulphonated at the ordinary temperature, as compared with, for instance, tetramethyldiamidodiphenylmethane, for the sulphonation of which with monohydrated sulphuric acid, a temperature of 110° C. is required. From this it would appear, *first*, that the entrance of a sulphonic acid group into the molecule of the leuco-base of a Patent Blue dyestuff, is favourably influenced by the presence in the molecule of a hydroxyl- or alkoxyl-group, and, *secondly*, that the hydroxybenzene residue is itself attacked by the sulphuric acid in preference to the aniline residues. Such being the case, it follows, according to the law of substitution, that the ortho- and para-positions relative to the hydroxyl- or alkoxyl group are those which are taken up by the sulphonic acid groups.

The circumstance that the dyestuffs of the Patent Blue class possess a green-blue or blue colour may be explained by assuming that the sulphonic acid group, which is in the position ortho to the central carbon atom, has a potent colouring influence. Thus, blue dyestuffs are formed by condensing tetramethyldiamidodiphenylcarbinol with (1) *m*-amidobenzene sulphonic acid (Ger. Pat. 65,017) and subsequently eliminating the amido-group and oxidising, and with (2) *m*-toluenesulphonic acid (Ger. Pat. 80,982), and oxidising the resulting product.

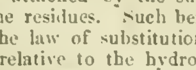
As the blue colour of the aqueous solutions of the dyestuffs in question is not affected by caustic soda in the cold, or by sodium carbonate or ammonia at the boil, it is probable that the sulphonic acid group is in the molecule in the condition of an anhydride of the type—



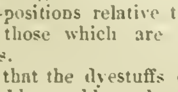
The first of these formulæ being considered the more probable, attempts are being made to prepare the dyestuffs from the anhydrides—



Benzylsultone.



Benzylsultame.



Benzalsultime.

or their derivatives.—E. B.

Cutch, The Preparation of. Indian Pharmacologist, 1896, **1**, 88.

The trees are regarded as mature when about a foot in diameter; they are then felled and cut into blocks two or three feet long. Trees between 25 and 30 years old are regarded as the most suitable. The bark and outer sapwood are generally removed and rejected, the red heartwood is then cut into chips; the branches are utilised in some districts, in others not. The chips are boiled in water for 12 hours; when the water is reduced to one-half, the chips are removed and the liquid boiled down in iron pans, with stirring, until it becomes syrupy; the stirring is continued during cooling until the mass is cold enough to be handled, when it is taken out and spread on leaves in a wooden mould or frame, and left over night. The details of the boiling and evaporating processes vary considerably in different regions; from the widespread conviction of the necessity for stirring or beating the concentrated solution, it might be inferred that an oxidation process is thereby effected. In Baroda the liquid is repeatedly wrung from a blanket and allowed to fall in drops from as great a height as possible. One ton of timber in the round may be taken as yielding 250 to 300 lb. of cutch.—A. C. W.

Madder Colouring Matters, History of. C. Liebermann and S. Friedländer. Ber. 1896, **29**, 2851—2854.

Owing to the difficulty experienced in the isolation of the different constituents present in the colouring matters derived from the madder root, their chemical constitution has for a long time been the subject of much speculation. Runge, in his prize essay on madder and its dyes, published in 1835, expressed a doubt as to the purity of Robiquet and Colin's alizarin discovered in 1826, and of the so-called matière colorante rose (presumably purpurin) described by Gaultier de Claubry and Persoz about the same time. As pure substances Runge mentioned two colouring matters, which he called madder red and madder purple, and from his description of their distinctive properties it is evident that they correspond with the alizarin and purpurin of the present day. The authors have recently had occasion to examine some of the dyed specimens prepared by Runge for the above work. In most cases the fents contained mixtures of both colouring matters. Almost pure purpurin was detected in several patterns, but these were marked madder red. Pure alizarin could not be obtained from any of the specimens, although one marked madder purple was found to be very rich in alizarin. Having found alizarin to predominate in the Turkey-red patterns prepared about 50 years ago by Persoz for his work, entitled "Impression des Tissus," the authors are satisfied that in Runge's specimens the alizarin could not have been oxidised to purpurin. They conclude that although from the description of the distinctive properties of alizarin (madder red) and purpurin (madder purple) it is evident that Runge successfully isolated the two colours on a small scale, their separation in large quantities could not have been effected with the same degree of accuracy. The raw material he had at his disposal being rich in purpurin, the separation with alum followed by Runge could scarcely give satisfactory results even under the most favourable conditions.—D. B.

o- and p-Nitrophenol and their Technical Uses. [Preparing Phenacetin.] L. Paul. Zeits. für ang. Chem. 1896, 587.

See under XX., *page* 62.

PATENTS.

New Azo Colouring Matters [Cotton Browns and Reds], The Manufacture or Production of. J. Levinstein and Levinstein, Ltd., Manchester. Eng. Pat. 23,523, Dec. 9, 1895.

Dyestuffs said to surpass in fastness to light most of the azo colours hitherto known, are produced according to this specification, from the mixed tetrazo colouring matters obtained by combining equimolecular proportions of a tetrazotised *p*-diamine, salicylic acid, or one of its homologues, and aniline, or one of its homologues. The colouring matter so formed is then further diazotised and combined with a naphthylamine, naphthol, amidonaphthol, or

dihydroxynaphthalene sulphonic acid, or with a corresponding compound in the benzene series. Thus the tetrazo solution from 18·4 kilos. of benzidine, is combined with 14 kilos. of salicylic acid, and the intermediate compound formed, is run into a solution of 26 kilos. of aniline hydrochloride. After stirring for about 12 hours and heating for another six hours to about 40°—50° C., the mixture is made alkaline and the excess of aniline removed. The yellow dyestuff having the formula—

$$H_2N.C_6H_4.N:N.C_6H_4.C_6H_4.N:N.C_6H_3(OH)CO_2H,$$

is acidulated with hydrochloric acid, diazotised with 7·2 kilos. of sodium nitrite, stirred for a few hours, and after standing for about 12 hours, the brown diazo compound formed is added with constant stirring to an alkaline solution containing 24 kilos. of γ-amidonaphthol sulphonic acid. The formation of the dyestuff is completed within a short time, when it is salted out, filter-pressed, and dried. It forms a brownish-black powder, which dyes unmordanted cotton a deep brown. The shades vary from red to deep brown by employing other *p*-diamines in place of benzidine, such as tolidine, dianisidine, ethoxybenzidine, or diamidostilbene disulphonic acid, and also by substituting *o*-toluidine or *p*-xylidine for the aniline. Moreover, these dyestuffs are suitable, not only for cotton, but also for wool, silk, jute, or other textile fibres.—T. A. L.

Para-nitrophenol and Ortho-nitrotoluene-para-sulphonic Acid, Impts. in the Production of. G. B. Ellis, London. From "La Société Chimique des Usines du Rhone, anct. G. P. Monnet et Cartier," Lyons, France. Eng. Pat. 24,193, Dec. 17, 1895.

The direct nitration of phenol always yields a mixture of *o*- and *p*-nitrophenol, together with a certain proportion of tarry matter. In the present invention, phenol is first condensed with an aromatic sulphonic chloride in presence of an alkali, and the ester thus obtained yields directly, without tarry products, a dinitro derivative, which, on hydrolysis with an aqueous solution of a caustic alkali, forms the *p*-nitrophenolate of the alkali together with the alkali salt of *o*-nitrotoluene-*p*-sulphonic acid.

The further nitration of the ester yields a product which is easily saponified even at the temperature of the water-bath, and with caustic alkalis in very dilute solution. *p*-Toluenesulphonic chloride is particularly specified, since it is produced in large quantities as a by-product. The following example gives the method and the quantities employed:— Sodium phenolate is dissolved in five times its weight of water at 80° C., and mixed with the theoretical quantity of *p*-toluene sulphonic chloride with constant agitation. The ester may also be prepared by dissolving 100 kilos. of phenol and 42 kilos. of caustic soda in 1,000 litres of water by means of a steam jet and gradually adding 210 kilos. of *p*-toluene sulphonicchloride. When the mixture no longer smells of this latter, it is cooled, the ester filtered off, washed, and dried. It has the formula—

$$(1.4)CH_3.C_6H_4.SO_2.O.C_6H_5$$

and melts at about 95° C. In order to nitrate it, a finely ground mixture of 100 kilos. of the ester with the same weight of potassium nitrate is gradually added to 1,000 kilos. of sulphuric acid (66° B.) at a temperature below 15° C. The nitro product is then separated, well washed, dried, and recrystallised from benzene, when it melts at 115° C. It has the formula—

$$(1.2.4)CH_3(NO_2)C_6H_3.SO_2.O.C_6H_4.NO_2.$$

The hydrolysis may be performed by suspending and heating the dinitro ester in its own weight of caustic soda lye (36° B.). On cooling, sodium *p*-nitrophenolate crystallises out, and *o*-nitrotoluene-*p*-sulphonic acid may be precipitated from the mother-liquor by dilute mineral acids. Other esters described in the patent are: benzene sulphonic phenol ester melts at 35° C. and gives a dinitro ester melting at 133° C.; the phenol ester of *o*-toluene sulphonic acid melts at 52° C. and yields a nitration product melting at 195° C., which on hydrolysis decomposes into *p*-nitrophenol and *p*-nitro-*o*-toluene sulphonic acid $(1.4.2)C_6H_3.(CH_3)(NO_2)(SO_3H)$; the phenol ester of α-naphthalene sulphonic acid melts at 75°, and gives a nitration product which on hydrolysis yields *p*-nitrophenol and several nitro-α-naphthalene sulphonic acids are obtained. In a similar manner β-naphthalene sulphonic chloride gives a phenol ester which melts at 99° C. and yields on nitration and hydrolysis, *p*-nitrophenol together with different nitrated derivatives of β-naphthalene sulphonic acid.—T. A. L.

Stable Double Salts of Chloride of Zinc and the Diazo or Tetrazo Compounds respectively of Amido-azo and Diamido-azo Compounds, Manufacture of. Farbwerke vorm. Meister, Lucius, and Brüning, Hoechst-am-Main, Germany. Eng. Pat. 1645, Jan. 23, 1896.

This patent claims the manufacture of the stable double salts of zinc chloride with the diazo or tetrazo compounds of amido-azo or diamido-azo bodies respectively, by precipitating with zinc chloride solution, the filtered aqueous solutions obtained by diazotising the base.—A. C. W.

Direct-dyeing Azo Colouring Matters [Cotton Blacks], Manufacture of. C. D. Abel, London. From "The Actien Gesellschaft für Anilin Fabrikation," Berlin, Germany. Eng. Pat. 2196, Jan. 30, 1896.

This specification is an extension of Eng. Pats. 4018, 7969, and 9431 of 1895 (this Journal, 1896, 110 and 270). The dyestuffs are produced by combining with amines, phenols, and amidophenols, the tetrazo derivatives of compounds of the following general formula—

$$C_{10}H_{\ -n}.X.Y.(N:N.C_6H_4.NH_2)(N:N.Z.NH_2)(SO_3H)_n$$

where X and Y represent amido or hydroxyl groups and Z a radicle containing a diazotisable amido group. For instance, 18·8 kilos. of the compound—

$$1.1'.2.2'.3.3'.C_{10}H_2\begin{cases} NH_2 \\ OH \\ N:N.C_6H_4.NH_2 \\ N:N.C_6H_4.NH.C_6H_3.SO_3H.NH_2 \\ (SO_3H)_2 \end{cases}$$

obtained from amidonaphthol disulphonic acid H, nitroamidodiphenylamine sulphonic acid, and *p*-nitraniline, are tetrazotised by means of hydrochloric acid and 3·5 kilos. of sodium nitrite. The tetrazo compound is run into a solution of 6·1 kilos. of *m*-tolylene diamine kept alkaline by means of sodium carbonate. The dyestuff separates as a black precipitate, and, after stirring and heating, it is filtered off and dried. It is readily soluble in water, and dyes unmordanted cotton a greenish-black. By employing an equivalent amount of β-naphthol in place of the *m*-tolylene diamine, a bluish-black dyestuff for cotton is produced.—T. A. L.

Blue Colouring Matters [Gallocyanines], The Production of. W. H. Claus and A. Rée, Droylsden, Manchester. Eng. Pat. 2705, Feb. 6, 1896.

The aniline addition product of gallocyanine (which is obtained by the action of nitrosodimethylaniline hydrochloride on gallic acid) yields, when sulphonated, useful bright blue dyestuffs, which have a great affinity for mordants, and can be employed for dyeing wool and for calico printing. Finely ground gallocyanine is mixed with four times its weight of aniline and allowed to stand for several hours, when the excess of aniline is removed by washing with benzene or spirit. The condensation product, after filter-pressing and drying, is sulphonated with 4 parts of sulphuric acid at about 70° C. The melt is then poured into water, when the colouring matter precipitates, and, after filtering off, it is well washed, treated with ammonia or an alkali, and evaporated to dryness. Another method employed for obtaining the condensation product in a form suitable for dyeing is to mix 20 kilos. of it with 180 litres of water, 4 kilos. of strong ammonia, and 3 kilos. of sodium bisulphite, 60° Tw. The shades obtained are much bluer than those produced with gallocyanine itself. The condensation may also be performed in spirit solution and the product thus obtained, or as above, after dissolving in sulphuric acid, pouring into water, and washing free from acid, separates as a very fine paste, which may be employed in calico printing. (See also Eng. Pat. 1890, 569; this Journal 1891, 131.)—T. A. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

PATENTS.

Cleansing and Recovering Grease and Oil from Cotton and other Waste and Wool, A Method of and Apparatus for. G. E. Wright, Nottingham, and W. Monk, Nottingham. Eng. Pat. 22,183, Nov. 21, 1895.

The method consists of circulating three charges of liquid solvent through the material, the solvent being pressed out each time and collected. On evaporating the latter, the oil remains behind, whilst the volatile liquid may be condensed and used over again. The washed cotton waste or wool is dried by passing hot air through the vessel containing it.

The essential features of the apparatus are a press fitted with two rams, one fixed and hollow, and suitable connections to a circulating pump and supply tank containing the liquid solvent. Evaporating pans, condensers, a fan, air-heater, and vacuum pump also form part of the apparatus.

The advantages claimed for this apparatus are, the combination of the fixed hollow ram of the press (which is in communication with the solvent supply tank, air heater, &c.) with a movable ram connected to the movable press bottom, which in turn is in communication with the solvent collecting tanks, and also the arrangement of suspended perforated platforms or horizontal chains for loosening the material, to expedite the operation of drying.—W. P. S.

Degreasing and Cleansing Wool, Cotton Waste, and like Fibrous Material; Impts. in Apparatus for. F. N. Turney, Nottingham. Eng. Pat. 1370, Jan. 20, 1896.

The material is passed in the form of a sheet or layer into the cleansing chamber by means of an elevator or endless lifting chain. The levelling apparatus is situated in an air-tight chamber between an air-lock and the degreasing apparatus. This latter consists of a combination of beaters carried in a shogging frame together with an arrangement of rakes and rocking forks for beating the material and advancing it through a series of shallow solvent tanks in the degreasing chamber. The heating of the material takes place upon perforated supports or trays having upwardly curved ends for directing it towards the squeezing rolls, by which it is pressed almost dry on leaving the machine. A second elevator raises the material to these rollers. Suitable means are provided for regulating the feed and thickness of the layer as it passes into the degreasing chamber. After leaving the squeezing rolls, the material falls into a drying chamber.—W. P. S.

VI.—DYEING, CALICO PRINTING, PAPER-STAINING, AND BLEACHING.

Wool, After-Treatment of Chlorinated. Clad and Co., Langensalza, Thuringia. Textile Colorist (U.S.), **18**, [215], 336.

Wool which has been chlorinated, for the purpose of giving it a silky lustre and feel, contracts a yellowish tint, which renders it unsuitable for being dyed very delicate colours. To remove this yellow tint the authors have devised the following process:—The well-washed wool is passed through a weak bath of hydrochloric acid, drained, and treated for about 20 minutes in a cold solution of sodium thiosulphate of ½ B., and rinsed in water slightly acidified with HCl. By this treatment the silky appearance and feel of the wool are promoted, but at the same time a strong yellowish tint. This may be removed by passing the wool through a reducing bath, which is made up as follows:—For 25 kilos. of wool take 800 litres of water at 40°—50° C., 2 kilos. of stannous chloride, and 4 litres of hydrochloric acid of 30 per cent. strength. From this bath the wool is again rinsed in acidulated water.—I. S.

Scouring of Woollen, Worsted, and Mohair Yarns. L. K. Frankel and A. Hamburger. Textile Colorist (U.S.), **18**, [215], 326.

The authors emphasise the necessity of removing every trace of oil from the yarns which they receive during the spinning process, prior to their being dyed, since otherwise the colours are liable to be cloudy, or the yarn may be stained. If lard or olive oil only has been used, the scouring is a simple matter. But the process is one of considerable difficulty if mineral oils, or a compound containing mineral oil, have been employed. Such compounds are generally used for coarse and cheap carpet yarns. The authors are of opinion that even for the coarsest yarns, only such oils should be used as are readily removable, since the saving in labour and soap would compensate for any difference in cost, whilst leaving the fibre in a better condition.

As the most suitable detergent, the authors recommend potash soaps made from olive oil, or olive-oil foots, and potassium carbonate, mixed in the proportion of 75 per cent. of the former to 25 per cent. of the latter. The quantity to be used will depend on the amount of fatty matter present in the yarn. For coarser yarns good palm-oil soap, with sufficient sodium carbonate or soda ash, "will be found to answer all the requirements of the carpet manufacturer"; but for woollen and worsted yarns used in finer carpets, potash soap and potassium carbonate are to be preferred.

Excessive heat of the scouring bath is to be avoided, as too high a temperature may cause the yarns to felt and render them harsh as well as "lean" in appearance. The temperature should be below 125° F.; for fine yarns, 115°—120° F.), which may be increased to 130° F. in case mineral oil is present.—I. S.

Scouring and Finishing Woollen, Worsted, and Mohair Piece Goods. L. K. Frankel and A. Hamburger. Textile Colorist (U.S.), **18**, [215], 326—327.

On the thorough cleansing of the piece before dyeing depends its finish and beauty. Soaps used for the scouring and fulling of woollen goods should therefore be free from alkaline silicates, which communicate a harsh feel; and from rosin, which causes cloudiness and makes the pieces sticky and ill-smelling. The authors prefer palm-oil to tallow soaps, because tallow contains a large proportion of stearin, the soap of which is not so soluble as that from palmitin or olein. Solubility of the soap is an important feature, so that it can be easily washed out. Tallow oil, that is the liquid constituents of tallow, in combination with palm oil, "makes an ideal fulling soap." The soap should be nearly neutral, as an excess of caustic alkali will corrode the fibre and render the goods harsh. To remove any fatty matter from the pieces, sodium or potassium carbonate may be added to the soap-bath, the authors giving preference to the latter.

For worsted goods, where thorough cleanliness and softness of finish is more desired than a felted surface, olive-oil potash soap, or a mixture of equal parts of olive-oil potash and olive-oil soda soaps will yield satisfactory results, giving the pieces a soft, smooth finish and a clear, bright colour.

Mohair and lustre fabrics attain their highest brilliancy, it is alleged, when scoured with a mixture of olive-oil potash soap and potassium carbonate.—I. S.

Tartar Emetic or Antimony Fluoride Salts? E. Eulenthaler, Leipziger Färber- u. Zeugdrucker Zeit. **45**, 491—492; and R. Erhardt, the same Journal, **45**, 515—516.

Certain manufacturers of "antimony fluoride salt" having claimed that by the use of this product instead of tartar emetic a saving in cost of 52 per cent. may be effected, E. Eulenthaler has tested this statement on the large scale, and is unable to confirm it. He finds, on the contrary, that amounts of the two substances containing an equal weight of Sb_2O_3, do not give equally good results, and that even when the same amount of each substance is used, a larger quantity of dyestuff is required when the fluoride is employed, than with tartar emetic, to obtain an equal depth of shade. This he attributes to the readiness with which

the fluoride suffers decomposition, free acid being liberated, which dissolves tannate of antimony from the fibre. The alleged saving in cost is due to the fact that dyers are in the habit of using far too large an amount of tartar emetic in the fixing bath.

In a reply to the above, R. Erhardt states that his experience fully substantiates the claims of the manufacturers; he finds the necessary amount of fluoride salt to be only half that of the tartar emetic usually employed, and the saving in cost amounts to 56 per cent. or more. The ready decomposition of the fluoride causes a much greater fixation of Sb_2O_3 on the fibre—in fact, the bath is perfectly exhausted, and the acid liberated is no more injurious than the tartaric acid from tartar emetic; however, in both cases, a thorough washing should follow the fixing operation. It is true that the amount of antimony compound employed, and also the quantity of tannin, is frequently too great, but the necessary amounts vary according to the colouring matter, and unless they are separately ascertained for each dyestuff, it is well to use quantities known to be sufficient.—R. B. B.

Cotton Cloth, The Drying of Mordanted. M. J. Wikwicki. Zap. imp. russk. techn. obschtsch. 1896, **30**, [6—7], 231.

If cloth impregnated with a tannin solution be dried on hot cylinders before passing it through the antimony solution, then the cloth will dye darker shades on one side than on the other, the side which first touches the hot cylinders eventually acquiring the darker shade. The difference is also the greater, the hotter the cylinders are. The colour will also be uneven, since knots, thicker threads, and in general all those parts which are pressed more firmly against the hot cylinders, will dye darker than the other parts. The cause of this is that where the cloth comes into contact with the hot cylinders, the solvent of the tannin is evaporated, and fresh moisture from the interior of the cloth is drawn by capillary action towards the partially dried surface, causing thereby a local accumulation of tannin. The author is of opinion that this tendency of the mordant to accumulate on the surface of the cloth during drying, might be utilised to effect a saving in mordanting and colouring materials, by so conducting the drying that only one side of the cloth shall touch the cylinders, and never the other. To ensure evenness, the temperature of the cylinders should be so regulated that the cloth shall be barely dry when leaving them.—I. S.

The Dyeing of Hard-milled Woollen Felts. W. Ringmann. Färber Zeit. **7**, 457.

The method of dyeing felt, 10—15 millimetres in thickness, depends upon the nature of the previous milling process. Goods which have been milled with soap are boiled for two hours with 10 per cent. of sodium sulphate only, half the necessary quantity of dyestuff is then added, together with 5 per cent. of sodium bisulphate, and after boiling for 1—1½ hours longer, the remainder of the dyestuff and another 5 per cent. of $NaHSO_4$ are added. When dyeing in old liquors the acid must be neutralised with soda before proceeding as above.

If the goods have been milled with acid, the acid remaining in the fibre is first neutralised by boiling for two hours with soda and a little sodium sulphate, after which dyeing proceeds as before.

In the case of felt 5—8 mm. thick, it is sufficient to neutralise, boil ¾ hour with dyestuff only, add the acid, and continue boiling.

Piano felts 10—20 mm. thick, which require to be perfectly dyed through, are half milled with soap, dyed as given above for thick felts, and the milling is then completed with acid and hot water.—R. B. B.

Stannous Chloride, Preparation of. [*Recovery of Tin from Spent Tin Baths.*] L. Vignon. Textile Colorist (U.S.), **18**, [215], 334.

See under VII., *page* 43.

PATENTS.

Waterproof Fabrics, Impts. in the Printing of, and Apparatus therefor. J. Berry, Salford. Eng. Pat. 1375, Jan. 20, 1896.

The inventor has found that by spreading a film of water on rubber, or the proofed surfaces of waterproof fabrics, before entering the printing machine, such goods can be printed on without the application of farina or other finely powdered material to the surface. To carry out the process, two (or more) troughs are placed in front of the printing machine, each being provided with one or more rollers, over which the fabric travels before entering the printing machine. One only of the troughs contains water into which the revolving rollers dip, whilst the other is empty, its rollers serving the purpose of removing excess of moisture.—I. S.

Dyeing [*Felt or Cloth*], *An Improved Method of.* A. H. Brownlow and J. Stansfield, Leeds. Eng. Pat. 2844, Feb. 7, 1896.

The improvement consists in dyeing thin felt or cloth a different shade on each side. The cloth is first dyed through any required shade of colour and finished in the usual way; a second dyestuff is then applied to one side of the cloth only on a printing machine "of any suitable construction."
—I. S.

VII.—ACIDS, ALKALIS, AND SALTS.

Sulphur, Extraction of, from Brimstone Earths. E. F. White. Eng. and Mining J. **62**, [23], 1896, 536.

The author deals with the "efficiency of apparatus" and the "economy of method" of a new steam separator for the production of crude sulphur. "Efficiency" he defines as the ratio expressed by $\frac{\text{available product}}{\text{total expenditure}}$, the two factors being developed in like terms.

"Economy of method" varies with the costs of labour, fuel, and water, and in some cases is attained without high "efficiency." Some Sicilian deposits, containing 20 per cent. of S, are more economically worked by simple methods, possessing an efficiency of only 40 per cent., than by apparatus yielding a much larger return. The purer sulphur obtained by distillation is never obtained direct from the natural earths. It is found much more economical to subject crude sulphur alone to distillation. The older forms of steam-heat separators used for the production of this crude sulphur, were wasteful of heat and inconvenient in manipulation. The separator described by the author possesses advantage in these respects. It is designed for use with water, steam, or air as heating media. A conical vessel, in shape resembling a blast furnace, is provided with a double outside casing. Two annular spaces are thus formed round the body of the apparatus. The sulphur earth in 3-in. lumps is fed continuously from the top into the middle annular space, whilst hot water or steam, from a boiler working under 80 lbs. pressure, is admitted to the outer annular space and to the inner cone. The sulphur in the earth, during its passage downwards between the two heating surfaces is melted, and is collected in, and automatically drawn off, from a steam-jacketed receiving-pan fixed beneath the cone. Mechanical arrangements keep the earth travelling downwards in the annular space, and remove the residual earth continuously from the draining plate and from the bottom of the receiving pan. The cost of treating sulphur earth by this apparatus at the mines on a scale of 25,000 tons of crude sulphur annually, with separators of a capacity of 20 tons of earth per 24 hours, is 7*s.* 3*d.* per ton. For a 65 per cent. earth, this works out to 11*s.* 2*d.* per ton of crude sulphur. Since the specific heat of sulphur and of the earths usually allied with it are nearly the same, and since the latent heat of fusion of sulphur is exceedingly small, the cost of treatment per ton of crude sulphur for poorer earths, can safely be calculated from these costs for earth containing 65 per cent. of sulphur.
—J. B. C. K.

Hydrochloric Acid free from Arsenic, Preparation of. G. Friese. Chem. Ind. 1896, **19**, 187.

Hydrochloric acid perfectly free from arsenic is prepared in the usual way from common salt and commercial sulphuric acid, except that the gas, before passing into the absorption apparatus, is washed, and remains in contact with boiling water or steam. The volatile arsenic chloride, which is carried over with the hydrochloric acid, reacts with the water, forming arsenious acid, which is not volatile, and hydrochloric acid. The wash-water must be kept boiling, as at a certain point of concentration of the acid solution formed, a re-conversion of the arsenious acid into volatile arsenic chloride takes place. After a long time, the wash-water will have become so charged with arsenic, that the hydrochloric acid carries over some arsenic trichloride with it. When this point has been reached, the wash-water must be renewed.

The hydrochloric acid obtained by this method, is so completely free from arsenic that it remains absolutely clear on warming with tin, but the author is uncertain whether the method will work as well on a large scale as on the laboratory scale.—A. S.

Fusibility, Abnormal; of Mixture of Alkaline with other Sulphates. Le Chatelier. Comptes rend. 1896, **123**, [19], 746—749.

The author finds, in opposition to the general rule, that the melting point of alkaline sulphates is elevated by an admixture of other metallic sulphates. Accompanying the paper is a set of curves which exhibit the influence on the melting point of sodium sulphate, of progressively increased admixtures of calcium sulphate, magnesium sulphate, &c., respectively. It appears that the melting point does not increase indefinitely as the proportion of added sulphate becomes more elevated, but attains a maximum at concentrations (above which the curve again descends) varying in the different examples. The increase is greatest in the case of $CaSO_4$, barely noticeable with $MgSO_4$, whilst $BaSO_4$, $CdSO_4$, and $PbSO_4$ occupy an intermediate position. Apart from this, the formation of double salts, such as $PbSO_4 + Na_2SO_4$, $2MgSO_4 + Na_2SO_4$, and $2CdSO_4 + Na_2SO_4$ is indicated by distinct breaks at corresponding points in the complete curves. In addition, it was observed that Na_2SO_4 and all its mixtures (within certain limits) with $CaSO_4$ or $PbSO_4$, after fusion, remain perfectly transparent, even when cooled to atmospheric temperature, and exhibit identical crystalline structure. It is concluded, therefore, that Na_2SO_4 is isomorphous with some of its own double salts.—H. T. P.

Stannous Chloride, Preparation of. [*Recovery of Tin from Spent Tin Baths.*] L. Vignon, Textile Colorist (U.S.), **18**, [215], 334.

The author has found that by treating the oxides of tin with hydrochloric acid, in the presence of metallic tin, they may be converted into stannous chloride, the reactions being—

$$SnO + Sn + 4(HCl) = 2(SnCl_2) + H_2O + H_2$$
$$SnO_2 + Sn + 4(HCl) = 2(SnCl_2) + 2(H_2O).$$

The process is utilised for the recovery of tin from old tin baths, or the waste liquors of the dyehouse containing tin in solution. From such waste solutions the tin is precipitated as SnO or SnO_2 by means of lime, sodium carbonate, sulphuric acid, sodium sulphate, &c. The oxides are digested in concentrated hydrochloric acid, and, if necessary, heated to 80° C. until solution is complete, and the metastannic acid which it may contain, is transformed into stannic acid. This hydrochloric acid solution is then treated with granulated tin, which dissolves without evolution of gas. The solution is then concentrated, and the stannous chloride allowed to crystallise.—I. S.

Borax, Commercial, Valuation of. G. Heid. Zeits für angew. Chem. 1896, 679.

See under XXIII., *page* 66.

PATENT.

Caustic Soda, Carbonate of Soda, and Sulphide of Sodium [*as By-products*], *Impts. in the Manufacture of.* H. R. Angel, London. Eng. Pat. 335, Jan. 6, 1896.

To obtain caustic soda and carbonate of soda, sulphate of soda is mixed with iron oxide, coal, or coke, and with roasted or unroasted lime, blende or other ores. During the process of smelting, the sulphate of soda is changed into sulphide and this is converted into caustic soda by the iron oxide. A portion of the charge is converted into carbonate of soda and a portion remains as sulphide if unroasted ores be used; if, however, the ores have been well roasted before treatment, this sulphide entirely disappears, and a solution is left of a strength such that it contains about 75 per cent. of caustic soda and 25 per cent. of carbonate of soda. The claim is for the "Recovery of caustic soda, carbonate of soda, and sulphide of sodium as by-products, from the reduction of refractory and other ores."—D. B.

VIII.—GLASS, POTTERY, ENAMELS.

Glass-Making, Aluminium in. Central-Blatt. f. Glas-Ind. **11**, 284.

According to L. Appert the employment of aluminium in glass-making is calculated to revolutionise the industry. From the results of numerous analyses of old window-glasses he is convinced that aluminium will increase the durability and elasticity of the product. Appert has found that 7 or 8 per cent. of this metal can easily be added to molten glass. The fusibility of the glass thus becomes increased, without any sensible loss of ductility. Impure aluminium colours the glass, through presence of traces of iron, but this should not be difficult to remedy.—C. S.

Glass, Proportions of Constituents in the Mass. R. Hohlbaum. Sprechsaal, **29**, 1170.

Frequently the defects in glass are due to wrong proportions of the materials having been taken. The simplest way of detecting errors of this class is to calculate out the composition of normal glass from pure materials to the materials at disposal, and compare the resulting proportions with those taken. For this purpose, taking normal soda-lime glass as a basis, the amount of silica is calculated according to Benrath's formula $z = 3\ (x + y)$, wherein x = alkali oxide, y = calcium oxide, and z = silica; and since x = 62 parts of Na_2O and y = 56 parts of CaO, then 354 parts of silica are required. The above amounts of Na_2O and CaO corresponding to 142 and 100 parts of chemically pure Glauber salt and chalk respectively, the following proportions result for the mixture (normal glass):—Sand, 354 parts; Glauber salt, 142 parts; chalk, 100 parts; and coke, 9 parts.

In practice it is found that the coke should not exceed 5 to 6 per cent. of the weight of Glauber salt, the carbon monoxide evolved during the reduction also assisting therein.

If now the materials at hand are: Sand, 99 per cent. pure; Glauber salt, 96 per cent. pure; and chalk, 98 per cent. pure, then the proportions must be modified from the foregoing, as follows:—

Sand.	Glauber Salt.	Chalk.	Coke.
358	148	102	9
or per 100 parts of sand			
100	41	28	2·5

When the Glauber salt is replaced by sodium carbonate the calculation is similar, the weight of the molecule (106) being taken; but in this case the coke may be omitted.
—C. S.

Earthenware Industry, Impts. in the. H. Hecht. Dingler's polyt. J., **302**, [10], 237—240.

The composition of Thuringian porcelain body is given in Sprechsaal, **28**, 59, 88; Illus. Faehbl. f. Glas- Porzell, u.

Steinguind. 3, 282. It appears that English and Bohemian earths are used along with the German raw materials, particularly for ware that is designed to have a fine appearance, some makers also importing earth and "body" from France.

According to M. Hosoki (Thonind. Zeit., 19, 319) *Imari porcelain* (Japan) is prepared from a natural porcelain earth, resembling that found in some parts of China, which needs no addition of quartz or felspar, but can be worked up direct. This earth, "Idzumiyama," consists of 49·07 per cent. of clay, 14·52 of quartz, and 36·61 of felspar, and is nearly allied in composition to hard German porcelain. By the addition of a certain proportion of wood ashes the earth is converted into a suitable glaze. The ordinary firing temperature is employed in the kilns.

In the *modelling of porcelain ware* the tendency is to discard turning in favour of moulding, the increased mobility necessary in the mass being obtained by additions of sodium carbonate or other alkali in flux. This action of the alkali is explained by Kosmann (Thonind. Zeit., 19, 382) by the assumption that the clay has a stronger affinity for the water of hydration in the alkali, which it displaces, the heat thereby generated increasing the fluidity of the mass. Moreover, soda, though inactive towards alumina, may dissolve some of the interstitial particles of silica, and so dilute the mass and increase its mobility. On the other hand, A. Zebisch (Sprechsaal, 27, 1005, 1895) considers the amount of soda too small for its action to be thus explained, and attributes the effects rather to osmosis. Seger (Gesammelte Schriften, p. 573) assigns the action of alkalis, and the converse effect of acids, to molecular alterations, and attributes the plasticity attained by porcelain earth in "rotting" to a putrescent bacterial fermentation of the organic matter in the water, acid compounds, which increase the pliability of the mass, being formed.

The *cracking of the glaze on ceramic ware* is explained by Le Chatelier (Monit. céram., 26, 162) as being due to the irregular expansion of the sand when heated. His experiments showed that all siliceous substances, including the amorphous silica present in ceramic earth, undergo sudden alterations in volume at certain temperatures, their expansion thus contrasting with that of the glaze, which is regular. The fact that porcelain alone expands regularly is ascribed to the probable conversion of its silica into the amorphous form during the partial vitrefaction the mass undergoes in firing.

O. Balthasar describes the *manufacture of porcelain flowers* (Monit. céram., 23, 136). The body consists of 20 parts by weight of quartz sand, 15 parts of felspar, 15 of kaolin, and 2 of calcspar, the plasticity being increased by syrup or gum arabic. The glaze is made from 12 parts of sand, 10 of felspar, 6 of calcspar, 3 of porcelain sherds, and 3 of calcined kaolin, mixed, and the ware is fired at 1,300° C.

The author (Thonind. Zeit., 19, 453) described the coloration of various porcelain and stoneware glazes by copper oxide. Pure silicates of potassium, sodium, or potassium-sodium ($RO : 3SiO_2$) dissolve the oxide and become dark blue in colour. On replacing part of the silica by its equivalent of boric acid the colour assumes a greenish shade, more noticeable with soda glass than with potash glass, and this tendency is intensified in presence of alumina, and by the substitution of the alkaline earths, wholly or in part, for the alkali metals. The lead-alkali silicates fused with silica only, are blue when the molecular proportion of potash is equal to or greater than the lead present; otherwise green, the effect of boric acid and alumina being as already stated. The potassium-zinc and potassium-bismuth glasses are coloured green when the metals are in equal molecular proportions. To raise the fusing-point of these glazes to the necessary degree for use on porcelain, they must be mixed with refractory bases exerting a minimum influence on the colour. The most suitable mixtures for Seger porcelain are:— $1K_2O$, $0·5 ZnO$, $4SiO_2$, and $1K_2O$, $1·25 ZnO$, $5·1 SiO_2$, prepared by the aid of the following frits:—$2K_2O$, $1ZnO$, $6SiO_2$, and $1K_2O$, $2ZnO$, $6SiO_2$, and mixed with 4 per cent. of CuO to produce a fine Turkish blue.

A New Continuous Furnace for firing muffle colours and gold decorations is the Fürbringer muffle (Sprechsaal, 29, 755). A channel furnace runs round three sides of a square, the fire being in the centre, and, the hot gases being diverted on either side, the ware is progressively heated as it is moved forward until it reaches the maximum, at the point nearest the fire, and thereafter is gradually cooled down in its progress towards the outlet. The pieces are placed in baskets of hoop iron, which can be pushed forward, by suitable means, at each angle of the furnace, at intervals of 6 or 7 minutes, the whole charge of 20 baskets passing through in about 2 hours. This continuous method obviates the loss of heat occasioned by the transference of the ware, in ordinary furnaces, and gives a finer lustre to the colours fired in it.—C. S.

Stone [Artificial] from Glass Sherds. Central-Blatt f. Glas-Ind. 11, 293.

See under IX., below.

PATENTS.

Metallic Decorations on Earthenware, Glass, and the like, an Improved Printing Process for Producing. H. Kunze, Kolmar, Germany. Eng. Pat. 19,123, Aug. 29, 1896.

"The printing and fluxing mediums are impressed together;" "the metallic powder, *free from flux*, is then dusted upon the drawing."—V. C.

Enamelled Metallic Ware, Impts. in the Manufacture of. A. Niedringhaus, St. Louis, U.S. Eng. Pat. 21,512, Sept. 29, 1896.

The process is one "of preparing steel for enamelling, which consists in placing it, a chloride, and saltpetre in the annealing pot, and subjecting the same to the ordinary annealing heat." After this the enamel is applied to the base, and fused thereon.—V. C.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Stone [Artificial] from Broken Glass. Central-Blatt f. Glas-Ind. 11, 293.

A new building material, called "ceramic stone" by its inventor, Garchey, is being tested in France. The chief raw material is broken glass, which is ground to powder, and de-vitrified by passing it through two furnaces, the second one at a very high temperature. Finally, the now pasty mass is pressed, and thereby acquires form and solidity.—C. S.

Cements, Hardening of. E. Candlot. Baumaterialienkunde, 1896, Heft 1–3.

The abnormal phenomena sometimes observed in using cements are, in almost all cases, referable to very simple causes. Of these, the following are some of those more frequently occurring:—(1.) The behaviour (solubility and crystallisation) of the aluminates, which plays such an important part in the hardening of cements, is varied (*a*) by the presence of other substances, and (*b*) by the hydration of the aluminates by exposure to the air or contact with moist sand. (2.) The carbonation of the free lime in the stored cement by the carbonic anhydride of the air. (3.) The presence of calcium sulphate, which often causes the crumbling of cement introduced into water immediately after mixing.—L. T. T.

Portland Cement, Impt. of, by Admixtures. L. Erdmenger. Thonind. Zeit. 20, [51], 757, [53], 786—788.

The result of Landteigen's experiments with admixtures of Portland cement and a siliceous American earth resembling trass (Thonind Zeit. 20, [32]) agree with those obtained in a northern laboratory, where an admixture of a siliceous by-product was employed in conjunction with sand, the advantage being attributed to combination of the siliceous material with the free lime in the cement, whereby the tendency of the latter to crumble is counteracted. The results, as regards the cohesive power of the mixed cement

at the end of a year, appear, from the tables given, to have been generally more favourable in proportion to the increase of siliceous material, up to between 20 and 50 per cent. (ratio of cement to sand, 1:3), according to the cement used. In two series of tests with a lightly burnt cement, it was found that the addition of from 10 to 25 per cent. of siliceous material rendered the product more durable than the unmixed fully burnt cement, especially when the semi-calcined article was previously slaked. This siliceous material differs from the American earth and trass in being more effective when previously dried, whereas the converse is the case with the two latter.

The author considers that greater value ought to be attributed to the increased durability imparted to the cement by the admixture of siliceous matter than is at present the case, more stress being now laid on quick-setting than on permanence.—C. S.

Clay for Brickmaking, Increase of Content in Sulphates during Firing. H. Günther. Thonind. Zeit. 1896, **20**, 583.

Bricks of a certain brand, when employed in building construction, regularly developed a whitish efflorescence consisting almost entirely of potassium and sodium sulphates; although the same bricks when stacked alone (without mortar intervening) remained practically unchanged for long periods of time, and although the clay from which they were made contained little more than traces of sulphates. It is generally assumed that an increase in soluble salts (especially sulphates) occurs during the firing of bricks, under the influence of certain constituents of the flue-gases. The author shows that such an increase likewise occurs even when fuel free from sulphur is employed, if the clay contain pyrites—as was the case in the present instance. As regards the source of the efflorescence, this was found to arise from the interaction of gypsum contained in the bricks with alkalis derived from the intervening mortar.—H. T. P.

Wood, Treatment of, with Antiseptics prepared from Petroleum. A. W. Adiasiewietsch. Trudy, bak. otd. imp. russk. Tech. obschtsch. 1896, **11**, 243.

The resinous matters obtained from petroleum by the author's previously published method (this Journal, 1896, 346 and also 267) are superheated by agitation with heated plates, and then distilled at 350°—400° C. The product flows in cascade fashion in a thin stream, through an air-tight cast-iron chamber, heated to 450°—480° C. The vapours produced are led into a still, filled with the same product at its boiling point. These vapours, owing to their higher temperature, much assist the distillation. A condenser, protected from too rapid cooling, is attached to the still. By this process, 75 per cent. of antiseptic compounds were obtained from a resinous substance prepared from a petroleum-still residue. Nine per cent. remained as a tar in the still.

Machinery oil residues, yielded resinous matter producing 58 per cent. of antiseptic compounds and 24 per cent. of tarry matter. The average composition of the antiseptic liquid, was 20·9 per cent. of oils, boiling below 300° C.; 31·2 per cent. between 300°—400°; 6·7 per cent. of solid anthracene; 4·2 per cent. of phenols; 8·0 per cent. of naphthalene and 10 per cent. of coke. Treatment with hydrochloric acid extracted basic oils containing aniline. The author considers that these together with the anthracene oils, form the real antiseptic agents; whilst the resinous constituents of the mixture confer on the wood greater density and durability. The wood must be carefully dried before treatment. The usual steaming is a mistake, since the after vacuum treatment fails to remove all the moisture. Treatment with the vapours from the boiling antiseptic is better. These coagulate the albumins, but at the same time expel all moisture. The usual vacuum treatment follows, and then treatment with the antiseptic fluid under $3\frac{1}{2}$—4 atmos. pressure.

The light hydrocarbons retained in the pores of the wood seem to dissolve the antiseptic, and to assist its permeation. Dried wood absorbs more of the antiseptic than steamed wood; but wood treated as described above, absorbs still more. Wood so treated loses 2—6 per cent. in weight in the first three months, owing to evaporation of the lighter antiseptic oils. A further loss is not advantageous, and may be prevented by dipping the wood at this stage for 2—3 minutes in the tar, heated to 150°—200° C., which remains as a still-residue when preparing the antiseptic This tar penetrates about 1 mm. in depth, and forms an impermeable outer coating.—J. B. C. K.

PATENTS.

Artificial Stones, Manufacture of [Use of Electricity]. F. Coulon and J. J. Defalque, Blaton, Belgium. Eng. Pat. 23,018, Dec. 2, 1895.

The inventors claim the use of electricity as a petrifying agent, in the manufacture of artificial stone.—V. C.

Kilns for Use in the Manufacture of Bricks, Terra-Cotta Goods, Tiles, and the like; Impts. in. J. W. Ormrod, Bolton. Eng. Pat. 1794, Jan. 25, 1896.

A series of chambers is placed on each side of a central flue, which serves for both. From the chambers, passages leading to the central flue conduct steam to a chimney. A partition in the central flue provides a passage for the smoke, which escapes by a second chimney. Any one of the chambers can be used as a separate kiln.—V. C.

Kilns or Furnaces, Impts. in, Applicable for Burning Lime, Cement, and other Minerals. H. Simmonds, Colne, Lancs, and John Delaney, Horton-in-Ribblesdale. Eng. Pat. 2843, Feb. 7, 1896.

The gases and products of combustion from the gas producer are raised to "a white heat" in a combustion chamber, supplied with heated air, before being admitted to the kiln.—V. C.

Wood, An Improved Composition for use in Imitation of or in Substitution for. F. L. Schauermann, Twickenham, and J. S. Cowper, London. Eng. Pat. 6232, March 20, 1896.

This composition is produced "by the action of nitric acid on a mixture consisting essentially of comminuted or disintegrated wood and a siccative."

The following proportions have been found suitable, *viz.*, sawdust, 1 pint; sulphate of zinc or sulphate of copper or a mixture of both in any proportions, $\frac{1}{4}$ pint; powdered rosin, 2 oz.; nitric acid, $\frac{1}{4}$ pint; and linseed oil, 2 fluid oz.—V. C.

X.—METALLURGY.

Gold, Chemistry of the Cyanide Process for Extracting. G. Bodländer. Zeits. für angew. Chem. 1896, 583—587.

The reaction which occurs in the solution of gold by potassium cyanide, was supposed by McArthur to take place according the equation—

$$2Au + 4KCN + 2H_2O = 2KAu(CN)_2 + 2KOH + H_2.$$

It has been shown, however, by Maclaurin, Goyder, and others that atmospheric oxygen takes part in the action, thus:—

$$4Au + 8KCN + 2H_2O + O_2 = 4KAu(CN)_2 + 4KOH.$$

The author finds that the reaction is not so simple as this equation would indicate, but that it takes place in two stages. In the first of these, hydrogen dioxide is formed—

$$2Au + 4KCN + 2H_2O + O_2 = 2KAu(CN)_2 + 2KOH + H_2O_2$$

and the reaction then proceeds without further aid from the atmospheric oxygen—

$$2Au + 4KCN + H_2O_2 = 2KAu(CN)_2 + 2KOH.$$

Using pure gold, 90 per cent. of the amount which is theoretically soluble in this manner, may be dissolved by a given quantity of potassium cyanide.

The rate of solution of gold in alkali cyanide solutions increases with the concentration of the latter, thus the times required for the solution of 46 sq. cm. of gold leaf

(= 7·1 mgrms.) in 20 c.c. of 0·1, 0·25, 1 and 3 per cent. solutions of potassium cyanide, are approximately 12, 5, 4, and 3 mins. respectively. This does not accord with the statement of Maclaurin that a maximum rate of solution is obtained with a 0·25 per cent. solution.

The solution of gold by reagents so indifferent as potassium cyanide and (at ordinary temperatures) oxygen, is best accounted for by the theory of free ions. In its simple salts, gold is present as cathion, but it cannot be made to replace hydrogen, and cannot be brought into the ionic state by the energy produced by the oxidation of hydrogen. Solution, in fact, occurs owing to the gold entering into union with cyanogen and forming a complex anion—

$$2Au + 4\overset{+}{K} + 4C\overline{N} + 2O\overline{H} + 2\dot{H} + O_2 = 2Au(C\overline{N})_2 + 4\overset{+}{K} + 2O\dot{H} + H_2O_2.$$

In view of the fact that hydrogen dioxide acts upon hydrocyanic acid, with the formation of oxamide, as Radziszewski has shown (Ber. **18**, 355), the production of hydrogen dioxide in the above reaction may to some extent explain the loss of hydrocyanic acid which is experienced in practice.—E. B.

Gold, Detection of, in Sea Water; also Oxidation of Ferrous Sulphate by Sea Water. E. Sonstadt. Proc. Chem. Soc. **12**, 236.

Reference is made to the author's papers (Chem. News, **25**, 196, 231, and 241) relating to the presence of calcium iodate in sea water, and to the absence in existing analyses of sea water of the recognition hitherto of the presence of any salt capable of acting as an oxidiser. Results were obtained which point to the presence in sea water of other oxidising salts besides calcium iodate, which is not in sufficient proportion to account for all the facts ascertained.

In the second part of the paper, on the detection of gold in sea water by means of mercury, experiments are described, in which pure mercury was agitated with sea water and then volatilised in a porcelain crucible. The black film left behind was digested in strong hydrochloric acid, which removed silver and other metals, leaving a stain, soluble in *aqua regia*, giving a minute gold bead on cupellation. It is suggested that the precious metals are probably present in sea water in a free state, owing to molecular strain from the great dilution of their salts.

The preceding experiments were made in 1895, on the Great Eastern Railway Company's sea water, conveyed in oak kegs. Reference is made to unpublished experiments on sea water from the same source, made a few years ago, in which assays were made of the ferric oxide obtained by treating the sea water, slightly acidulated, with ferrous sulphate, which experiments showed that water from this source was much poorer in gold than the water of the Irish Sea, on which the author's first experiments in 1872 (Chem. News, **26**, 159) were made.—E. S.

Gold, Absence in Granite Rock of Bald Mountain District, Wyoming. F. D. Smith. Eng. and Mining J. **62**, [23], 1896, 535.

The author was sent down to confirm or refute the report that the whole of the granitic rock in this district contained gold. The manager of the Fortunatus Company, which works the conglomerate of this locality for gold, had asserted the presence of gold as a "telluride." The author's results show that no gold is present, and that the report was erroneous.—J. B. C. K.

Silver [and Uranium] Mines at Joachimsthal, Bohemia. R. Helmbacker. Eng. and Mining J. **62**, [23], 1896, 533—534.

The mine was originally worked for silver, which exists in rich pockets, but, owing to the wide space over which such pockets are scattered, greater attention was at length paid to the cobalt, nickel, bismuth, and uranium, formerly considered as by-products. Owing to the high value of the uranium, more recent development has been chiefly undertaken in this direction, and the mine is now an uranium mine, with silver and the other metals considered as by-products. Besides developing the mine, the old waste heaps from the early workings have been utilised with satisfactory results. The ore is usually massive or in grains, with a colour from greyish to velvet-black, and a dull, submetallic, pitch-like lustre. If pure, the composition should be U_3O_4, but analysis shows that it contains only 68·5 to 86·5 per cent. of U_3O_4, whilst the admixtures are FeO, 2 to 5·7 per cent.; CaO, 0 to 5·8; MgO, 0 to 0·5; SiO_2, 2·4 to 5·4; PbS, 2·8 to 0·6; PbO, 0 to 0·7; Al_2O_3, 0 to 0·3; Bi_2O_3, 0 to 1·2; Bi, 0·5 to 0·6; Cu, 0 to 3·9; CO_2, 0 to 3·3; MnO, 0·1 to 0·8; As, 1·2 to 4·3; Zn, 0 to 0·7; and H_2O, 0·4 to 2·6; together with SO_3, V_2O_5, and traces of other metals. The coloured ores are all derived from this black species. It is stated that in no other district in the world is uranium ore found in extractable quantity, and the works erected at Joachimsthal for its conversion into colours for use is the only one in existence, and the Crown monopolises the trade.

The treatment of the dressed ore is as follows:—It is first calcined in a reverberatory furnace to decompose the sulphides, arsenides, &c., after which 15 per cent. of sodium carbonate and 2 per cent. of sodium nitrate are added, and the whole again strongly heated. The U_3O_4 is thereby converted into a form readily soluble in dilute acids, whilst the impurities are transformed into salts soluble in water. The latter are extracted with hot water, and the uranium oxide dissolved in dilute sulphuric acid. Caustic soda is added in excess to the sulphate solution, so as to precipitate other metallic oxides whilst re-dissolving the uranium hydrate. This solution, after separation, is treated with sodium carbonate, and yields an insoluble sodium uranite, $Na_2U_2O_7 + 6H_2O$. This is the original process of treatment, and, with the addition of a few improvements, is that now in use. The dressed ore treated in 1887 was 30 cwt., at 337 florins per cwt., whereas in 1894 it was $87\frac{1}{2}$ cwt., at 657 florins per cwt. The marketable products consist of a yellow-orange or dark orange uranite of soda, a hydrated compound of the formula $U_2(NH_4)_2O_7 + 6H_2O$, and the black oxide, and they are used for glass staining and porcelain painting. The output is variable, amounting to from 20 to 120 cwt. per annum. Two-thirds of the production of 1895 was consumed in Austria, whilst one-third only was exported.—A. W.

Steel, Use of Phenol (Carbolic Acid) for Tempering. M. Levat. Comptes. rend. **123**, 945.

The author describes experiments made with chisels and with ingots.

In the former case, the chisel, tempered to a blue tint in carbolic acid, was superior in hardness to the one tempered at a cherry-red in water. The ingots, of ordinary cast steel and of rolled steel, showed a similar improvement in hardness, elasticity, and also in grain when tempered at a white heat in carbolic acid. The amount of carbon in the steel was not increased by tempering in carbolic acid.

—J. B. C. K.

Aluminium, Behaviour towards Ammonium Compounds; and the Utilisation thereof for the Chemical Modification of the Surface of the Commercial Metal. C. Goettig. Ber. **29**, 1671.

The fact that, by the action of alkaline bases, of hydrofluoric acid, and other substances upon aluminium, an alteration of the surface colour of that metal can be produced, caused by the elimination of certain impurities through the above-named agents, suggested the attempt to produce a similar result by a treatment resulting in the partial solution of the aluminium, but without interfering with the impurities contained in it. Such a treatment, which is generally accompanied by a brownish discoloration of the metal, consists in the action upon aluminium of very dilute solutions of ammonia, or of ammonia and ammonium salts. In the first case dark brown tints are obtained, in the second case light brown ones. The reaction taking place appears to be expressed by the equation—

$$2Al + 6NH_4OH = Al_2(ONH_4)_6 + 6H,$$

the soluble ammonium aluminate subsequently splitting up into insoluble alumina and ammonia.—C. O. W.

Gold and Silver, Estimation of, in Auriferous Minerals. P. Truchot. Ann. Chim. Anal. Appliq. 1896, 1, 365.

See under XXIII., *page* 66.

Commercial Copper, Analysis of, by the Electrolytic Method. A. Hollard. Comptes rend. 123, 1003.

See under XXIII., *page* 66.

PATENTS.

Cast Steel and Homogeneous Iron [Furnace Lining], Impts. in the Manufacture of. J. Gjers, Middlesbrough-on-Tees. Eng. Pat. 22,727, Nov. 27, 1895.

An ordinary open-hearth steel-melting furnace is lined on its bottom and sides with a rich natural or artificial iron oxide on a silicious or magnesia foundation. If a granulated iron ore be used, it is damped with brine to ensure a caked and solid bottom. The furnace may be charged with crude pig-iron, either cold or run in fluid, and the temperature is high enough to maintain the charge in the liquid state after its conversion into steel, which, after tempering with ferro-manganese or ferro-silicon, is run off into ingots. It is stated that crude pig-iron containing much or little phosphorus may, with equal advantage, be boiled, as described, on a rich iron cinder bottom. Reference is made to the inventor's patents of 1867, 3140; 1894, 2720; 1895, 10,815 (this Journal, 1896, 457); and 1896, 123 (abstract following).—E. S.

Cast Steel and Homogeneous Iron [Duplex Furnace], Impts. in Furnaces for the Manufacture of. J. Gjers, Middlesbrough-on-Tees. Eng. Pat. 123, Jan. 2, 1896.

A duplex furnace is described, consisting of two open-hearth furnaces, arranged at a small angle side by side, connected at the rear ends by a semi-circular flue, and having a combustion chamber in front of each furnace, each chamber being surmounted by a regenerator, preferably of the cellular type, with reversing mechanism. The bottoms and linings of the furnaces may be of iron oxide, as described in the previous abstract of Eng. Pat. 22,727, 1895, or they may be of acid or basic material. A fan is used to supply a mild air blast. In working, the hot gases from one combustion chamber pass into the corresponding furnace, melting the material upon the bed, and then traverse the connecting flue and melt crude pig-iron on the bed of the second furnace, whilst the charge in the first furnace is being converted into finished steel. The fire gases then pass through the second combustion chamber, in which the fuel has been allowed to burn low, and through the regenerator above, which they heat, whence they have exit to a chimney. The reversing takes place when the crude pig-iron has been brought to the melting stage in one furnace, and the finished steel been tapped from the other furnace. A modified duplex furnace is also shown, in which each combustion chamber is provided at its forward end with a number of passages through which producer or other combustible gas can be admitted from an underground gas flue, thus dispensing with solid fuel without disturbing the general arrangements.—E. S.

Iron and Steel and other Metals, Process for the Direct Production of, from their Ores. E. J. M. Servais, Luxemburg, and P. Gredt, Esch-on-the-Alzette, Luxemburg. Eng. Pat. 2997, Feb. 10, 1896.

This is a "process for the direct production of iron and steel and other metals from their ores by smelting the latter under admixture of easily fusible reducing bodies, fats, oils, resins (for example, petroleum and petroleum residues) under partial or complete exclusion of the air, or by the introduction of the mixture of the ores with the reducing substances into the liquid-bath in the melting furnace, or by melting the spongy iron produced from iron ore by the reduction by means of oils or reducing gases, such spongy iron being protected against oxidation by a coating of a protective compound of iron with nitrogen or other suitable element, or by the introduction of the spongy iron produced, into the liquid-bath of the smelting furnace, whether continuously by means of a feeding worm or intermittently by a piston."—E. S.

Amalgam, An Improved [Copper-cadmium Amalgam] for the filling of Hollow Teeth. W. Randler, Friedland, Mecklenburg. Eng. Pat. 23,050, Dec. 2, 1895.

Precipitated copper is mixed with mercury and 1 to 1½ per cent. of cadmium is added so as to hasten its hardening. —J. H. C.

Metallurgical Furnaces [Annealing tubes], Impts. in. G. Platt, Wednesbury, Stafford. Eng. Pat. 23,385, Dec. 6, 1895.

The furnace is a combination of a fire-place, an annealing chamber, and of drying stoves, one on either side of a central flue, which the fire-gases enter, after their passage through a pair of lower flues and the annealing chamber. The apparatus is applicable to annealing and drying seamless steel and other metal tubes, rods, bars, sheets, &c. The annealing chamber is placed immediately above the two lower flues, and is surmounted by drying stoves and the upper central flue. When the annealing chamber is required for close annealing, it is heated by side flues as well as by those beneath, the gases in this case passing on to the upper flues without traversing the annealing chamber. —E. S.

Nickel Ores, and other Materials containing Nickel, Impts. in Apparatus for treating with Carbon Monoxide. L. Mond, London. Eng. Pat. 23,665, Dec. 10, 1895.

The apparatus consists mainly of two upright cylinders containing superposed trays, down the series of which nickel ore is caused to descend to the bottom. The first cylinder, into which the ore is first charged, receives, near the bottom, a current of a reducing gas, such as water-gas, or producer gas containing hydrogen, which traverses the column. A compartment beneath each of the upper trays is connected by a side flue to a stack-shaped combustion chamber, from which heating gases enter, and by another flue to a chimney, so that the heating gases do not mingle with the reducing gases led up the cylinder. The lower compartments are provided with taps connecting with a water or air supply for cooling the descending ore. The second cylinder, or "volatiliser," receives the ore, now containing metallic nickel, from the reducing cylinder, and this is caused to descend from tray to tray, whilst a stream of carbon monoxide is led into the lower part of the volatiliser, and is taken from the upper part into cooling chambers, in which nickel is deposited from the gaseous compound formed. A pair of conveying columns is set between the two cylinders, receiving respectively, by suitable mechanism, the ore discharged from the bottoms, and delivering the same to the top of the opposite cylinder, so that the ore is subjected successively to repeated alternate treatment in the two cylinders. A special rotary feed valve is described and shown, to enable this interchange to be effected without permitting mixture of the different gases employed. (Eng. Pat. 23,665 A, Dec. 16, 1895. See page 34.)—E. S.

The Treatment of Metals [Alloying with Chromium], Impts. relating to, and to Products resulting therefrom. E. Placet, Paris. Eng. Pat. 202, Jan. 3, 1896.

Pure chromium, coated with another metal (such as copper, nickel, aluminium, gold, silver, &c.), is introduced into the fused metal or alloy with which it is desired to alloy it. The purpose of this coating is to prevent—at the moment of its introduction into the molten metal or alloy—the oxidation of the chromium. One or other of several specified metals may be simultaneously introduced. The solution of the chromium in the fused metal is facilitated, it is said, by the addition of auxiliary metals, and also of certain oxides and salts. The improved properties of the chromium alloys thus formed in respect to various applications, are described.—E. S.

Caustic Soda, Carbonate of Soda, and Sulphide of Sodium, Impts. in the Manufacture of. H. R. Angel, London. Eng. Pat. 335, Jan. 6, 1896.

See under VII., *page* 43.

Gold, Silver, or Platinum, Treatment of Antimonial and Arsenical Ores, Materials, or Furnace Products, containing. W. Blackmore, Sheffield. Eng. Pat. 519, Jan. 8, 1896.

The material is fused with an alkali or alkaline compound, such as carbonate of soda, together with sulphur or some suitable sulphur compound. A reverberatory furnace may be employed at a dull red heat. The fused mass is treated with water when the arsenic and antimony go into solution, and the precious metals remain in the residues and may be recovered by smelting.—J. H. C.

Gold and other Metals, Impts. relating to the Extraction of, from Ores, Tailings, Slimes, and other Compounds containing the same, to Apparatus therefor, and to the Preparation of Solvents for Use in such Extraction. H. R. Lewis, London, and C. GelsthARP, Manchester. Eng. Pat. 23,052, Dec. 2, 1896.

The material in a fine state of division is treated with a solution containing hypochlorous or hypobromous acid or other oxy-acid of chlorine or bromine, with or without admixture of other suitable chloride or bromide: or it is mixed with bleaching powder or bleaching liquor, and carbonic, acetic or other suitable acid is added; or it is mixed with a finely divided carbonate, sulphate, phosphate, arseniate, or oxide and water, and then gaseous or liquid chlorine or bromine, or hypochlorous or hypobromous acid is added. The mixture is agitated when necessary until all the gold is dissolved. The gold is precipitated from the solution by carbon, sulphuretted hydrogen or other suitable precipitant; or by means of a current of electricity, in which case the chlorine, hypochlorous, or hypobromous acid, liberated at the anode, may be conveyed into tanks containing milk of lime, so as to be employed again. If the material contain silver, salt or other suitable chloride may be employed so as to dissolve the whole of it with the gold, or if little or no chlorides are present, the hypochlorous acid will convert it into silver chloride, which will remain with the solid residue, from which it may be removed, after separating the gold solution, by means of a strong solution of salt or by hyposulphite of soda [thiosulphate] solution, and precipitated by electrolysis or other means.—J. H. C.

Precious Metals, Impts. in or relating to the Extraction of, from their Ores. W. H. James, and C. J. Norris, London. Eng. Pat. 23,492, Dec. 7, 1896.

The substance obtained by saturation of bromine with the oxides of nitrogen (called "nitrobromine"); or the somewhat similar substances formed "when any halogen is acted on by the oxides of nitrogen"; or a "substance formed by heating to dull redness equal weights of bleaching powder and potassium cyanide," or "potassium hypochlorite or hypobromite, with or without the addition of hypochlorous or hypobromous acid" is used alone or added to the cyanide solution ordinarily employed in treating gold ores so as to increase its solvent action.—J. H. C.

Extracting Metals [Amalgamation], Impts. in Apparatus for. E. Fischer, Breslau, and C. G. Penney, London. Eng. Pat. 19,883, Sept. 9, 1896.

The mercury is heated in a retort, heated air being forced in at the same time. The mixture so produced is conducted by a pipe covered with a heat insulator, and returned axially into a slightly inclined drum and cylinder which are made to rotate. Within the cylinder the pipe is perforated. The wet or dry material to be treated is introduced at the upper end of the cylinder and gradually makes its way into the drum where it comes in contact with the heated air and mercury vapour, and forms an amalgam which is deposited on the inner surface of the drum. The drum is constructed of copper, its inner surface is amalgamated and made to form the cathode of an electric circuit while an anode of carbon or other suitable material is suspended from the axial pipe.—J. H. C.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(A.)—ELECTRO-CHEMISTRY.

PATENTS.

Electric Battery [Primary], An Improved. P. Falle, Toulouse, France. Eng. Pat. 22,142, Nov. 20, 1895.

The claim is for "An electric battery in which the negative pole is formed of a sheet of zinc immersed in a solution of chloride of sodium or chloride of zinc, the positive pole being formed of a cylinder of porous carbon containing an agglomerate composed of sulphate of zinc, a double sulphate of aluminium and potassium and of potassium bichromate, these substances being ground and mixed to a thick paste with sulphuric acid."

The agglomerate is made up of equal weights (100 grms.) of alum, potassium bichromate, sulphate of zinc, and sulphuric acid.—J. C. R.

Ozone, Impts. in or relating to the Manufacture of and Apparatus therefor [Corrugated Glass Plates]. C. J. Yarnold, Brixton, London. Eng. Pat. 24,289, Dec. 18, 1895.

In this apparatus the gas or air treated, is first purified by means of caustic soda, and then dried and cooled to a temperature of 32° F. It is then passed into an expansion chamber provided with a thermometer, and thence into the ozoniser proper, which consists of a number of compound plates, each formed of a sheet of corrugated glass, enclosing between them a layer of silver foil, which forms a "tab" or extension, connected by means of a screw and washer to its respective terminal. There are two terminals, one on either side of the box. The plates are cemented at their edges, and arranged so that the summits of the waves on one plate are opposite the depressions of the next. Each compound plate is let into the casing of the apparatus, which is formed of insulating material, preferably slate. Their lower edges are placed between ribs on the floor of the casing and their upper edges are insulated from each other by fingers extending from a bar secured to each sleeve. Any oxides which may be formed are dealt with as in Eng. Pat. 12,692, 1895.—G. H. R.

Electrodes [Carbon], Impts. in and relating to, for Use in the Production of the Electric Arc. J. A. Deuther, Boston, U.S.A. Eng. Pat. 20,600, Sept. 17, 1896.

The object is to construct one or both of the opposite electrodes at the point where the arc is formed with a section of carbon in any form (but where found useful, composed of alkaline earths and carbon), which, when consumed, can be replaced by a similar section, without disturbing the other parts of the electrode.—J. C. R.

Electric Furnaces [Calcium Carbide and reduction of Refractory Compounds], Impts. in and relating to. J. A. Deuther, Boston, U.S.A. Eng. Pat. 20,601, Sept. 17, 1896.

The inventor shows an oscillating electrode and mechanism whereby material to be smelted is fed within the path of the arc.—J. C. R.

(B.)—ELECTRO-METALLURGY.

Electro-Chemistry, Inorganic, recent Progress in. C. Chéneveau. Rev. d. Phys. et d. Chimie. **1**, 55—62.

These notes are mostly derived from papers read before section X. at the Congress of Applied Chemistry in Paris, July 1896.

Treatment of Tin Scrap.—The electrolytic recovery of tin from this material, is now carried out with a 10 per cent. sodium hydrate solution containing sodium chloride as an electrolyte. 10 per cent. of the tin remains dissolved in the electrolyte as a stannate. Both the tin and the alkali may be removed from this by chemical methods.

Minet's Aluminium Process.—Data are given of experiments carried out at St. Michel, Savoy. A mixture of 70

per cent. of sodium chloride, and 30 per cent. of a natural or chemically prepared cryolite is used in the bath. Bauxite and aluminium fluoride are fed into it, as the electrolytic process continues. 80 kilos. of this mixture are used as a charge; its melting point is 675° C., 10 h.p. hours are required to produce 1 kilo. of aluminium. The current efficiency is 70 per cent. The voltage is 7·5, of which only 3 volts are actually used in separating the aluminium. A secondary reaction due to the free hydrofluoric acid and the deposited aluminium, has been said to absorb 25 per cent. of the current. Experiments with Heroult's process at Froges do not confirm this. The difference of potential observed at the terminals of the bath, varies with the nature of the electrodes. With electrodes of copper, platinum, and carbon it is *nil*; whilst between carbon and molten aluminium it is at a maximum. It also varies proportionately with the current intensity. $E = 3 \cdot 1\ i$ for a current density below 10 ampères per sq. ft. Above this limit it can be calculated from $E = e + r\,i$, where e = voltage required to decompose the aluminium salt, and r = the resistance of the electrolyte; e = 3 volts at 800° C., and = 2 volts at 1100° C.; r is about 0·00125 ohm.; e and r have been experimentally proved constant for currents between 1,000—4,000 ampères. Minet estimates that 20,000 h.p. is used for aluminium production in France, and that the use of natural power would allow it to be obtained for $\frac{1}{10}$th of the present cost.

Güntz's Lithium Process.—If a mixture of equal parts of potassium and lithium chlorides be electrolysed with a current of 10 ampères at 20 volts and a temperature of 450° C., lithium will be produced. The temperature must be kept above the melting point of lithium chloride, otherwise a subchloride of lithium will be formed from the deposited metal.

Electrolysis of Sodium and Potassium Chloride Solutions.—Minet states that asbestos diaphragms give the best results. As anodes, Peyrusson's silver coated with platinum or iridium, Hoepfner's ferro-silicon and graphitoidal carbon obtained by the methods of Castner and others, are mentioned.

Electrolysis of Fused Salt.—Hulin describes renewed experiments with the Vautin process, carried out at Modane, Savoy. Molten lead forms the cathode in this process. The unsatisfactory results obtained in earlier experiments were due to the formation of subchlorides, and to the liberation of the sodium in globules, which did not alloy rapidly with the molten lead. These difficulties are overcome by electrolysing a definite mixture of sodium and lead chlorides, the bath being continually fed with the same. The anodes are formed of receivers containing lead, and receive only part of the current. A portion of the liberated chlorine acts on the lead, and the lead chloride formed diffuses into the bath. By regulating the current passing through the molten lead anode, the amount of lead chloride formed can be increased or diminished. The best current density is 0·7 ampère per sq. ft., and produces 0·081 kilo. of Cl, and 0·054 kilo. of sodium per h.p. hour.

An E.M.F. of 7 volts is required. Peroxide of lead, and alloys of sodium or barium with tin or zinc, may also be obtained by this method.

Plating on Aluminium.—Margot's process consists in removing the thin film of oxide which causes non-adherence of the deposit, by immersion in a solution of alkaline carbonate, followed by treatment with hot hydrochloric acid (1 in 20) and final immersion in a dilute copper sulphate bath. The ordinary electrolytic process is then proceeded with. The same preliminary operations are necessary, in silvering aluminium by Wagner's process. If the solution used be that of the double cyanide of silver and potassium, the best results are obtained by coating the aluminium first with zinc. Copper cannot be used.

—J. B. C. K.

Copper, Silver, and other Metals, Hoepfner's Electrolytic Process for the Production of, directly from the Ores. Eng. and Mining J. 1896, **62**, [23], 584.

The powdered ores are first leached with a warm solution of cupric chloride, containing some chloride of sodium or calcium, by which the silver, copper, lead, bismuth, and some iron and arsenic are extracted. From the solution containing cuprous chloride, the silver is precipitated by metallic copper, the chloride of lead deposited by cooling, and the antimony, bismuth, and arsenic precipitated by powdered limestone. Finally, the iron, when in excess in the solutions, is separated by the action of air, according to the following equation:—

$$2Cu_2Cl_2 + 2FeCl_2 + 3O = 4CuCl_2 + Fe_2O_3$$

The solution of cuprous chloride thus purified is then conveyed to the electrolytic baths, which contain cells with anodes of artificial carbon and cells with cathodes of sheet copper, separated from each other by diaphragms consisting of linen, cotton, or the like. This solution is divided into two separate currents, of which one brought into contact with the cathodes, loses all or part of its copper contents, whilst the other, brought in contact with the anodes, "is reconverted into a solution of cupric chloride." After leaving the bath the currents are reunited, thus forming a solution of cupric chloride similar to the original one for the treatment of the ore. If zinc or nickel be present in the ores, all or part of it will be extracted and can be electrolytically deposited from the purified solution.

In a series of baths first the copper, then the lead, nickel, and zinc will be deposited. The cost of extracting the various metals is given, and owing to the better electrical conductivity of the chloride solutions as compared with the sulphate hitherto used, twice the amount of copper can be deposited in the same time with the same current. Analysis of the copper thus directly obtained from ores containing considerable impurities, show that it is practically pure.

By means of the process described, neither gold nor the whole of the zinc is extracted. If these metals be present in remunerative quantity the ores or the residue of the ores subjected to the preliminary extraction of copper and lead, are roasted in an ordinary furnace to convert the sulphides into oxides, and the gold is then extracted by chlorine. The oxide of zinc is converted into chloride by means of chloride of calcium or any other convenient means, and the metal electrolytically deposited with the simultaneous production of free chlorine.—A. W.

Slimes (Copper), Present Method of Treating, from the Copper Refineries. T. Ulke. Eng. and Mining J. 1896, **62**, (22), 512.

In the electrolytic method of refining copper, the slimes after screening from scrap copper, constitute about 4 per cent. of the total weight of the anode copper refined, and contain on an average from 15 to 30 per cent. of copper, 45 to 50 per cent. of silver, less than 1 per cent. of gold and from 20 to 35 per cent. of arsenic, antimony, tellurium, and other impurities. The usual dry methods of treatment with various charges in lead refining all have the objection that part of the silver and copper can be recovered only by treating quantities of by-products such as litharge, copper matte, or speiss. The wet method as carried out by the Baltimore Electric Refining Company, consists of treating the screened slimes in lead-lined vats with dilute sulphuric acid [1 to 4], through which air is injected, for two or three hours. The solution, containing arsenic, copper, and most of the impurities, is siphoned from the residue, which contains lead sulphate, tellurium, a little bismuth and antimony, in conjunction with the silver and trace of gold. This residue is melted on a cupel hearth, at first without any flux, when a brownish slag containing lead and antimony, together with some silver prills, is skimmed off. The slag, after cooling, is picked over to separate the silver and is then added to molten lead in the cupelling furnace, by which means the last traces of gold and silver are removed therefrom. The second stage of treating the slimes, after removing the brown slag, consists of heating with nitre, which extracts the tellurium. The silver metal on the cupel is now practically pure with the exception of a little copper, and is cast into bars ready for parting in the usual way. The copper in the acid solution obtained by the first treatment is recovered by means of scrap iron.—A. W.

Separation of Metals, Electrolytic, by Use of Soluble Anodes. R. Pauli. Elektro-chem. Zeits. 1896, **3**, 180.

The author notes that in the Daniell type of cell, the solution of the one electrode and deposition of a metal on the other, depends upon the relative osmotic pressure of the two metals towards the electrolyte used. If a solution of mixed salts surround the cathode, that metal will be first deposited which for the given electrolyte, exerts the least osmotic pressure. A separation of metals is thus possible without use of dynamos: and as a current will be at the same time produced in the depositing cell, this method may in some cases be cheaper than the usual one.—J. B. C. K.

Copper Refining, Electrolytic, in the United States. G. Kroupa. Oesterr. Zeits. Berg. u. Hüttenw. 1896, **55**, 541.

A comparison of the "multiple" and "series" systems of vat-working. In the former the vertical electrodes are hung on copper bars, which serve as current conductors and run along the length of the vats. In the series system, only the first and last plates in a vat are connected to the current conductors; all the intervening ones being insulated. Although the latter arrangement has been much experimented with in the United States, it is now being given up. Barnett states that in comparing the working of these two systems, one must note (*a*) Current efficiency; (*b*) Amount of copper locked up in the vats; (*c*) Working costs; (*d*) Capital outlay on plant. The multiple system (M.) requires low E.M.F. and gives 95 per cent. of efficiency; the series system (S.) requires high E.M.F., and, owing to short-circuiting through the vat walls and silver mud, the current efficiency is only 85—90 per cent. The locked-up copper is only half so great with (M.) as with (S.), whilst much more impure anode copper may be safely used. The capital outlay is greater with (M.) than with (S.) Balancing these various items, the total cost with (M.) is found to be about 8s. 4*d*. per ton of refined copper less than with (S.) The Anaconda Refinery has changed its entire system from the series to the multiple.—J. B. C. K.

PATENTS.

Metalliferous Ores and Products, An Improved Process for the Treatment of. [*Matte Anodes.*] E. A. Ashcroft, Grays, England. Eng. Pat. 14,783, Aug. 3, 1895.

The process, which is an improvement on that described in Eng. Pats. 13,850, 1894, and 13,534, 1895, consists in the employment of anodes composed of iron or copper mattes in a zinc-depositing bath, which has been prepared by leaching roasted zinc ores with a solution of ferric salts. The solutions of copper and iron thus obtained are electrolysed in vats provided with diaphragms between carbon anodes and copper cathodes, where the copper is deposited, and all the iron raised to the ferric state, and the solution is thus regenerated ready for leaching a fresh quantity of zinc ore. The copper may be removed from the solution by the simple cementation process of the contact of metallic iron, or soluble iron anodes may be employed in the electro-deposition of the copper, in which case the electro-motive force developed, is utilised for the deposition of the zinc by suitably connecting the baths. If pure zinc be required, a diaphragm must be dispensed with in those cases where the product desired is an alloy of the different metals. The proportion of the metallic salts in solution is then maintained sufficiently constant to ensure a uniform product. —G. H. R.

Zinc, Impts. connected with the Electrical Deposition of. R. Heathfield and W. S. Rawson, London. Eng. Pat. 23,097, Dec. 3, 1895.

The zinc-iron couple employed for enriching the alkaline bath, as described in Eng. Pat. 16,105 (this Journal, 1895, 874), is, according to this improvement, hung on to the side of the anode remote from the cathode, and is so connected that, by means of a switch, it may be put out of or into circuit, according to whether the current is passing from the anode to the cathode or not. The anodes are constructed of perforated iron or iron gauze, through which the electrolyte can freely pass, and if two cathodes be employed, the anode is made double, and the basket filled with scrap zinc, or zinc plates connected with iron plates are suspended between them so that it may be put in or out of circuit as desired. Rotating anodes may be employed as described in Eng. Pat. 20,636, 1894 (this Journal, 1895, 871), and the cathodes are suspended between the anodes in the usual way, and are preferably kept in motion.—G. H. R.

Metals, Impts. in the Electro-Deposition of. [*Jet Electrolyte.*] J. C. Graham, London. Eng. Pat. 986, Jan. 14, 1896.

The electrolyte is contained in a tank placed from 1 to 2 ft. above the depositing bath, and is delivered against the face of the cathode in a jet. If the jet is delivered from a pipe $\frac{3}{8}$ in. in diameter, placed $1\frac{1}{2}$ in. from the cathode, and using as the electrolyte a nearly saturated solution of copper sulphate containing 5 oz. of commercial sulphuric acid to 1 cb. ft. of water, a good deposit is obtained on the cathode over the area controlled by the jet, which is a circle of 5 in. in diameter, and the current density may be as high as 300 ampères per square foot. Beyond the controlled area the cathode is discoloured, and the deposit non-coherent, so that a sufficient number of jets must be employed to control the required cathode surface. The anode, which is copper, should have a surface eight or nine times greater than that of the cathode, and is preferably given a spiral corrugated or grid-like form, and the jets are arranged so as to play through it on to the cathodes. To prevent the formation of a non-coherent growth at the edges of the cathode, they are protected by shields of some insulating material, between which and the cathode face, the electrolyte flows.—G. H. R.

XII.—FATS, OILS, AND SOAP.

Soap made with Carbon Tetrachloride [*for Dry-Cleaning*]. L. Axtheim. Leipziger Färber- u. Zeugdrucker Zeit. **45**, 519.

A substance has long been sought, equal in cleansing properties to petroleum spirit, and possessing the advantage of non-inflammability. Such a substance is carbon tetrachloride, a colourless liquid (of sp. gr. 1·63, and boiling at 76° C.) which is not only non-inflammable, but extinguishes a flame when poured upon it.

O. Schöning, of Bromberg, has succeeded in preparing a soap containing this substance, and the addition of this soap to petroleum spirit, in which it is readily soluble, greatly diminishes the inflammability of the latter; such a solution containing 10 grms. of soap per litre forms, it is stated, an excellent cleansing liquid.—R. B. B.

Strophanthus Hispidus, Oil of the Seeds of. A. Mjoen. Arch. der Pharm. **234**, 283.

See under XXIV., *page* 72.

PATENTS.

Cleansing and Recovering Grease and Oil from Cotton and other Waste and Wool, A Method of and Apparatus for. G. E. Wright, Nottingham, and W. Monk, Nottingham. Eng. Pat. 22,183, Nov. 21, 1895.

See under V., *page* 41.

Degreasing and Cleansing Wool, Cotton Waste, and like Fibrous Material: Impts. in Apparatus for. F. N. Turney, Nottingham. Eng. Pat. 1370, Jan. 20, 1896.

See under V., *page* 41.

Cocoa or Coprah Oil, Manufacture of an Edible Fat from. M. C. A. Ruffin, Paris. Eng. Pat. 1827, Jan. 25, 1896.

The oil is heated to its melting point (about 23° C.) and allowed to stand for 48 hours. A large proportion crystallises out and is separated from the liquid portion by means of a filter press, dry cakes being thus obtained amounting to about 45 per cent. of the mass treated. These cakes are then melted again and purified by treatment with lime, the lime emulsion being evaporated in a vacuum pan furnished with an agitator. The temperature must not be over 20°—25° C. The lime soap forms in granules, from which

the melted fat can be removed by pressure. The final product melts at 31° C.

To remove insoluble fatty acids from the fat thus prepared, the latter is melted, treated with quicklime, and the little water it contains, evaporated off in a vacuum pan at a temperature of 20° to 25° C. The lime salts, together with the excess of quicklime, separate out completely as soon as all moisture has disappeared.—W. P. S.

Raw Wool Fat, Impts. in Processes for Producing Chemical Combinations from. [*Use of Alcoholic Ammonia.*] C. Schmidt, Magdeburg. Eng. Pat. 17,379, Aug. 6, 1896.

By treating raw wool fat with alcoholic ammonia at a temperature of 100° C., solution of the fat is obtained. On cooling, a portion is deposited consisting of ammonium salts of fatty acids, and fatty alcohols, whilst free fatty acid remains dissolved.

The separation of the deposit into nearly pure "sebacic" fatty acid and fatty alcohols mixed with a little sebacic acid, is carried out by treating the deposit with acetone, amyl alcohol (fusel oil), ether, chloroform, &c. The soaps are soluble with difficulty in these liquids, and by recrystallisation a product may be obtained, melting at 60° to 75° C., and consisting chiefly of sebacic acids.

The impure fatty alcohols are clarified by boiling and by kneading with dilute alcohol and finally with water. They may also be purified by washing the soaps out of the solutions of the fatty alcohols, or by distillation. Mixtures of the fatty alcohols of wool-fat with water are used in pharmacy under the name of "lanoglycerin."—W. P. S.

Soaps, An Impt. in the Manufacture of. [*Admixture of Ground Oyster Shells.*] F. M. Sanders, Birmingham, and The Digby Patents Company, Birmingham. Eng. Pat. 19,257, Sept. 1, 1896.—W. P. S.

Distilling Apparatus, Impts. in. [*Glycerin Still, &c.*] J. V. Ruymbeke and W. F. Jobbins, both of Aurora, Kane Co., Ill. Eng. Pat. 20,982, Sept. 22, 1896.

Two distinct distilling plants are employed, each comprising a still, a series of separating drums, and one or more condensers. The necessary vacuum is produced in the apparatus by means of "a valved mechanical vacuum pump, common to both vessels, for maintaining a vacuum therein, and a second valved mechanical vacuum pump connected with and supplementing the action of the first vacuum pump." The crude material is placed in the first still and distilled by means of steam, &c., the vapours being condensed in the separating drums and condensers. The liquor condensed in the first drum consists of glycerin and impurities, and may be returned to the still: that in the succeeding drums is of sufficient purity to be used in the manufacture of dynamite, or it may be further purified by redistillation. The liquors condensed in the last separating drum and in the condensers, consist essentially of glycerin and water, and are collected in "sweet water" drums, from which they are ultimately removed and concentrated. The concentrated "sweet waters" are then introduced into the second still and distilled, as in the first apparatus, pure glycerin being collected in the intermediate separating drums, and water, with any glycerin which may be carried over, in the last drum and condenser. Collecting drums provided with suitable taps are arranged beneath the separating drums, to enable the contents of the latter to be drawn off without destroying the vacuum. The apparatus can also be used as a fractionating plant for separating glycol from the glycerin.—R. A.

XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

(*A.*)—PIGMENTS, PAINTS.

PATENTS.

Varnish, A Process of Manufacturing. C. W. Jurany, Vienna. Eng. Pat. 23,262, Dec. 4, 1895.

A "PROCESS of manufacturing white varnish, consisting in mixing sulphide of zinc colour containing 40—70 per cent. of ZnS and only 30 per cent. of BaS, in pure alcohol to paste form, the superfluous alcohol being pressed out or expelled by distillation." Coloured varnish is made by mixing aniline colours with the preceding. The preceding products "are then dissolved in alcohol or benzene varnish."—F. H. L.

White Lead for White-Lead Paint, Impts. relating to the Manufacture of [*without Stove-drying*]. C. D. Ismay, Newcastle-on-Tyne. Eng. Pat. 23,969, Dec. 14, 1895.

To avoid the necessity for stove-heating, and the dangers of handling the dry powder, white lead is often ground into oil while still wet with water; but it is found difficult to remove the last traces of the latter liquid, the presence of which is very detrimental to the finished material. According to the present invention, the wet pigment is mixed with the oil in a steam-jacketed covered pug-mill under reduced pressure and at a temperature of 65° to 120° C.: so that most of the water is evaporated during the process, the remainder being removed by conducting the final grinding on a roller mill having hollow rolls supplied with steam. —F. H. L.

Paint [*Ferric Oxide*], *Impts. in the Production of.* C. Sullivan, Neath, South Wales. Eng. Pat. 2219, Jan. 30, 1896.

THE waste (sulphuric) acid of tinworks is evaporated to dryness and the product furnaced to yield the pigment for an oxide paint.—F. H. L.

(*B.*)—RESINS, VARNISHES.

Sagapenum, Composition of. Hohenadel. Arch. der Pharm. 1895, **33**, 259.

THIS gum resin contains: essential oil, 5·8 per cent.; resin soluble in ether, 56·8; gum, 23·3; impurities, 10·6; and water, 3·5 per cent.

The essential oil, obtained by steam distillation, is easily soluble in ether, petroleum spirit, glacial acetic acid, chloroform, acetone, benzene; less readily in acetic ether and alcohol. The alcoholic solution, on the addition of hydrochloric acid, becomes violet, even in the cold; thus this reaction given by tincture of sagapenum is due to a constituent of the essential oil. The oil contains 9·7 per cent. of sulphur, valeric acid, and perhaps borneol valerate.

The resin, obtained by extracting with ether, distilling off the solvent, and then driving off the essential oil with steam, with the exception of 0·11 per cent. of free umbelliferone, is entirely composed of umbelliferone (15·7 per cent.) combined with the alcohol, $C_{24}H_{27}O_4(OH)$, sagaresinotannol. This ester, treated with nitric acid, gives oxypicric and not camphoric acid. The gum has not been examined. —A. C. W.

Copal, Zanzibar. A. Stephan. Pharm. J. 1896, **57**, 525.

THE author examined specimens of this variety of copal sent from Bagamoyo, in East Africa. The copal, finely powdered, melts at about 140° C.: it is slowly but completely soluble in alcohol: benzene, chloroform, and glacial acetic acid dissolve about 30 per cent., ether about 34 per cent., and petroleum spirit and carbon bisulphide about 10 per cent. When boiled with alcohol, the resin caked and only a slight proportion dissolved, but by repeated digestion with alcohol, it could be brought entirely into solution and then reprecipitated with water. The resin, thus purified, was more soluble in the menstrua previously mentioned, and was also soluble in very dilute boiling potash solution (0·1 per cent.). It appeared to consist of resin-acids, the principal of which, constituting about 80 per cent. of the resin, was called trachylolic acid. This acid was obtained with difficulty in minute sphero-crystalline masses, melting at 165° C. From it the potassium, copper, and iron salts were prepared. A second acid, called isotrachylolic acid, present to the extent of about 4 per cent., was also obtained. These two acids, together with about 6 per cent. of α-copal resin and β-copal resin, a bitter principle and volatile oil, were the only constituents the author succeeded in separating. —A. S.

(C.)—INDIA-RUBBER, &c.

Balata Industry of Dutch Guiana. J. Soc. Arts, **44**, 908.

Balata is obtained from the bullet or bully tree, *Mimusops Balata*, a large forest tree of the order *Sapotaceæ*. The tree reaches maturity at a height of 120 ft., with usually a large spreading head, a trunk 60 to 70 ft. high, and 4 to 5 ft. in diameter; but a tree with a trunk above about 30 ins. in thickness is generally not considered worth "bleeding." Balata commands a higher price than gutta-percha—in fact, it is used as a superior variety thereof. Nevertheless some of its physical properties show that balata is distinctly different from gutta-percha, though its chemical composition is probably quite identical therewith, as also with that of caoutchouc. A very marked difference between gutta-percha and balata is be found in the fact that whereas the former, when exposed to light and air, soon becomes altered on the surface and changed into a brittle resinous substance, the latter is but slowly acted on under the conditions named.—C. O. W.

PATENTS.

Gutta-Percha, Improved Process of Obtaining and Purifying. E. Sérullas and F. Hourant, Suresnes, France. Eng. Pat. 654, Jan. 9, 1896.

The raw material is first purified by treatment with hot or cold water and alkali (preferably under pressure), and then is extracted with a solvent, such as toluene. Instead of removing this by distillation, however, the gutta-percha is precipitated, either by the addition of acetone or by lowering the temperature of the solution, the deposit being finally washed with acetone in a filter press. It is claimed that, whilst distillation or evaporation of the solvent injures the product, separation of the latter from the solvent by precipitation preserves all its good qualities whilst eliminating all other resinous matters, such as chlorophyll and the like. —F. H. L.

Waterproof Fabrics, Impts. in the Printing of, and Apparatus therefor. J. Berry, Salford, Eng. Pat. 1375, Jan. 20, 1896.

See under VI., *page* 42.

Vulcanising Apparatus, An Impt. in. P. J. Davis, Rochester, Monroe Co., N.Y. Eng. Pat. 20,959, Sept. 22, 1896.

The object of the invention is to produce a vulcanising apparatus in which the fastening devices for the cover of the retort are easily managed and readily adjustable.

XIV.—TANNING, LEATHER, GLUE, SIZE.

Fermentation Phenomena in Tan Liquors. F. Andreasch, Imp. Research Laboratory, Vienna. Der. Gerber, **21**, [506], **22**, [513], 15.

The author divides the third group (see this Journal, 1896, 911, final paragraph) into three sub-divisions: 1. Alcoholic ferments. 2. Acetic acid ferments. 3. Lactic acid ferments.

As he confines his researches to this group, we may consider his work in two parts: (1) General considerations and results of research in the above three sub-divisions; and (2) Details of researches of the fermentation of typical tan liquors.

Alcoholic Ferments.—The alcoholic fermentation in tan liquors is produced by various yeasts, principally bottom-fermentation species. Other yeasts form a film on the surface of standing liquors. Some concentrated extracts, such as pine-bark extract, are fermented by yeasts, with evolution of CO_2. Myrabolans liquors mashed cold contain much finely divided material and an easily fermentable sugar from the myrabolam rind; in these the alcoholic fermentation is generally marked. The constitution of the glucosides, which form a large part of the non-tannin of the liquors, is little known. Dextrose, however, is found, and this splits up into alcohol and CO_2. The amount of alcohol is nearly always under 2 per cent., but in abnormal cases may reach 3 per cent. In the majority of cases the alcohol is changed into acetic acid almost as fast as produced.

The following yeasts have been found in tan liquors:—

Saccharomyces pastorianus (Hansen), S. ellipsoïdeus II. (Hansen), S. apiculatus (Reess), S. ellipsoïdeus I. (Hansen), and S. acidi lactici (Grotenfeld).

The two first and most frequently occurring, which are disease organisms in beer, are the principal cause of alcoholic fermentation in the tannery.

In addition the following fungi occur—Rose-coloured torula and orange-yellow torula.

These form small quantities of alcohol, but are only of botanical interest; the orange-yellow torula was first isolated from old mixed liquors by the author.

2. *Acetic Acid Fermentation.*—The author has found no organisms in tan liquors capable of fermenting carbohydrates direct to acetic acid. Such fermentations are theoretically possible, but can only be considered as exceptional. In the very numerous tan liquors examined, alcohol was always the primary product, this being oxidised to acetic acid. The chief organisms causing this oxidation are Bacterium aceti (Hansen), B. Pasteurianum (Hansen). The third species, B. Kützingianum, he has not hitherto found in tan liquors.

The two first differ in their behaviour toward iodine solution. B. Pasteurianum is stained blue by iodine; B. aceti only yellow. Their action on nutrient solutions containing small quantities of alcohol is about equal.

Pellicles of B. aceti and B. Pasteurianum are not often seen on tan liquors, being broken up by the moving of the liquor and the raising of the skins; their formation is also interfered with by Saccharomycetes and various forms of mycoderma, which, at the usual tannery temperature, develop much more quickly than acetic acid bacteria. The acetic fermentation does not go on at a less temperature than 12° C. It is possible by sowing the requisite organisms in the liquor to induce the proper acidity, which is sometimes prevented by the absence of alcoholic ferments. In a tan liquor 20 months old, containing a normal amount of tannin, and non-tannin, and acidity, but no acetic acid, a bacteriological examination showed the absence of alcoholic and acetic ferments, and it was evident that the lactic organisms had ceased to grow for want of albuminous material. The liquor, however, contained abundant *mycoderma* and *kefir* organisms; milk inoculated from it underwent a vigorous fermentation with copious evolution of CO_2. Fresh tan liquor inoculated from the same, developed only mycoderma; no acetic acid was formed for want of alcohol, though doubtless the spores of acetic bacteria were present. This is frequently the case in practice, and the addition of sour liquor to a fresh tannin infusion, instead of souring it, will sometimes actually prevent the souring.

Amongst the fungi found in the liquors, the various forms of *mycoderma* are of importance here, as they not only decompose the alcohol, but also the acetic acid; they ferment dextrose, levulose, invert sugar, and cane sugar, using albuminous bodies and even simple ammonia compounds as nitrogenous nutrient material, whereas the true yeasts draw their nitrogen from amides and peptones, and only assimilate albumins with difficulty. The CO_2 produced by the mycoderma has only a very slight swelling effect on the hide fibres. The chief cause of injury is that these organisms overgrow most others in a sweet liquor, and hinder or prevent the acetic fermentation; the most favourable temperature for their growth is 12°—20° C. whereas the optimum for *B. Aceti* is 33°—37°.

As showing the rapidity of development of mycoderma, the author has observed the formation of a thick pellicle in a single summer night on the weak liquors of an American sole-leather tannery where the hides are moved during the day by "rockers." *B. aceti* grows best in a solution containing 2·5 per cent. of alcohol and 1·5 per cent. of acetic acid, as well as the usual nutrient materials. No tan liquors, of course, contain so large a quantity of acid; but acid is necessary for the growth of the organism, which grows with difficulty in a neutral medium. The tanner cannot regulate the acetic fermentation of his liquors like the vinegar manufacturer, but by approaching as near as possible to the best conditions for the growth of the

bacteria, he may greatly influence the fermentations to his advantage.

Summing up the processes connected with the acetic acid fermentation of tan liquors, we have:—

(1.) Action of *Saccharomycetes.*—Sugars fermented to alcohol and CO_2.

(2.) Action of acetic bacteria.—Alcohol fermented to acetic acid and water.

(3.) Action of *Mycoderma.*—Alcohol and acetic acid split up directly into CO_2 and water.

Lactic acid fermentation is somewhat more complicated than acetic acid fermentation, and continues after the cessation of the latter.

A large number of lactic ferments occur in tan liquors, some of which set up a pure fermentation; others cause, in addition to the production of lactic acid, further changes, which may be considered as diseases of the liquors.

In the lactic acid fermentation of milk, the sugar is decomposed with evolution of a little gas, the caseïn is coagulated by the acid formed, and in the case of a pure fermentation, no further change takes place. In tan liquors, the sugars are also decomposed with formation of lactic acid, but the fermentation is complicated by the presence of yeasts and acetic bacteria, besides other organisms.

Certain lactic bacteria cause the liquors to become slimy—an evil which delays the tanning process by making diffusion slower. The age of the liquors, their concentration, the kind of tannin, and the condition of the hides, determine the species of lactic organism present.

In the same tannery different species of lactic bacteria were found in the different liquors, and mostly in a state of comparative purity.

In place of the caseïn in milk, the peptones formed from the hide substance by other bacteria previously mentioned (see this Journal, 1896, 911), and in some cases the gelatinous substance of the hide itself, supply the nitrogenous food for the bacteria. The spontaneous lactic acid fermentation as it takes place in milk is of most importance: and if a large amount of acidity be required in the liquors, such organisms should be introduced as produce acid rapidly and are not influenced by the presence of the acetic acid previously formed.

The list of organisms given below are those which the author has found in tan liquors, though there are doubtless other species he has not met with. There are also varieties of these types.

He divides them into four groups:—

A. Lactic acid bacteria of milk.

Bacillus acidi lactici (Hueppe).

Bacterium acidi lactici (Grotenfeldt).

Bacterium lactis acidi (Marpmann).

All three species may occur in normal tan liquors.

B. Lactic acid bacteria which cause the ripening of cheese.

Bacillus XIX. (Adametz) from old liquors containing much peptone. (Found twice.)

Bacillus α (Freudenreich), or a variety of the same.

Lactic acid—forming tyrothrix (Duclaux) from very old sole-leather liquors, and a first weak liquor ("Stinkfarbe").

C. Specific bacteria of lactic fermentation of tan liquors, isolated from tan liquors by plate cultures with milk-serum gelatin, and cultivated by Hansen's method.

Lactic acid bacterium of tan liquors I.

Lactic acid bacterium of tan liquors II.

Lactic acid bacilli of tan liquors *a* and *b*.

Lactic acid micrococcus of tan liquors *a*.

D. Lactic acid yeasts.

Saccharomyces acidi lactici (Grotenfeldt).

Lactic acid yeasts of tan liquors α, β, γ.

All three forms are of equal activity, and were first cultivated from old pine-bark liquors.

The following accessory lactic acid bacteria also occur:—

Bacterium acidi lactici (Pasteur), identified from the description of Boutroux, Duclaux, and Zopf; found in pure myrabolans liquor after undergoing alcoholic fermentation.

Gas-forming, liquefying lactic acid bacillus, from upper leather weak liquors after the dung bate.

Bacillus lactis viscosus (Adametz), causes slimy or ropy liquors, repeatedly isolated from the liquors of tanneries, using river water in which the Adametz bacillus was found.

A more exact description of the new lactic bacteria isolated by the author, Bacterium 1 and 2. Bacillis α and β, and Micrococcus α, will be given in another place.

Groups A., C., and D. may be considered as normal producers of lactic fermentation; Group B., especially Tyrothrix, generally occurs in bad liquors, containing much albuminous matter in solution. The presence of any organisms of Group B. may be regarded as an indication that the liquors are going wrong.

Group A. does not grow so well in tan liquors as in milk; on the other hand, the yeasts of Group D. are the principal causes of sound lactic fermentation of tan liquors, no doubt owing to the fact that the presence of large amounts of tannin does not hinder their development. In general, one or two species preponderate in a given liquor, depending on its character (upper or sole, old or new liquor) and on the amount of undecomposed sugars and nitrogenous compounds.

The hides, owing to their nitrogenous contents, have a far greater influence on the lactic than on the acetic fermentation, where only small quantities of nitrogenous nutriment are required. The author finds that the lactic acid bacteria draw upon the hide substance for their nitrogen, either in the unaltered form of the intercelluiar substance, or the same peptonized by putrefactive bacteria.

The influence of albuminoids on the activity of the lactic acid yeasts and bacteria explains the well-known fact, that in old liquors where such materials are present, the amount of lactic acid is greater, and it also explains why the maximum formation of lactic acid in tan liquors occurs much later than in milk, because the necessary nutrient materials are not present in so large amount, but are supplied gradually to the liquor. For the same reason, the addition of a drop of sour milk to sterile neutral tannin infusion, sets up lactic fermentation with difficulty, but if ammonium salts and phosphates be added, the fermentation proceeds rapidly.

The most favourable temperature for lactic fermentation lies between 30° and 35°, but it can also go on at 12°. This optimum is naturally never reached in tan pits, but in summer these bacteria will frequently overcome the other ferments, producing then a much purer lactic fermentation. Liquors prepared in summer contain a much greater number of lactic organisms than other bacteria, as may be shown by comparing sour bark liquors from layer pits (Versetzgruben) which have often stood for a whole year exposed to great variations of temperature, with upper leather weak liquors which are only used in summer for a few weeks. The presence of phosphates is very favourable to lactic fermentation, but they are not present in such quantity as in milk, except in certain fruit tannins, Myrabolams, Dividivi, and Algarobilla, all of which easily undergo lactic fermentation.

Unless the acid produced be neutralised, its formation ceases when the amount of acid reaches 0·6—0·8 per cent. The lime in the hides neutralises a portion of the acid, and the hide fibres take up and hold a certain amount. The presence of the hide is necessary whether, like Timpe, we regard the albuminous bodies as merely neutralising the acid, or, like Hueppe, as food stuffs for the bacteria; at any rate, the hides materially assist the lactic fermentation. The amount of tannin also influences it, a high amount of tannin rendering the fermentation slower, but never stopping it. The yeasts are not affected by the tannin, and can grow even in thick extract of 25° B. The influence of foreign organisms on the lactic fermentation of tan liquors has yet to be studied exactly, but the author finds that when once the lactic acid is produced, it protects the liquors from infection by such organisms.

After acetic and lactic acids, carbonic acid is quantitatively the most important product of fermentation, being produced both by Saccharomycetes and many species of bacteria. The gas both de-limes and swells the hides. The lactic bacteria only produce a small amount of CO_2. The amount of the gas present depends on the temperature of the

liquors, the moving of the hides assists its escape, so that at any given time the amount dissolved in the liquors is usually small (see tables); in practice, therefore, this product need not be considered.

Propionic acid has also been found in small quantities in tan liquors (Wladika, Zur Kentniss d. Org. Säuren in Fichtenbrühen. Der Gerber **16**, 28). It occurs as a by-product of alcoholic fermentation, by the action of yeasts on the glycerin formed, and in some cases by direct fermentation of the calcium salts of lactic and malic acids by bacteria (Fitz and Duclaux).

Malic acid exists in large quantities in some fruit tannins and Wladika has found it in pine bark liquors. The formation of propionic acid in tan liquors is only of theoretical interest.

Butyric fermentation is in most cases of even less importance than propionic. The various bacteria causing it are chiefly anaerobic, and require a fairly high temperature for their proper development (40°). Their principal nutrient material is calcium lactate, so that except in such old liquors as have been used for de-liming, the conditions of growth, are not favourable. Besides which, the aëration of the liquors by the regular shifting of the hides further hinders their growth. In a large number of liquors examined, *Clostridium Butyricum* (Prazmowski) was only twice found.

In any case butyric acid has not the importance for the tanner Lietzmann assumes in his "Herstellung der Leder."

Viscous or ropy fermentation often occurs along with the lactic fermentation, and may also be caused by peptonising bacteria. The liquors become thick and in bad cases may be drawn up by the hand in long threads. Slight cases much hinder the proper diffusion of tannin in the liquors.

From ropy liquors examined, *B. lactis viscosus* (Adametz) and B. viscosus (Frankland) were twice isolated. In some cases the ropiness appeared to be produced by *B. mesentericus fuscus* (Hueppe), other liquors from which no slime organisms could be isolated produced ropy fermentation in tannin infusions containing peptones, but in pure fresh tan liquors under exactly similar conditions of temperature, &c., *no* ropy fermentation was produced. The author has shown that B. viscosus (Frankland) only produces ropy fermentation in materials containing peptones; this naturally applies to tan liquors.

Origin of the Ferments and the Infection of the Liquors.—The chief sources are the water used, the hides, the tanning materials, and the air. The water organisms are introduced directly in the preparation of the liquors and also indirectly with the hides; with the latter also are brought in the various bate organisms, the previous washing by no means freeing them from bacteria. At the same time the special dung and urine bacteria do not attain to any great development in the liquors. Those found have been already mentioned (*vide* this Journal, 1896, 910—911).

In order to determine what micro-organisms the tanning materials themselves contained, a bacteriological examination of fresh pine and oak barks and myrabolans (as fresh as possible) was made. The result was that lactic and acetic organisms were only sporadic, and it was doubtful whether the spores of those found did not come from the air of the tannery from which the material was taken.

In infusions of the above materials made with sterile water, no sensible acid fermentation took place during periods of 2, 4, 8, 20, and 30 days, but in each case a weak alcoholic fermentation was set up by the saccharomycetes which were always present.

The following organisms were found on barks:—*Micrococcus flavus liquefaciens, M. versicolor, Bacillus ochraceus*, the potato bacillus, various species of *Sarcina*, and some gelatin liquefying bacteria. The bacteria were cultivated from fresh bark and also from bark a year old, in the latter no moulds were found.

These results are of importance as showing that the sowing of the liquors cannot be caused by bacteria present in the barks, &c. used; on the other hand, the alcoholic fermentation set up by the saccharomycetes, because they are found in large numbers on the bark, leaf and fruit tannins.

The bacteriological examination of the air of tanneries was made by drawing a measured volume through tubes coated with sterile petone-, wort-, or milk-serum-gelatin; dishes of these materials were also allowed to stand in the tannery for different periods of time.

All the organisms previously arranged in groups were found (see this Journal, 1896, 911). Yeasts and moulds were most abundant, then followed putrefactive, acetic, and lactic bacteria. The number of germs and species was far larger in tanneries working on the old system owing to the heaps of bark and the dust. Where liquors only were used the air was much freer from micro-organisms.

The infection of the liquors takes place naturally from material left clinging to the sides of the pits, &c., for liquors made from hot extracts ferment in much the same manner as those prepared in the old way. By a proper choice of inoculating material very certain results may be obtained.—J. T. W.

Tanning, The Use of Mangrove Bark for. M. Gürke. Notizbl. d. botan. Gart. u. Mus. zu. Berlin, 1896, **1**, 169.

The author recommends that, notwithstanding the non-success of previous attempts to introduce the mangrove bark into Europe for tanning purposes, fresh trials should be made with this bark, which might now be procured from the German East African colonies.—I. S.

PATENTS.

Skins and Hides, A Novel and Quick Process in Preparing and making them into Buff or Oil Leather in less than 24 Hours. H. Riches and W. G. Riches, Godalming. Eng. Pat. 16,865, Sept. 10, 1895.

The skins, after previous treatment in the lime pits, are bated or drenched in a bath containing a 1 per cent. solution of ammonium sulphide or sodium hyposulphite (thiosulphate). This process takes one or more hours, according to the thickness of the skins. The skins may, after drying and pressing, be milled with cod oil, sod oil, or neats-foot oil, or white fats or greases, and afterwards washed in the usual way or with ammonium carbonate.
—W. P. S.

Manure, Process for rendering Waste Leather soluble and applicable as a. J. Hudson, London. Eng. Pat. 289, Jan. 4, 1896.

See under XV., *page* 55.

XV.—MANURES, Etc.

PATENTS.

Blood and Slag Manure, Process for the Production of. W. Barclay, Newcastle-on-Tyne. Eng. Pat. 22,160, Nov. 21, 1895.

Disintegrated blast furnace slag, either produced from ordinary iron ore, or hematite ore, or Normanby patent slag, is mixed with fresh, coagulated, dried, or dissolved dried blood, and the mixture is dried at between 14° C. and 37° C. or left to dry naturally, and can then be applied to the soil for manurial purposes.—D. A. L.

Superphosphate and the like, Impts. in Apparatus for Drying. J. Y. Johnson, London. From the Verein Chemischer Fabriken, Mannheim. Eng. Pat. 22,713, Nov. 27, 1895.

A number of slightly inclined sieves or gratings, made of barbed wire or perforated metal plates, in any case preferably provided on the upper surface with small vertical spikes, are arranged one above another in a shaft furnace, drying oven, or kiln, and are connected with a shaking mechanism. Steam coils or other means are used for heating and the material is charged in at the top; the lumps are retained by the spikes, until disintegrated and dry, the fine falling through the sieves.—D. A. L.

Manure, Process for rendering Waste Leather soluble and applicable as a. J. Hudson, London. Eng. Pat. 289, Jan. 4, 1896.

Waste leather is first thoroughly soaked with water containing barm or other ferment, then spread in successive layers over a floor until fermentation is set up. The latter may be started by employing a portion of leather already fermented. When fermentation is complete, the material may be used as a manure direct, or may be dried or pulverised before application.—D. A. L.

Ground Slags intended for Use as Fertilisers, Impts. in the Treatment of. C. H. and A. W. Langdale, Newcastle-on-Tyne. Eng. Pat. 22,666, Oct. 13, 1896.

From 2 to 20, preferably, 8 per cent. of a saturated solution of magnesium salts in brine, is mixed with the ground phosphatic basic slags intended for use as fertilisers.

—D. A. L.

XVI.—SUGAR, STARCH, GUM, Etc.

Sugar Juice, Observations on the Boiling of. J. Curin. Zeits. fur Zuckerind. in Böhmen, 1896, **25**, 176—183.

Having occasion to determine the boiling point of various sugar solutions, the author became aware of the magnitude of the error, especially with a good vacuum, when the temperature is measured merely by the usual vacuum gauge without regard to the atmospheric pressure. Temperatures given in Regnault's table corresponding to the vacuum in the apparatus, if taken without regard to the atmospheric pressure, may render the results of a test quite useless. From the table following, it appears that with a vacuum of 650 mm., and an atmospheric pressure of 720 mm., the error amounts to 9° C. (ÖZ. 1894, 985.)

Vacuum in Cm.	Error in C. with an Atmospheric Pressure of 750 mm.	740 mm.	730 mm.	720 mm.
70	3·4	7·5	12·6	19·0
65	1·9	4·0	6·4	9·0
60	1·4	2·9	4·4	6·2
55	1·1	2·2	3·4	4·6
50	0·9	1·8	2·8	3·8
45	0·8	1·6	2·4	3·3
40	0·7	1·4	2·15	2·9
35	0·6	1·2	1·8	2·6
30	0·56	1·1	1·7	2·3
25	0·51	1·0	1·52	2·1
20	0·47	0·95	1·4	2·0
15	0·44	0·9	1·34	1·8
10	0·42	0·84	1·26	1·7
5	0·40	0·8	1·2	1·6
0	0·35	0·7	1·1	1·5

After establishing this fact the author endeavoured to find the relation between the vacuum and temperature in the vacuum space (above the juice) during the boiling of sugar juice, employing for this purpose a cylindrical syrup pan with steam coils. To measure the vacuum he used his closed mercury vacuum gauge which is not exposed to the influence of the atmospheric pressure (Zeits. Zuckerind. Böhm. 1893—1894, 541). This and a tested thermometer were placed at the highest point of the pan immediately under the cover.

The results are collected in the table below:—

Space above the Juice.			Temperature of Juice, B° C.		Density of the Juice ° Balling.	Differences of Temperature.		
Vacuum Cm.	Corresponding Temperature. A°.	Actual Temperature. A_1°.	In the Upper Layer.	In the Lower Layer.		$\Delta_1 = B - A$.	$\Delta_2 = B - A_1$	$d_2 = A_1 - A$.
66·2	51·3	58·0	65·5	65·5	83·0	14·2	7·5	6·7
66·2	51·3	59·0	66·5	66·3	..	15·2	7·5	7·7
66·0	51·7	59·5	67·9	67·5	..	16·2	8·4	7·8
65·9	51·9	60·0	68·4	68·4	..	16·5	8·4	8·1
66·5	50·6	59·5	68·2	68·0	..	17·6	8·7	8·9
66·5	50·6	62·5	68·2	68·2	..	17·8	5·9	11·9
66·2	51·3	62·5	71·5	71·5	..	20·2	9·0	11·2
66·6	50·4	64·0	72·2	72·5	89·8	21·8	8·2	13·6
66·9	49·8	64·0	72·4	72·7	..	22·6	8·4	14·2
(2) 50·2	72·5	63·0	80·2	80·5	..	7·7	17·2	− 9·5
45·2	76·7	62·0	83·8	83·4	..	7·9	21·8	−14·7
40·1	80·1	62·0	86·8	86·8	..	6·4	24·8	−18·4

Several other similar tables are given in the paper. From the foregoing table we see that the first reading was made at the moment the syrup was at a concentration of 83° Balling, the vacuum being 66·2 cm.

The thermometer A_1 showed 58° C., although, according to the tables, this vacuum corresponds to a temperature of 51·3° C. The thermometer A_1 shows therefore the actual temperature, which is higher than the calculated; the difference $d_2 = A_1 - A$ amounts to 6·7° C.

This difference increases the thicker the syrup becomes, and reaches 13·6° C. at a density of 89·8° Balling. The figures given, relate to regular boiling without the drawing in of juice.

On the other hand, the figures under (2) were taken after the air-pump was shut off and stopped, and have no relation to the boiling, but were introduced merely as of interest. They show that during the warming of the syrup, while its temperature rises, the space above the juice becomes saturated by the escaping vapour, so that the vacuum falls and the temperature A_1 rises, or the difference d_2 quickly becomes negative. Another table showed that even during the introduction of fresh juice, d_2 remained positive; therefore, in all cases during the boiling of syrups A_1 is greater than A.

The author proceeds to show that this difference is not due to froth or waves of syrup coming into contact with the thermometer, which would cause irregular variations, but to small particles of syrup carried away with the vapour, and he is following up the relation between the difference d_2 and the loss of sugar during boiling.

In conclusion, columns 4 and 5 show that the temperature in all layers of the boiling juice is the same. Although Jelínek (Zeits. 1890—1891, 91) arrived at the same result, yet it is often stated, especially in foreign literature, that the lower layers are hotter in proportion to the pressure due to the column of syrup. Calculation shows that with a vacuum of 66 cm. and a height of 50 cm. between the two thermometers, a syrup of sp. gr. 1·45 should show a difference of 8·8° C. if this were the case. No differences greater than 0·5° C. were found, and he concludes that the column of syrup has no influence on the boiling point of the lower layers.—L. J. de W.

Massecuites, Critical Observations on the different Method of Working. H. Claassen. Bull. de l'Assoc. des Chimistes de Sucr. et de Dist. 1896, **14**, 447—452.

The author is of opinion that the value of a process for treating massecuites depends entirely upon the method of boiling. He has investigated the composition of the mother-liquors adhering to the crystals during the various stages of boiling, and considers that the coefficient of supersaturation should be regarded as the criterion of any boiling process.

The Bock, Kutte, Manoury, Huch, and other methods of treating massecuites are discussed; and it is insisted that, whatever the process, the following conditions are necessary:—1. A regular and slow boiling to be carried out subsequent to the last addition of syrup; the latter should not be saturated at the temperature of ebullition. 2. The massecuite to be concentrated until the syrup adhering to the crystals is not too supersaturated; the coefficient advised is 1·2 to 1·3. 3. The massecuite to be run into suitable mixers provided with heating and refrigerating arrangements. The chilling must be so conducted that the coefficient of supersaturation of the mother-liquors is about 1·1. 4 The boiling or mixing to be discontinued when the purity of the syrup has fallen to 70°; this value, however, should be higher for strongly coloured massecuites.

—J. L. B.

Raw Sugars: Decrease of Alkalinity. O. Mittelstaedt. Bull. de l'Assoc. des Chimistes de Sucr. et de Dist., 1896, **14**, 446—447.

New raw sugars which have been derived from juices treated with sulphurous acid contain alkaline sulphites, hyposulphites (thiosulphates), and probably also polythionates. The formation of these salts arises from incompletely burnt sulphur, or by a too rapid passage of gas, whereby particles of sulphur pass into the strongly alkaline juice. During storage the sulphites are gradually oxidised to sulphates: no diminution of alkalinity can, however, result. The hyposulphites (thiosulphates) and polythionates, on the other hand, are decomposed into sulphurous acid and sulphur, and then to sulphuric acid and sulphur; these products of oxidation destroying the alkalis.—J. L. B.

Sugar, Action of Sulphurous Anhydride on. W. Grundmann. Bull. de l'Assoc. des Chimistes de Sucr. et de Dist. 1896, **14**, 431—440.

The author has carried out an extensive series of experiments on the action, under varying conditions, of sulphurous acid on cane-sugar solutions. He finds in some cases that juices, which before sulphuration reduced Fehling's solution, failed to do so afterwards; this is probably due to the sulphurous acid modifying or destroying certain reducing substances present. It was also found possible to leave a saturated solution of sugar containing a slight excess of sulphurous acid for several weeks without any inversion taking place. On increasing the acidity, the solutions underwent inversion according to the amount of acid which they contained. The quantity of acid required for decoloration is at all temperatures lower than that necessary for inversion. Acidity increases more rapidly in cold than in hot juices. Up to a certain limit molasses are, however, an exception. These latter can moreover absorb a greater amount of sulphurous acid owing to the salts they contain. Acid juices invariably undergo inversion when boiled. The presence of ammonia appears to hinder inversion.—J. L. B.

Gum Ammoniac [Ammoniacum], Composition of. H. Luz. Arch. der Pharm. 1895, **33**, 540.

This gum has the following composition:—Resin soluble in ether, 69·0 per cent.; matters soluble in water (gum), 22·7; matters insoluble in water, 3·5; and water, 4·4 per cent., together with a small quantity of an essential oil, which has not been completely examined, but which is distinguished from the essential oils of sagapenum and asafœtida by not containing sulphur.

The resin varies in colour from a golden to a reddish-yellow, is soluble in alcohol, carbon bisulphide, chloroform, and glacial acetic acid; it is not completely soluble in alkalis; its ethereal solution and aqueous extract contain small quantities of salicylic acid, to which is due the violet coloration with ferric chloride, given by aqueous decoctions of the gum. By shaking the ethereal solution of the resin with potash, the resin is separated into an acid resin soluble in the alkali, and a neutral resin remaining in the ether; neither of the two contains sulphur. The acid resin saponified by potash yields salicylic acid and small quantities of valeric and butyric acids, and an alcohol, ammoresinotannol, $C_{18}H_{29}O_2(OH)$, isomeric with galbaresinotannol.

The gum, when purified, contains 1·2 per cent. of lime, and in this respect resembles gum arabic.—A. C. W.

Molasses, The Clarification of Solutions of, for Inversion. K. C. Neumann. Zeits. Zuckerind. Böhmen, 1896, **21**, 183.

See under XXIII., *page* 68.

Molasses, Contributions to the Analysis of. D. Loiseau. Bull. de l'Assoc. des Chimistes de Sucr. et de Dist. 1896, **14**, 349.

See under XXIII., *page* 68.

Massecuites, Determination of Density of. St. Tirpitz. Bull. de l'Assoc. des Chimistes de Sucr. et de Dist. 1896, **14**, 372.

See under XXIII., *page* 69.

Sugar, A New Method for the Direct Estimation of, in the Beetroot, by the Le Docte Apparatus. F. Sachs. Bull. de l'Assoc. des Chimistes de Sucr. et de Dist. 1896, **14**, 377.

See under XXIII., *page* 69.

Polarisation, Basic Nitrate of Lead as a Clarifying Agent for. F. Herles. Zeits. Zuckerind. Böhm. 1896, **21**, 189.

See under XXIII., *page* 67.

Raw Sugar Factories, Collated Methods of Analysis for the Control and Working of. Felcman, K. C. Neumann, and F. Herles. Bull. de l'Assoc. des Chimistes de Sucr. et de Dist. 1896, **14**, 392.

See under XXIII., *page* 69.

PATENTS.

Extracting Sugars [Use of Lead Oxide], Impts. in, from its Solutions. A. Wohl. Charlottenburg, Germany. Eng. Pat. 22,859, Nov. 29, 1895.

Yellow lead oxide (PbO) is used for extracting sugar from molasses or other salty after-products, with the addition of alkaline hydrates or carbonates to decrease the reaction between the lead oxide and the salt-like non-saccharine bodies present. The lead saccharate is washed with water at gradually increasing temperatures. The last traces of lead are removed from the sugar solution, after carbonating, by treatment with calcium hydroxide and carbonic acid, and filtering hot, with or without subsequent treatment with animal charcoal or very small quantities of magnesium powder.

The lead oxide is regenerated by heating the precipitate from the saturation stage, first at a low temperature with access of air, and then to 600° C., air being excluded. An alkaline earth in the form of a hydrate or carbonate may be added to the precipitate before burning, or it may be previously boiled with a dilute alkaline solution.

—L. J. de W.

Centrifugal Machines, Impts. in, for Drying Sugar and other Granular Substances. W. P. Abell, Essequibo. Eng. Pat. 22,900, Nov. 29, 1895.

The perforated basket is formed of two perforated cones placed base to base, with a permanent opening between their bases, and a casing, which encircles the basket, is adapted to revolve therewith, and has an independent movement in respect thereto, and a closing device or door secured to the casing and adapted to close the opening between the edges of the cones.

After the basket has received the regulated charge of massecuite, it is revolved for a length of time, previously decided upon as necessary to dry the quantity of massecuite dealt with. During this revolution the granular substances are retained in the basket by the perforated cones and door, while the liquids are driven, by centrifugal force, either directly into the fixed molasses casing or into the director or revolving molasses casing, which also delivers them into the fixed casing. When the massecuite is

sufficiently dry, a sleeve is lowered, and, with it, the molasses director and the door, whereby the dry sugar is discharged by centrifugal force through the permanent opening on to the inturned edge of the fixed dry sugar casing, and is deflected through the central outlet.

—L. J. de W.

By-Products of Sugar Works and Sugar Refineries, An Improved Process for Treating the. M. Sachs, Kiew, Russia. Eng. Pat. 1101, Jan. 16, 1896.

The process claimed is the boiling of by-products for crystals, along with repeated additions, in a vacuum-pan, or other like evaporating apparatus, and keeping the products to be supplied to the apparatus at a higher temperature than that of the apparatus.

The second product is boiled to grain instead of to string-proof, and half of the contents is dropped as soon as the grain is sufficiently large, and the quantity drawn off is replaced by repeated supplies of the same product, and the whole mass is kept boiling. The withdrawal and replenishing of the product is, preferably, continuous, and is maintained until a secondary crystallisation of a mealy or floury character appears, when the whole is drawn off. The vessels thus filled are kept at a temperature of 88° F. or higher for about six days to complete the crystallisation.

—L. J. de W.

XVII.—BREWING, WINES, SPIRITS, Etc.

Brewers' Yeasts, Contribution to the Study of Several. E. Boulanger. Ann. Inst. Pasteur, Oct. 1896. La Bière, **4**, [12], 179—182.

The yeasts selected for the investigations detailed were: Neunkirchen, Bass, Copenhagen 48, Hofbrau, Weihenstephan, Loewenbrau, Riga X, Frohberg, Saaz, and a high-fermentation yeast from Brussels.

The *Brussels yeast* was killed by a temperature of 55° C.; it yielded no spores: droplet cultures on gelatin liquefied the medium in $3\frac{1}{2}$ months.

The *Frohberg* (low-fermentation type) succumbed at 50° C.; liquefied gelatin in 2 months; produced no spores.

The *Neunkirchen* (low-fermentation) forms groups adhering to the sides of the fermenting vessel and clarifies quickly. It dies at about 50° C., and produces spores in about 240 hours on plaster and in 15 days in Liebig bouillon. The colonies from droplet cultures spread out considerably, but scarcely liquefy the gelatin in 6 months.

The *Bass yeast* gave no spores, died at 55°, and liquefied gelatin in 5 months.

The *Copenhagen* 48 (low-fermentation) was nonsporiferous, perished at 55° C., and liquefied gelatin in 3 months.

The *Hofbrau* (low-fermentation) succumbed at about 50° C., and gave no spores. The droplet colonies spread considerably, forming "craters," but took 4 months to liquefy gelatin.

The *Weihenstephan* (low-fermentation) yeast furnished no spores, and died at 55° C. Its colonies from droplet cultures were of a clear reddish-grey colour, and liquefied the gelatin in $4\frac{1}{2}$ months.

The *Saaz yeast* (high-fermentation type) was nonsporiferous, and perished at 50° C. After 4 months, a slight liquefaction of the gelatin was effected.

The *Loewenbrau* (low-fermentation) settled down in large granules. In the other particulars it resembled the preceding, but liquefaction had barely set in at the end of 6 months.

The *Riga X* (a low-fermentation Russian yeast) perished at 55° C., yielded no spores, and liquefied the gelatin after 4 months.

Two cases of degeneration were noticed, the Bass and Neunkirchen yeasts being directly descended from yeasts which in 1889 were sporiferous, and at that time able to withstand a temperature of 60° C. On the other hand, some of the Bass yeast spores, kept the same length of time, preserved their spore-producing power, although in respect of resistance to high temperature they had degenerated to the extent of 5° C.

In the attenuation tests the high-fermentation yeasts worked at 18° in the primary and 12° in the secondary fermentation, the process lasting 35 days, whilst the low-fermentation types worked at 8° for 20 days, the secondary fermentation lasting $2\frac{1}{2}$ months at 5°. The greatest attenuation was produced by the Frohberg (76·5), Neunkirchen (76·3), and Weihenstephan (76·4), and the lowest by the Bass (67·4) and Saaz (66·9) yeasts. The residual maltose was greater with the "high" than the "low" yeasts. Frohberg, Neunkirchen, and Hofbrau exhibited the greatest power of attacking dextrin, but the actual proportion eliminated was difficult to ascertain owing to the inefficiency of the Fehling test for the determination of maltose. The ratio of non-maltose to maltose was greatest (3·3) with Frohberg, and least (2·2) with Saaz yeast.

In ascertaining the amount of nitrogen consumed by each yeast, care was taken to prevent the precipitation of albuminoid matters by other agents during the fermentation. To this end the wort was acidified by 1 grm. of oxalic acid per litre, heated to nearly 100° C., and filtered, the free acid being then neutralised with lime. After a second filtration, and cold sterilisation, the wort was kept at 28° for 3 days and pitched. No precipitate was formed during fermentation, so that the weight of yeast and amount of nitrogen therein could be easily determined, the latter being estimated by the Kjeldahl-Aubin method. The results were as follows, the initial quantity of nitrogen in the wort being 0·640 grms. per litre:—

Yeast.	Weight of Yeast.	Nitrogen consumed.
	Grms. Per Litre.	Per Cent.
Brussels	2·69	33·7
Saaz	3·25	37·6
Bass	2·94	35·9
Frohberg	3·11	32·8
Neunkirchen	2·84	28·1
48	2·90	36·7
Hofbrau	2·54	32·9
Weihenstephan	2·69	29·8
Meurant	2·97	26·0
Loewenbrau	2·62	36·8
Riga X	2·90	23·7
Maltose	2·18	17·1

from which it appears that as a rule, the "high" yeasts have the greater power of elimination; these figures are, however, only relative, and would appear proportionally smaller with increased percentages of nitrogen in the wort. An attempt was made to ascertain the nature of the nitrogenous matter consumed, and, though the method employed (that of Stutzer) is imperfect, the author concludes from the results that the albuminoids coagulable by cuprous hydroxide are but little, if at all, attacked by yeast, although the results might have been different in the absence of amide nitrogen.

From an experiment made with Hofbrau yeast, it would appear that in the first few days of fermentation the yeast absorbs nearly all the nitrogen it requires, the increase after the third day being only 0·36 per cent. during 10 days. After fermentation is over, the yeast yields up nitrogen to the wort—a fact which may explain the contradictory results obtained by Hyde and Wahl and Hantke. The decrease in the amount of dextrin was regular up to the 13th day, by which time one-third had disappeared.

The effect of aeration on nitrogen consumption was also studied, by the aid of low-fermentation Hofbrau and high-fermentation Brussels yeast in deep tubes and shallow vessels, the proportion removed being found greatest under the last-named conditions. This agrees with Briant's results, but the amount consumed by the yeast could not, in the flat vessels, be differentiated from that precipitated as a result of the exposure. In the deep wort a greater amount of dextrin was eliminated than in the shallow vessels, particularly with the low-fermentation yeasts. On the other hand, the "high" yeast attacked maltose least in the deep tubes, whereas the converse occurred with the "low" yeast.—C. S.

Attenuation, Effect of Various Methods of Brewing on. P. Petit. La Bière, 4, [12], 187—189.

Experiments were made, with 100 kilos. of malt in each case, to ascertain whether any alteration in the attenuation could be brought about by modifying the method of brewing or fermentation, the same yeast being used throughout.

By the infusion method, with a steeping temperature of 51° C. for 30 minutes, heated to 74° for 20 minutes, agitated for 15 minutes, and left at rest for 30 minutes, the total maltose in the extract amounted to 68·9 per cent., and of this, one-third was formed during the first stage, 57 per cent. during the second, and 9 per cent. in the last two: this probably resulting from the solution of starchy matter in the grains, since complementary saccharification does not, according to the malto-dextrin theory, occur at this high temperature. A combination of the infusion and decoction methods gave a beer with an apparent attenuation of 72 per cent. (1 per cent. less than by the above method), but the yield was somewhat higher, being 61·5 (out of a theoretical yield of 70·5) per cent., against 60·2 per cent. by infusion alone.

With decoction methods it was found that whether two or three mashes were made, the resulting attenuation remained unaffected beyond a variation of 1 per cent., provided the same malt and yeast were employed in all cases. The maximum amount of sugar formed in the steeping stage was obtained at about 45° C., but the total amount at the end of the mashing process was approximately the same whatever the steeping temperature.

The only way in which the attenuation could be appreciably reduced was found to be by reserving 20 per cent. of the malt for addition in the final mash, which brought the total sugars down to 42·7 kilos. instead of 44 to 45 kilos., and produced a difference of from 5 to 7 per cent. in the attenuation. Similar results were obtained with 10 per cent. of rice, but the beer would not keep longer than 18 days. When rice is used in the final mash the saccharification should be tested by iodine to make sure of its completion, the conversion at this stage proceeding but slowly.—C. S.

Cold Hop Storage. L. Sterne. J. Fed. Inst. Brewing, 1896, 2, 537—541.

The author refers to the observations of Braint and Meacham (this Journal, 1896, 731) on the subject; and, whilst generally endorsing them, states that in the opinion of American brewers the temperature of storage should be 32° to 34° F. instead of 38° to 40° F. A description is also given of the De la Vergue system of refrigeration.—J. L. B.

Albuminoids, The Part played by, in Producing Lasting "Heads" in Beer. W. Windisch. Wochens. f. Brauerei, 1896, 13, 1253—1254.

The author infers from our present knowledge of the subject that two factors, *viz.*, carbonic acid and albuminoids, are together responsible for the production and retention of a "head" in beer. It is not only necessary that sufficient carbonic acid gas should be present, but also that a regular disengagement of gas should be going on in the liquid in order to enable it to retain this foam. The part played by albumose in this connection has been already indicated by the author (Wochens. f. Brauerei, 1893, 1358), albumose being a soluble, non-coagulable, peptonised albuminoid. Wahl's conclusion that peptones possess a "heading" influence is probably based on the fact that the commercial peptone used by him contained albumose, since this body is very commonly present therein. Römer has shown that albumoses are precipitated by zinc sulphate, whilst peptones are not; and by employing this method the author has separated from wort and beer, albuminoids possessed of marked foam-producing properties equal to those of albumose prepared by heating egg-albumin under pressure. During malting, a conversion of both starch and albuminoids occurs, the latter of which form albumoses, amides, and perhaps peptones; the more advanced the growth of the malt, the more complete being the conversion, so that an undergrown malt contains more albumose than a fully grown one. Kusserow has also studied the influence of malting upon the composition of wort, especially with regard to albuminoids, and the behaviour of the wort towards yeast and beer "condition." The process of kilning also influences albuminoid conversion, the lengthy process of kilning adopted in England being recommended by the author. The reason why many excellent malts yield beers deficient in condition, and not retaining a "head," is to be sought in the excessive albuminoid conversion which takes place in the mash-tun, for such malts are rich in enzymes and readily convertible. To correct this fault a curtailing of the mashing process and approximation towards the English mashing temperatures of 158° to 163° F. are advised, for even at this temperature an abundance of albuminoid nourishment for the yeast is produced.—T. A. G.

Alcoholic Fermentation, Retarding Influence of Various Reagents on. T. Bokorny. Allgem. Brauer u. Hopfen Zeit. 1896, 36, 1573.

A *résumé* of investigations made by the author and other observers. Dilute sulphuric acid or potash (1 : 5,000) arrests fermentation completely. Copper sulphate (1 : 20,000) does not act as a complete preventative, whilst mercuric chloride has a slightly more pronounced effect. Certain oxidising bodies ($KMnO_4$, Cl, I) are very destructive to yeast. Bromine, however, forms an exception, slight fermentation being observed in solutions containing 1 : 10,000. Potassium chlorate and iodate in 1 per cent. solution fail to arrest fermentation. Phosphorus (1 : 20,000) permits a slight fermentation to occur when the solution is incubated. A solution containing ortho-nitrocinnamic acid (0·1 per cent.) exhibits no fermentation when incubated for 24 hours, at 30° C.; under similar conditions the para-compound proved less effective. Ortho- and para-nitrotoluene (0·02 per cent.), ortho- and para-bromotoluene (0·1 per cent.), and ortho-toluidine sulphate (0·1 per cent.) arrest fermentation completely; whilst nitrobenzaldehyde (0·1 per cent.), potassium cyanide (0·02 per cent.), strychnine nitrate (0·02 per cent.), and quinine acetate (0·1 per cent.) fail to do so.—H. T. P.

Alcohol derived from Cellulose and Wood. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, 14, 456.

Systematic investigations upon the various conditions influencing saccharification, such as the nature and quantity of the acid, pressure, temperature, &c., have shown that if cellulose be treated with bisulphites, a maximum yield of 42 to 45 per cent. of sugar is obtained. 40 grms. of dry cellulose were heated for an hour and a half with 258 c.c. of 0·5 per cent. sulphuric acid, under a pressure of 6 to 8 atmospheres. When pine sawdust was used, the maximum yield of sugar (22·5 per cent.) resulted by heating for 15 minutes under the above-mentioned conditions. By longer heating the yield was reduced. Simultaneously with the production of sugar, an organic acid is formed which has not yet been investigated. After neutralisation the hydrolysed product was fermented.—J. L. B.

Wines: Amount of Acetic Acid in Italian, Greek, and Spanish Red Wines. H. Eckenroth. Chem. Rundschau, 1896, [6], 103—104.

Different wines vary in their tendency to the formation of acetic acid, this frequently depending upon climatic conditions, cultivation, nature of the soil, &c.; but, generally speaking, red wines show a greater tendency to acidity than white wines, and the red wines of Italy, Greece, and Spain possess this property in a much higher degree than German and French wines. It is generally assumed that 0·2 grm. of acetic acid or more, per 100 c.c. will render a wine sharp and unpalatable. According to Nessler, ordinary white wines should not contain more than 0·07 grm. and red wines more than 0·1 grm. volatile acid (calculated as acetic) per 100 c.c. In judging a wine in this respect, it is not sufficient, however, to determine the amount of volatile acid, as no definite limit can be fixed as applicable in all cases: the wine must also be tested. An acid wine poor in alcohol will often be unpalatable if it contain 0·1 grm. of volatile acid in 100 c.c. whilst a sweet wine rich in alcohol and sugar may contain 0·2 grm. or more without any acidity being noticeable. There can also be no doubt that the taste of wines is considerably influenced by the

character of the volatile acids, and that much smaller quantities of butyric acid, than of acetic acid, will render wines unpalatable. The author has determined the total acidity, volatile acidity, and alcohol percentages in 24 samples of Italian, Greek, and Spanish red wines, and in almost all of these he found an exceptionally high percentage of volatile acids. Only 3 samples contained less than 0·2 grm. of acetic acid in 100 c.c., whilst in the others the percentages varied between 0·207 and 0·270. The total acidity varied between 0·743 and 0·895 and the alcohol between 12·05 and 14·84 per cent. by volume. All these samples were faultless wines and some of them of high quality, thus showing that 0·2 per cent. of volatile acid may in some cases be even considerably exceeded without injury to the wine.

—A. K. M.

Wine, Effect of the too Frequent Sulphuring of Casks on. W. Fresenius, Chem. Zeit. Rep. **20**, [101], 302.

The author gives a warning against the repeated sulphuring of casks, and using them for the storing of wine without further treatment; for though such casks prevent the formation of mildew, they are nevertheless unfitted for the storing of wine on account of the formation of considerable quantities of sulphuric acid.—I. S.

Alcohol, The Estimation of. H. D. Richmond. J. Fed. Inst. of Brewing, 1896, **2**, 529.

See under XXIII., *page* 69.

Beer, Detection of Salicylic Acid in. J. J. La Bierè. **4**, [12], 182.

See under XXIII., *page* 67.

Invert Sugar [Wine Analysis], The Influence of Lead Salts on the Estimation of, by the Fehling-Soxhlet method. A. Bornträger, Bull. de l'Assoc. des Chimistes de Sucr. et de Dist. 1896, **14**, 453.

See under XXIII., *page* 69.

PATENTS.

Malt for Brewing, Impts. in the Treatment or Preparation of. E. W. Walker, Leeds. Eng. Pat. 22,059, Nov. 20, 1895.

In order to separate the perfectly malted grains of malt from the others, and stones, &c., the malt is mixed with water, when the first will float and may be skimmed off and dried.—A. L. S.

Beer, New and Improved Process for Adding Iron to [as Lactate]. J. Kwiatkowski, Sereth, Bukowina, Austria. Eng. Pat. 21,118, Sept. 23, 1896.

The inventor proposes to add lactate of iron to beer, in the proportion of 5—60 parts per 100,000.—A. L. S.

Meters [Distillery] for Measuring Spirits and Distillates, Impts. in. C. F. Chapp. Basse Terre, Guadaloupe. Eng. Pat. 23,849, Dec. 12, 1895.

See under I., *page* 33.

Specific Gravity, A New or Improved Apparatus for Separating Bodies of Different [such as Malt, Light and Heavy, in Water]. C. S. Meacham, Maidstone. Eng. Pat. 1177, Jan. 17, 1896.

See under XXIII., *page* 65.

Filters, suitable for Wines, Spirits, or other Liquors; Impts. in. R. Rendle, Pinner, Middlesex. Eng. Pat. 3911, Feb. 20, 1896.

The filtering device consists of a shallow lenticular-shaped chamber, one side of which is composed of sheet metal and the other of finely-meshed metallic gauze, which, when in use, is kept uppermost. Any number of these chambers are fixed axially on a tube, which communicates with their interior and serves to carry off the filtered liquid. The filtering material is composed of finely-divided asbestos, which is mixed with the first portion of the liquid to be filtered: the tube and filtering chambers being immersed in this liquid and suction applied to the tube, or pressure to the liquid, the finely-divided asbestos is deposited on the surface of the gauze, and the liquid to be filtered soon passes through bright.—A. L. S.

Seasoning Casks, Intended to Contain Wine, Spirits, and other Delicate Liquids, Ale, Stout, and the like; Impts. in. F. Ivison. Jerez de la Frontera, Spain. Eng. Pat. 11,162, June 26, 1896.

A small quantity of liquid ammonia is poured into the cask to be seasoned, which is then filled with steam at about 15 lb. pressure, and allowed to stand one hour and a half. The cask is then washed, when it will be found to be perfectly seasoned. For a 14 gallon cask, about one pint of ammonia of a sp. gr. 0·94 will be required, and a proportionate quantity for other casks of different surface area. American oak requires longer treatment and more ammonia. —A. L. S.

Apparatus for bringing to a desired Temperature, Articles of all Kinds, more particularly Wines: an Improved. C. Fischer, Hamburg. Eng. Pat. 20,869, Sept. 21, 1896.

See under I., *page* 34.

Filters for Wines, Spirits, and other Liquids; Impts. in. W. Elze, Köstritz, Thüringen, Germany. Eng. Pat. 21,610, Sept. 22, 1896.

See under I., *page* 34.

XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

(*A.*)—FOODS.

Wheaten Flour, the Suitability of, for Bread Making; A Chemical Method for Determining. E. Fleurent. Comptes. rend. 1896, **123**, [19], 755—758.

The comparative suitability of different flours for bread making, is essentially determined by the physical properties (in respect to elasticity, &c.) of the gluten respectively contained in them. The author shows that such differences in properties are clearly marked by corresponding variations in the relative proportions of glutenin and gliadin present in the crude gluten, and bases on this discovery the following method of examination. The gluten, obtained in the usual way (by kneading and washing in a current of water) from 33·33 grms. of flour, is divided into small pieces, and mixed in a wide-mouthed stoppered bottle with a quantity of glass beads, and 80 c.c. of alcoholic potash of known strength (3 grms. of KOH per litre of 70 per cent. alcohol). The mixture is well shaken at intervals until complete solution is effected (36—48 hours). A current of CO_2 is then passed to saturation through the liquid, and the latter transferred to a graduated flask, diluted to 110 c.c. with water and briskly mixed: 20 c.c. are then withdrawn before the precipitate has time to settle, evaporated in a tared dish, and dried to constant weight. The weight of residue, minus the quantity of K_2CO_3 contained in 20 c.c. of the liquid, represents *total gluten*. Another portion of the liquid is filtered to separate the insoluble glutenin, and 20 c.c. of the clear filtrate evaporated, &c. as above. The *gliadin* (with traces of conglutin) is thus found. The difference between this and the previous result represents *glutenin*. In case of flours containing 9 per cent. or more of gluten, it is advisable to use 150 c.c. of alcoholic potash for solution and to make up to 200 c.c.

A series of flours, previously examined as above, were submitted to careful practical tests, the results of which lead to the following conclusions:—

1. Leaving out of consideration the percentage of total gluten, the best results are obtained with flour, the gluten of which approaches most closely to the centesimal composition, glutenin, 25 per cent.; gliadin, 75 per cent.; (ratio, one-third).

2. Flour, in which the ratio of glutenin to gliadin is one-fourth, develops well during fermentation; but the dough again collapses and becomes compact during baking.

With such flour, the proportion of water normally employed must be reduced.

3. When the ratio of glutenin to gliadin rises to one-half, the flour becomes almost unworkable; the dough does not develop either during fermentation or baking, and the bread remains solid and indigestible.

4. A comparison between bread baked from flour, conforming to the standard laid down under (1), and that produced from flour varying 2 per cent. either way from the typical values, reveals differences that are readily perceived by an expert.—H. T. P.

Bread-Making, Mineral Oil used in. [*Brotöl.*] L. Hanemann. Chem. Zeit. 1896, **20**, 1023.

The sides of loaves which are to be packed in the ovens so closely that they are in contact, have to be smeared with grease before introducing into the oven. If this smearing is omitted, the loaves cake together into one mass on baking. Generally they are smeared with lard or butter, but a few years ago, "Brotöl" (bread oil) was introduced by a Hamburg firm as a cheap substitute. It was effective for the baker's purpose, but being a mineral oil it was in other respects quite unsuitable. One baker, when preparing a meal, used it in the place of butter, and illness of the whole family resulted. The Supreme Imperial Court has recently made the smearing of bread with this oil a penal offence in case of resulting injury to health. A warning to bakers not to use the oil has been issued in Bremen.
—J. A. B.

Army Bread, Note upon a New. Balland. Comptes. rend. **123**, 1007—1009.

The new army bread which is being substituted for the old army biscuit is made from a fine flour "bolted" (sifted) at 30/100, water, salt, and fresh yeast. The biscuit contained only flour, "bolted" at 20/100, and water. The author gives details of the size of the new bread rolls, and of their characteristics as regards crumb and crust. He has analysed three samples of the new bread, one of the old army biscuit, and also four other samples of breads made in France. The results of these eight analyses are given; and an examination of the figures, in conjunction with the results of some earlier study of his own on flours, demonstrates, he considers, the following points:—

1. The constituents of a pure wheat-flour bread, in its dry state, agree with those of the flour from which it is made. The nitrogen, cellulose, and phosphates are in the same proportion. The ash is increased owing to the salt. The fat constituents, more or less modified by the treatment undergone in baking or in the analysis, are reduced.

2. The nitrogenous matter and the phosphates which are least in the well-bolted (sifted) flours, are naturally in less proportion in bread made with flour bolted at 40/100 than in bread made with flour "bolted" at 20/100. The latter bread is consequently the more nutritious.

3. The new army bread, made with a better bolted flour than the old army biscuit, contains less nitrogenous matter. On this account the campaign rations of fresh meat have been raised from 300 to 500 grms., and the soldier does not lose by the change in the flour.

4. The change to cylinder rolls in milling has caused a complete disappearance of brown bread in the towns; and this displacement of brown bread by white is spreading rapidly over the country districts. Whereas 30 years ago, 83 kilos. of bread-flour were generally obtained per 100 kilos. of wheat; to-day only 71—72 kilos. are recovered from the same amount on farms in the country districts of Bresse; and this return is probably correct for other country districts. This loss of nitrogenous matter and phosphates has not been balanced by any increase in meat or other nitrogenous food-stuff for the greater number of consumers. Many doctors, including Prof. Tarnier, believe the lessening vitality of the race in the richest towns and departments of France, is due to such a cause, and it would seem desirable to prevent this excessive bolting (sifting) of flour intended for nourishment.—J. B. C. K.

Critical Temperatures of Solution [*Butter Analysis*], *in Open Tubes.* L. Crismer. Bull. Assoc. Belge des Chimistes **10**, [8], 312.

See under XXIII. *page* 70.

Theobromine in Preparations of Cocoa, Determination of. A. Eminger. Apotheker Zeit. 1896, 716.

See under XXIII., *page* 71.

PATENTS.

The Preservation [*with Iodine and Iodides*] *of Meat, Fish, and other Animal Substances, and the Manufacture of Food Stuffs therefrom, An Improved Process for.* L. P. J. Revel and J. M. Campagne, Paris, France. Eng. Pat. 22,989, Nov. 30, 1895.

The meat (raw or cooked), or fish, eggs, &c. is steeped for 15 minutes in a bath composed of water, 1,000 grms.; potassium iodide, 15 grms.; iodine, 5 grms. (or tincture of iodine, 20 grms.), and allowed to dry. Meat, fish, &c. thus treated, will retain their original freshness for 4—5 days, and then commence to slowly dry (without becoming in the least putrid), and in this dried condition may be preserved almost indefinitely. Instead of steeping in the bath, animal substances may be brushed over with or wrapped in cloth saturated with the preservative solution. Another claim relates to the production of "iodized meat bread," made by incorporating about 3 per cent. of ground, dried, iodized meat with the dough.—H. T. P.

Effervescent Beverages, Impts. in the Manufacture of, in the Preparation of the Materials Used in the said Manufacture, and in Obtaining Useful Products therefrom. [*Bisulphate and Bicarbonate of Soda.*] J. Hargreaves, Farnworth-in-Widnes. Eng. Pat. 925, Jan. 14, 1896.

Bisulphate of soda or other acid sulphate of soda is used in combination with bicarbonate of soda for the production of carbonic anhydride in gasogenes or similar apparatus. The bisulphate is prepared by fusing and casting it into spheres, rods, or plates with a known and defined content of free acid. It is packed into parcels or portions together with such a proportion of bicarbonate of soda as will neutralise its content of free acid. After the evolution of the carbonic anhydride, the mother liquor is crystallised to form Glaubers salts or concentrated to form anhydrous or other desired sulphate. To obtain the largest production of sulphate of soda, the mother liquor from one charge is added to the above-mentioned mixture, which is used for succeeding charges.—D. B.

Cocoa or Coprah Oil, Manufacture of an Edible Fat from. M. C. A. Ruffin, Paris. Eng. Pat. 1827, Jan. 25, 1896.

See under XII., *page* 50.

Sterilising Apparatus [*for Milk, &c.*], *Impts. in.* S. S. Bromhead, London. From F. Dierks and J. Möllmann, Osnabrück, Germany. Eng. Pat. 3572, Feb. 17, 1896.

An apparatus so arranged that the bottles, &c., containing the material undergoing sterilisation, may be automatically closed prior to their removal from the heating chamber. To this end the latter is traversed by one or more excentric shafts from which the bottles are suspended by their stoppers (some form of lever stopper), a rigid connection being secured by means of square pins projecting from the shaft, and corresponding "eyes" fitted to the stoppers. A half-turn imparted to the shaft from the outside of the chamber then suffices to close (or open) the bottles when required.—H. T. P.

Sterilising or Pasteurising Milk and Cream, An improved Process of. L. G. Fagersten and C. F. P. Kerssell, Chicago, U.S.A. Eng. Pat. 18,275, Aug. 18, 1896.

The improvement consists in adding to the milk, prior to sterilisation, sufficient wax or solid paraffin, &c. to form, when melted, a continuous layer on the surface of the

liquid. The solid crust of wax left on cooling, will, it is claimed, prevent that separation of milk-fat, to which sterilised milk is ordinarily somewhat liable.—H. T. P.

Apparatus for Heating [Pasteurising] and Cooling Liquids, Impts. in. W. F. E. Casse, Copenhagen. Eng. Pat. 22,060, Oct. 5, 1896.

See under I., *page* 34.

(B.)—SANITATION; WATER PURIFICATION.

Sewage, The Ultimate Purification of. G. Thudichum. A paper read before the Society of Engineers, Dec. 7, 1896.

The author's object was to describe, for the benefit of engineers and others, the biological purification of sewage by filtration, and to contrast this method with purification by land irrigation.

Mr. Dibdin had shown that any process adopted for the purification of sewage must be subordinated to the requirements of the micro-organisms. Sterilisation was a mistake, since as soon as antiseptic action was neutralised by dilution, putrefaction was set up. Direct oxidation by air could not be induced in the absence of microbes. An attempt to effect oxidation without putrefaction by adding organisms to the sewage, failed, and the general experience of the author goes to prove that all sewage effluents produced by chemical precipitation are finally purified only by a putrefactive process. The discovery of these facts naturally led to the attempt to effect purification by adding the sewage to the organisms previously established in a filter bed, under the necessary conditions. The Massachusetts experiments showed that, in this way, 97 per cent. of the organic matter contained in raw sewage could be removed, but not more than 60,000 gallons of sewage per acre per diem could be re-treated. Mr. Dibdin had gone further, and had succeeded, by experiments conducted upon London sewage, in purifying 1,000,000 galls. of sewage effluent per acre per diem, the oxidisable organic matter being reduced on an average by 80 per cent. (See this Journal, **14**, 1895, 915—922). The depth of Mr. Dibdin's filter must not be taken as a necessary factor in its efficiency; cubic contents and not surface area should be considered, as the work is all done in the body of the filter. The depth is probably limited only by the necessity for free air supply.

The conditions unfavourable to the success of irrigation farms were then pointed out, the principal objection being the necessity for applying sewage to the land irrespective of the condition of the latter or of the requirements of the crops. Also nuisance, more or less pronounced, is almost always caused. It is suggested that where sewage farms already exist, the filter system should be adopted, and the filtrate, which contains nitrates and other fertilising ingredients, should be applied to the land when likely to prove beneficial, and should at other times be allowed to escape directly into a watercourse.

The attitude of the Local Government Board in insisting upon irrigation, was criticised and condemned, and mention was made of various places — Wolverhampton, Sutton, Aylesbury, Exeter—where the method of filtration adopted in the Barking experiments has been successfully followed.
—L. A.

Flood-Water of the Seine, the Yonne, and the Marne: Nitric Acid in. T. Schloesing. Comptes rend. **123**, 919—923.

The author refers to a test made by Boussingault in March 1876, when the flood-water of the Seine at Paris contained 1·2 mgrm. nitric acid per litre, and he compares this with his own tests, made in November 1896, of the flood-waters of the Seine, the Yonne, and the Marne. These varied from 3·13 up to 5·08 mgrms. of nitric acid per litre.—J. B. C. K.

Phosphorus, The Toxic Action of Aqueous Solutions, and the Solubility in Water of. Th. Bokorny. Chem. Zeit. 1896, **20**, 1022.

The author prepared aqueous solutions of phosphorus by adding alcohol to solution of phosphorus in carbon bisulphide, and pouring the mixture into a large volume of boiling water.

As a result of the experiments, phosphorus cannot be regarded as a very powerful poison towards the lower organisms. Water in which phosphorus had for some time been kept was much more rapidly fatal to them than the aqueous solution prepared as above. The powerful toxic properties of this water appear to be due to the ozone, hydrogen peroxide, and phosphorous acid which the phosphorus produces rather than to the phosphorus itself. To the higher animals and man, phosphorus is a virulent poison; but the author is not aware that the action of aqueous solutions on vertebrates has yet been studied.
—J. A. B.

Nitroglycerin, Researches on the Toxic Power of. Th. Bokorny. Chem. Zeit. 1896, **20**, 1021.

Aqueous solutions of 0·1 per cent. of mineral salts (calcium nitrate, magnesium sulphate, and mono-potassium phosphate) were prepared, containing the requisite quantity of 30 per cent. solution of nitroglycerin in absolute alcohol to give them percentages of 0·2, 0·1, 0·05, and 0·01 of nitroglycerin. The vitality of lower organisms in each of these four solutions was observed, and it was found to be impaired only in that containing 0·2 per cent. of nitroglycerin. In 24 hours the diatoms, infusoria, and amœbæ therein ceased motion; but even after three days many filaments of spirogyra remained distended, though the nuclei and chlorophyll bands were displaced. On adding 10 per cent. of cane sugar, some ammonium sulphate and a trace of brewers' yeast to the solutions, the yeast propagated itself freely. Some of the organisms at least had assimilated nitroglycerin, as it could not afterwards be detected in the solutions used. Therefore, it appears to be only poisonous to a small extent to the lower animals and plants, though powerfully toxic in the case of the higher animals and man. Its action on animals is considered by Cagnoli to be due to formation of nitrous acid, and, since the latter is poisonous to plants also, it is argued that the lower organisms can assimilate nitroglycerin without formation of nitrous acid.

Nitro-ethane is also a powerful poison towards the higher animals. A series of experiments similar to those made on nitroglycerin showed that it is quite harmless in the case of lower organisms.—J. A. B.

Lead in Water, A Simple Method of Detecting and Estimating Minute Quantities of. J. C. Berntrop. Chem. Zeit. 1896, **20**, 1020.

See under XXIII., *page* 66.

PATENTS.

Sewage, Treatment of, at Precipitating and at Outfall Works [Liming, followed by Manganate of Soda Treatment]; Improved Means and Appliances for. R. H. Reeves, London. Eng. Pat. 22,502, Nov. 25, 1895.

Lime-water is prepared by the action of high-pressure jets of water on unslaked lime (and other chemical solutions similarly). This is caused to commingle with the sewage whilst in a state of agitation. After settling, the supernatant liquid is run off, and treated with, preferably, manganate of soda and sulphuric acid; it is then filtered through artificial filter-beds. The sludge is not filter-pressed, but is dried in a thick state, as peat is; the manurial value is thus said to be greater.—L. A.

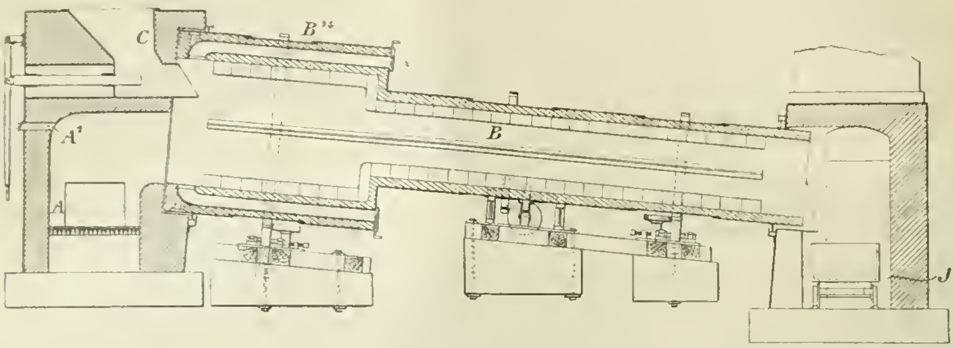
Sewage and other Liquids, Filtering [Combined Filtration and Aeration]; Impts. in or connected with Apparatus for. T. B. Jones, Harrogate. Eng. Pat. 24,943, Dec. 30, 1895.

The filtering material is contained in a series of trays supported one above another, with intervening air-spaces, by a vertical open framework. The bottoms of the trays are perforated, and the filtering material in each is covered by a loose perforated plate. The liquid falls in a thin stream on to the perforated plate of the topmost tray, and descends through the entire series. The trays are made to slide in and out on rollers.—L. A.

Destructor Furnaces for the Treatment of Town Refuse, Impts. in or connected with. S. Willoughby, Chiswick, Middlesex. Eng. Pat. 2901, Feb. 8, 1896.

The general principles of the destructor, which formed the subject of a previous specification (Eng. Pat. 20,719, 1894), are shown in the illustration.

Fig. 1.



Destructor Furnace.

The refuse matter is fed into the hopper C, and falls into the inclined rotating flue or combustion chamber B, in travelling along which it is consumed by the flame and hot air from the furnace A, and the ashes fall into the truck J. The improvements now described consist chiefly of arrangements for the admission of an auxiliary supply of air to burn the refuse more completely. The anterior end of the combustion chamber is enlarged and provided with passages, B¹⁴, through which air may be drawn by the chimney draught, or forced under pressure; or passages, A', may be provided, through the crown or walls of the furnace, so as heat the air before it meets the refuse.

—L. A.

XIX.—PAPER, PASTEBOARD, Etc.

Indian Hemp [Refuse Plant Fibre]. H. C. Dyer. Indian Pharmacologist, 1896, **1**, 85.

There is no reason why the refuse from the plants cultivated for the sake of the drug should not be utilised. The refuse plants are well fitted for the manufacture of paper.

The term "hemp" is employed for the fibres from several distinct plants. Thus, New Zealand hemp is obtained from *Phormium tenax*, sisal hemp from a variety of aloes, sun hemp from *Crotalaria*, Manilla hemp from *Musa textilis*, &c. The best—hemp proper—from *Cannabis sativa*, is produced in Italy.

In cultivating hemp for its fibre, a rich soil is essential. The plant thrives particularly well in moist alluvial situations, in a fine soil rich in vegetable matter. The carefully selected seed is sown rather thickly—in India generally broadcast, in Europe in drills. The ground is usually tilled earlier, and well pulverised and levelled. The plant is diœceous. The male plants flower first, and are cropped off close to the ground; the female plants are left till they produce seed, and are then cropped. The stems are handled one by one, and the leaves cut off. They are then tied in bundles and steeped in water. The retting process is also effected by exposure to dew, chemicals, or snow. When the soft tissues have more or less disappeared, the bundles are unloosed and the stems spread out to dry, and when dry the fibre is stripped by hand. The coarse fibre so obtained is finally cleaned by scutching.

To preserve the quality of the fibre, change of seed must be made every season. India exports hemp fibre to the value of 45,000 rupees, while the imports amount to over 10 lakhs of rupees' worth annually.—A. C. W.

PATENT.

Paper which is Proof against the Action of Water, Air, Grease, and Weak Acids; Impts. in the Manufacture of. A. Zimmermann, London. From The Chemische Fabrik auf Actien vormals E. Schering, Berlin. Eng. Pat. 22,625, Nov. 26, 1895.

Parchment paper is drawn through a 2 to 3 per cent. solution of pyroxylin in ether-alcohol or other solvent. The film left by evaporation is firmly united to such paper, whereas from ordinary paper, water detaches it easily. If the parchment paper be first treated with a 3 to 5 per cent. solution of cuprammonium, a successful result is certain, even if the paper be very stout and hard.—L. A.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

Angelica Oil. G. Ciamician and P. Silber. Ber. **29**, 1811.

In the lower fractions of the oil of *Angelica archangelica* previous investigators observed one of the valeric acids and oxymyristic acid, also a terpene boiling at 172° C., but the contents of the higher fractions were never investigated. This oil was found by the authors to consist chiefly of a high boiling terpene and an oxy-acid $C_{15}H_{30}O_3$. The latter, however, is not present in the free state, but is formed on saponifying the oil with alcoholic potash. Amongst the products of this saponification a valeric acid, which was subsequently identified with methyl-ethyl acetic acid, was also observed.—C. O. W.

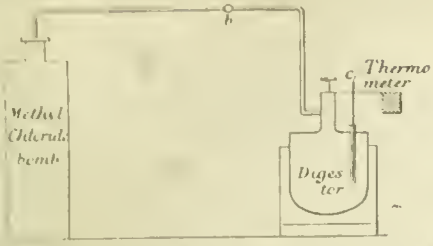
o- and p-Nitrophenol and their Technical Uses. [Preparing Phenacetin.] L. Paul. Zeits. f. ang. Chem. 1896, 587—595.

Phenol, on nitration, gives two mono-nitro derivatives, which, as is known, are separated by distilling with steam. The *o*-nitro compound passes over with the steam, and is thus obtained in a condition sufficiently pure for conversion into *o*-nitranisol without further preparation. The *p*-nitro compound which remains behind in the retort along with tarry matters is isolated by repeated crystallisation from xylene. It serves as the intermediate material for the manufacture of *p*-nitrophenetol, phenacetin, and *p*-amidophenol.

As the latter compounds are of less industrial importance than the compounds derived from *o*-nitrophenol, a method for preparing the latter from phenol without forming the *p*-isomeride, is desired. A method devised with this object by The Farbenfabriken vormals F. Bayer and Co. (Ger. Pat. 43,515), consists in converting phenol into its *p*-sulphonic acid, nitrating this to obtain the o-nitro derivative, and then hydrolysing to remove the sulpho group. It, however, gives an unsatisfactory yield.

o-Nitranisol.—A hot solution of 50 grms. of *o*-nitrophenol and 15 grms. of caustic soda in 100 c.c. of water, is placed with 300 c.c. of methylic alcohol and 10 grms. of

sodium carbonate in a digester, attached to a methyl chloride cylinder.



The taps (*b*) and (*c*) are kept closed, while the tap connected with the cylinder is opened for a few minutes, then this tap having been closed taps (*b*) and (*c*) are successively opened. After a while, (*b*) is opened and finally the tap connected with the cylinder. The pressure inside the digestor rises to 3—4 atmos. and the reaction is allowed to continue for half-an-hour. The taps are thereupon closed, and the digester is heated gradually to 100°, the pressure increasing to 6—7 atmos. After heating for about 15 hours, the contents of the digestor are freed from alcohol by distillation and the lower oily layer of *o*-nitranisol is separated from the aqueous solution. To purify the product, it is extracted with ether, or is distilled. The yield (45 grms.) is about 83 per cent. of the theoretical.

Dianisidine.—100 grms. of *o*-nitranisol, 200 grms. of methylic alcohol and 60—70 grms. of caustic soda solution at 35°—40° B. are heated together to 70° in a cast-iron digester, furnished with an agitator. 150 grms. of zinc dust in portions of 5—10 grms. are added, the temperature being allowed to fall to 50°—55°, and being maintained thereat until the reaction is completed. 200 c.c. of hot water are next added and the mixture is distilled until the alcohol and about 25 c.c. of a milky liquid (containing the anisidine formed) have passed over. The residue is placed in a large dish and washed first with 425—450 c.c. of concentrated hydrochloric acid diluted with 2 litres of water and cooled to below 10°, and then several times with water. It is next treated with hydrochloric acid (about 100 c.c. of concentrated acid, and 1000 c.c. of water) below 10°, any lumps of hydrazo-anisol present being crushed to bring them into action. The mixture is then filtered and the dianisidine precipitated as sulphate by the addition of sodium sulphate. The yields obtained in two experiments amounted to 41 and 44 grms. respectively.

p-Phenolsulphonic Acid.—25 kilos. of phenol are mixed with 28 kilos. of concentrated sulphuric acid, and heated at 90°—100° for 24 hours. The solution is poured into 150 kilos. of a solution of common salt, the precipitated sodium phenol-*p*-sulphonate being collected on a woollen filter, pressed, and dried; it amounts to 76 kilos. It is purified by re-crystallising from 30 litres of water.

o-Nitrophenol-p-Sulphonic Acid.—31 kilos. of sodium phenol-*p*-sulphonate, purified as described, are dissolved together with 31 kilos. of sodium nitrate in 150 litres of water at 40°. 31 kilos. of concentrated sulphuric acid are added in a fine stream, its addition producing a rise of temperature. When this reaches 65°—70°, the reaction commences. After it is over and the solution has cooled, the aqueous solution is withdrawn from the mass of crystals which deposits, the latter being pressed, dissolved in boiling water, and again crystallised, or, to save time, the acid is converted into its readily crystallisable sodium salt, which is accomplished by adding common salt to the solution.

o-Nitromethoxybenzene-p-sulphonic Acid.—A mixture of 100 grms. of sodium *o*-nitrophenol-*p*-sulphonate, 25 grms. of sodium carbonate, 300 c.c. of water, and 66 grms. of methyl iodide, is heated in an autoclave at 100°—110° until the pressure, which at first is considerable, has decreased. The sodium salt of the methoxy-compound which is produced is purified by crystallisation.

Anisidine- and Phenetidine-sulphonic Acids.—100 grms. of *o*-nitrophenetol-*p*-sulphonic acid are added to a solution of 400 grms. of stannous chloride in 400 grms. of hydrochloric acid at 20° B., at a temperature of about 60°. The mixture is diluted with water and the tin precipitated by means of sodium sulphide, after which the solution is filtered and evaporated. Common salt first separates, and then sodium phenetidine-sulphonate.

Methoxy- and Ethoxyazobenzene-disulphonic Acids.—250 grms. of sodium *o*-nitrophenetol-*p*-sulphonate, 750 c.c. of water, and 500 c.c. of caustic soda solution at 40° B., are heated together to 70°, and, while the temperature is maintained at 70°—80°, 100—150 grms. of zinc dust are gradually added. The mass is mixed with 2 litres of common salt solution, the residue filter-pressed, dissolved in 1½ litre of hot water, filtered, and the sodium salt precipitated by the addition of 250—260 grms. of common salt. 192 grms. of it, or 90 per cent., of the quantity theoretically producible, are obtained.

Dianisidine- and Diphenetidine-disulphonic Acids.—For the preparation of diphenetidine-disulphonic acid, 47 grms. of sodium ethoxyazobenzene-disulphonate are dissolved in 300—400 c.c. of water, and are treated, at a temperature not exceeding 10°, with a solution of 23·5 grms. of stannous chloride in 45 grms. of hydrochloric acid at 12° B., 50 c.c. of a 16-per cent. solution of the same salt being added when the solution has become colourless. The tin is next precipitated with sodium sulphide, and then the diphenetidine-disulphonic acid with sodium sulphate. The latter is washed with sodium sulphate solution and pressed, 113 grms. of a paste containing 43 grms. of diphenetidine-disulphonic acid being obtained.

o-Amidophenol.—3 kilos. of o-nitrophenol are ground with 750 c.c. of water and 2 litres of concentrated ammonia. The pasty mass of the ammonium salt thus obtained is placed in a suitable vessel with 6 litres of water, and heated to 20°. The reduction is effected with hydrogen sulphide, which is passed into the solution while it is slowly heated to 40°. When the conversion of the nitro-compound is complete, the current of gas is stopped. The solution is allowed to stand for two days and is then filtered. The residue, after being made into a paste with water, and filter-pressed, is dissolved in hot water and crystallised. 1·5 kilos. of the amido-compound are obtained, for the production of which 15 kilos. of ferrous sulphide and 65 kilos. of hydrochloric acid at 12° B. are consumed in generating the hydrogen sulphide required.

p-Amidophenol.—25 kilos. of *p*-nitrophenol are added to 180 kilos. of hydrochloric acid at 20° B. and 75 kilos. of tin, contained in an earthenware vessel, the temperature rising to 100°—106°. 200 kilos. of concentrated sulphuric acid are next added in a fine stream. After standing for three days, the *p*-amidophenol sulphate which has separated, is filtered off, drained, dissolved in 200 litres of cold water, and decomposed with soda, 1 kilo. of sodium bisulphite being added to the solution to protect the precipitated base from oxidation. The precipitate is filter-pressed and dissolved in 200—300 litres of boiling water, along with 10 kilos. of sodium bisulphite, the filtered solution on cooling, depositing the base in colourless needles.

p-Nitrophenetol. — For the preparation of this, the *p*-nitrophenol used is first purified by a method such as the following:—800 grms. of crude *p*-nitrophenol are dissolved in 8—10 litres of hot water, 250 grms. of chalk added, the solution filtered, treated with 200 grms. of soda and 5 kilos. of common salt, again filtered, and set aside to crystallise. 950 grms. of the purified sodium compound were thus obtained in one experiment.

The ethylation is effected by heating together under a reflux condenser for 10 hours, a mixture of 480 grms. of the salt purified as above described, 3,120 grms. of methylic alcohol, 300 grms. of ethyl bromide, and 100 grms. of sodium carbonate. After cooling, the separated *p*-nitrophenetol is filtered off, washed with hot water until the wash-water has only a pale brown colour, and crystallised from alcohol. 340 grms. of it are obtained.

p-Amidophenetol. — 212 grms. of *p*-nitrophenetol are gradually added to a solution of 848 grms. of stannous chloride in 1,060 grms. of hydrochloric acid at 50°—60°. The amido-compound formed is precipitated from the solution as hydrochloride by the addition of concentrated hydrochloric acid. It is filtered off, drained, and freed from tin by dissolving it in hot water, acidulated with hydrochloric acid, and placing a few strips of sheet zinc in the

solution. Long, colourless prisms of the hydrochloride are obtained, in amount corresponding with 60 per cent. of the theoretical yield.

Phenacetin. — 16·7 grms. of *p*-amidophenetol hydrochloride, 8·7 grms. of anhydrous sodium acetate, and 83 grms. of acetic acid, are heated under a reflux condenser for 3 hours. The mixture is poured into 10 times its volume of boiling water and the solution filtered. On cooling, the phenacetin produced, deposits in pale red crystals. It is purified by repeated filtration of its solution through animal charcoal and crystallisation, being ultimately obtained in minute, lustrous crystals. The yield obtainable is 90 per cent. of the theoretical.—E. B.

Apiol, On a New. G. Ciamician and P. Silber. Ber. **29**, 1799.

The higher boiling fraction of a dill oil, obtained from Indian seed, was found to consist chiefly of a new apiol, boiling at 285° C. This new apiol contains two methoxyl groups, but differs from ordinary apiol in that it is an almost odourless, oily, and non-crystallisable liquid, insoluble in water, but readily soluble in all other ordinary solvents. Its behaviour towards caustic soda and sulphuric acid is the same as that of ordinary apiol. On treatment with alcoholic potash or sodium ethylate, the new apiol forms a beautifully crystallised isapiol, melting at 44° C. This isapiol from dill oil is identical with ordinary apiol. Like ordinary apiol the new isapiol on oxidation, yielded the same tetramethylapionol—

$$C_6H_2\begin{cases}(OCH_3)_2\\(OCH_3)_2\end{cases}$$

The new apiol produces a dimethyl apionol, which is isomeric with that obtained from ordinary apiol, its diacetyl derivative melting at 85° C., whilst that of the latter melts at 144° C. The dimethylapionol obtained from the new apiol, on methylation yields however, a tetramethylapionol identical with that obtained by the same treatment of the dimethylapionol derived from ordinary apiol. From this it follows that ordinary apiol and the new one, are derivatives of the same tetravalent phenol.—C. O. W.

Rose Water, An Acid contained in. E. Charabot and G. Chiris. Comptes rend. 1896, **123**, [19], 752—753.

The authors have observed that rose water always contains a trace of an organic acid, and consider it to be produced during distillation (*i.e.*, of roses with water) by the partial saponification of the levo-gyratory ether, which Dupont and Guerlain (Comptes rend. **123**, 700) have shown to be present in oil of roses. The softer odour and higher rotatory power of French, as compared with Turkish rose oil, are ascribed to the more perfect method of distillation employed for the preparation of the former essence, whereby the destruction of the ethereal substance is reduced to a minimum.—H. T. P.

Quinine, Sulphate, The Efflorescent Nature of Crystallised. A. J. Cownley. Pharm. J. 1896, **57**, 525.

The author, some years back, published a paper (Pharm. J. [3], **7**, 189) drawing attention to the peculiar efflorescent character of crystalline quinine sulphate, and he now again sets forth the facts then established.

1. Crystallised quinine sulphate, containing 7½ mols. of water of crystallisation, when freely exposed to air at the ordinary temperature, rapidly effloresces until it attains the composition of a sulphate containing 2 mols. of water.

2. Freshly-crystallised quinine sulphate approximates to 7½ mols. of water of crystallisation.

3. Crystalline quinine sulphate is rendered anhydrous at 100° C.

4. The anhydrous salt, when freely exposed to air at the ordinary temperature, rapidly absorbs water until it has the composition of a sulphate with 2 mols. of water, but when access of air is retarded, the water of crystallisation in the salt is of a varying quantity, and bears no constant relation to the salt until 2 mols. of water have been absorbed.

The author, in view of these results, advocates that the official quinine sulphate should be the air-dried salt, having the following composition :—

$$(C_{20}H_{24}N_2O_2)_2.H_2SO_4.2H_2O$$

The variation in the amount of water in commercial samples of quinine sulphate is shown by the fact that in 40 samples examined, the amount ranged from 8·1 to 15·95 per cent. (Pharm. J. [3], **16**, 797).—A. S.

Indian Hemp, &c. (Apocynum cannabinum, &c.), [Refuse Plant Fibre]. H. C. Dyer. Indian Pharmacologist, 1896, **1**, 85.

See under XIX., *page* 62.

Jalap Tubers, Cinchona Plants, &c., Beneficial Effect of Judicious Manuring on the Production of Essential Principles. D. Hooper, Indian Pharmacologist, 1896, **1**, 89.

See under XXIV., *page* 72.

Quinine, Note on the Estimation of. D. Howard. Pharm. J. 1896, 505.

See under XXIII., *page* 70.

PATENTS.

New Medicinal Compounds [Anti-Rheumatic Agent]. Manufacture or Preparation and Production of. J. Y. Johnson, London. From The Vereinigte Chininfabriken Zimmer and Co., Frankfort-on-the-Maine. Eng. Pat. 1202, January 17, 1896.

This patent claims as new bodies, alkyl derivatives of the compound of *p*-amidophenol with *m*-hydroxysalicylaldehyde, $RO.C_6H_4N:CH.C_6H_3(OH)(OR)$, ($R = CH_3$ or C_2H_5), and the process of manufacture, which consists in condensing the two substances mentioned and alkylating the product, or in condensing the previously alkylated substances, or alkylating one of them, then condensing, and finally alkylating the second hydroxyl group.—A. C. W.

Aromatic Waters, such as Elder Flower Water, Rose Water, and the like [Combined Action of Acid and Fermentation]; Impt. in the Manufacture of. P. Auchinachie, London. Eng. Pat. 19,603, Sept. 4, 1896.

The addition of tartaric, citric, or other non-volatile organic or inorganic acid to the aromatic herbs or flowers, and after heating to boiling, allowing to stand for two or three days, or until fermentation begins, is claimed. The distilled product is of purer quality and odour.—A. C. W.

Saccharine, Impts. in the Manufacture of. [Action of Ammonia on o-Benzoyl Sulphonic Dichloride.] Chemische Fabrik von Heyden, Radebeul, Germany. Eng. Pat. 21,026, Sept. 22, 1896.

The process claimed in Eng. Pat. 1885, 6626, for preparing the ammonium salt of saccharine by treating o-benzoylsulphonic chloride with ammonium carbonate gives a very small yield. The present patent claims the process which consists in converting the dichloride into the diamide by adding it to a cooled aqueous solution of ammonia or ammonium carbonate in excess with constant stirring, finally heating to 100° C., and then boiling the diamide with a dilute alkali (1 molecule) to convert it into a saccharine salt.

$$NH_2CO—C_6H_4—SO_2.NH_2 + NaOH =$$

$$C_6H_4\left\langle\begin{matrix}CO\\SO_2\end{matrix}\right\rangle NNa + NH_3 + H_2O.$$

—A. C. W.

XXI.—PHOTOGRAPHY.

PATENTS.

Coloured Plates for Obtaining Coloured Representations by Photography, Impts. relating to. A. Baumgartner, Lorrach, Germany. Eng. Pat. 22,138, Nov. 20, 1895.

One, or more, oil colour is printed on to a celluloid plate, previously coated with gelatin, from a granulated lithographic stone or zinc plate. The plate is then dipped into

an aniline dye, which will dye those parts only which are not covered by the oil paint, after which process another oil colour may be printed on, partly on the dyed, and partly on the printed spots, whereby compound shades may be produced.—I. S.

Photographic Developers [Quinoline Hydride Derivatives], Impts. in. A. M. Clark, London. From Lembach and Schleicher, Biebrich-on-the-Rhine. Eng. Pat. 371, Jan. 6, 1896.

The use of the hydrides of hydroxyquinoline and hydroxytoluquinoline and their substitution products as photographic developers is claimed, and as a new developer, a solution of tetrahydro-o-hydroxyquinoline or other of the above compounds in alkaline sodium sulphite solution. The concentrated solution is diluted before use. Fogginess due to overdeveloping has not been observed in the use of these compounds.—A. C. W.

XXII.—EXPLOSIVES, MATCHES, Etc.

Cap Composition, The Analysis of. F. W. Jones and F. A. Willcox. Chem. News, 1896, **74**, 283.

See under XXIII., *page* 67.

Nitroglycerin, Researches on the Toxic Power of. Th. Bokorny. Chem. Zeit. 1896, **20**, 1021.

See under XVIII. B., *page* 61.

Phosphorus, the Toxic Action of Aqueous Solutions, and the Solubility in Water of. Th. Bokorny. Chem. Zeit. 1896, **20**, 1024.

See under XVIII. B., *page* 61.

PATENTS.

Smokeless Explosives, Impts. in the Manufacture of. H. Maxim, London. Eng. Pat. 14,311, Aug. 30, 1895.

In making explosives consisting of nitrocellulose and nitroglycerin gelatinised by means of acetone, the inventor proposes to add to the highly nitrated guncotton, 8—10 per cent. of nitrocellulose soluble in nitroglycerin, and then treat the mixture of these nitrocelluloses and nitroglycerin with acetone in a closed vessel which can be heated.

The addition of the nitrocellulose soluble in nitroglycerin is said to render the material much more plastic, and to prevent cracking in drying.—W. M.

Explosives [Flame extinguishing] applicable for Use in Coal or other Fiery Mines; Impts. in the Manufacture of. W. J. Orsman, Gathurst. Eng. Pat. 22,698, Nov. 27, 1895.

The object of this invention is to produce an explosive which shall liberate hydrochloric acid on detonation, but at the same time obviate the necessity of chlorinating the nitro compound serving as the combustible in the mixture. Naphthalene or some other easily attacked non-nitrated hydrocarbon is chlorinated. This highly chlorinated hydrocarbon is mixed cold in suitable proportions with ammonium nitrate and dinitrobenzene to produce an explosive.—W. M.

Explosives [Safety], Impts. in or relating to. W. Greaves and E. M. Hann, both of Aberaman, Glamorganshire. Eng. Pat. 24,847, Dec. 27, 1895.

The object of this invention is to render safe and flameless, the explosives of the nitroglycerin and guncotton class, by the addition of either ammonium oxalate, tartrate, formate, or acetate, but preferably oxalate.—W. M.

Explosives, Impts. in Apparatus [Vacuum Drier] for the Manufacture of. H. F. Talbot, Birmingham. From O. Siedentopf, Berlin. Eng. Pat. 5640, March 13, 1896.

The patent relates to a vacuum drier partially constructed with plates of a less strength than the body. In the event of explosion the former only are destroyed and can be readily replaced.—W. M.

Explosives, Impts. in the Manufacture of. W. P. Thompson. From "La Société des Explosifes Industriels," Paris. Eng. Pat. 20,069, Sept. 10, 1896.

The object of this invention is to produce a shattering explosive by the addition of picric acid and naphthalene, potassium chlorate, tar and dinitrobenzene to gelatinous explosives composed of nitroglycerin and nitrocotton.
—W. M.

Gunpowder, Smokeless. F. A. Halsey and W. C. Savage, both of San Rafael, Marin, Cal., U.S.A. Eng. Pat. 22,120, Oct. 6, 1896.

A smokeless gunpowder is described. Its composition for use in shot guns is: ammonium picrate, 68 per cent.; potassium bichromate, 25; and potassium or sodium permanganate or potassium pertungstate, 7 per cent.; but for rifles and other arms, the proportion of ammonium picrate may be increased and the other ingredients diminished.
—W. M.

XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

PATENTS.

Products of Combustion, A New or Improved Apparatus [Two Gas Meters] for Estimating the Carbonic Acid or other Constituents in Gases or. G. Craig, Cumnock, Ayrshire, N.B. Eng. Pat. 22,303, Nov. 22, 1895.

This is an arrangement and combination of a gas-analysis apparatus comprising two gas meters, with a vessel containing material which shall absorb the constituent desired to be estimated in the gases passed from one meter to the other, and an aspirator, or suction pump, or ejector.

The first meter measures the original volume of gas taken, and the second, the balance of gas or product left, from which may be deduced the efficiency of combustion.
—L. J. de W.

Specific Gravity, A New or Improved Apparatus for Separating Bodies of Different. C. S. Meacham, Maidstone. Eng. Pat. 1177, Jan. 17, 1896.

The apparatus consists of an exterior outer vessel which when in use is filled with water, an internal conical vessel having its sides perforated, and closed by a plug at the bottom, and a receiving tray which is placed beneath the plug-hole of the internal vessel. The apparatus is more particularly adapted for separating malt into the portion which sinks and the portion which floats in water. The measured portion of malt is thrown into the internal conical vessel and stirred up with the water; the sinking corns pass through the plug-hole and are caught in the receiving tray placed beneath; the floating corns remain in the internal vessel, and on inserting the plug can be withdrawn, the water draining away through the perforations.—A. L. S.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Pyrophosphoric Acid, Estimation of. Berthelot and G. André. Comptes rend. 1896, **123**, 773—776.

During their researches on the phosphoric acids, the authors discovered that magnesium pyrophosphate usually carries down with it more or less magnesium, according to the conditions of precipitation. Instead of weighing the precipitate thus obtained, the authors redissolve it in dilute nitric acid and boil gently for an hour, thereby converting the pyrophosphoric acid into orthophosphoric acid, which can then be precipitated and weighed in the usual manner.

The analysis of a mixture containing pyro- and orthophosphoric acids is conducted in the following way:—The pyrophosphoric acid is first precipitated as magnesium pyrophosphate, which is redissolved and reprecipitated as indicated above. The filtrate from the first precipitation contains all the orthophosphoric acid; hence it is only necessary to concentrate the solution and precipitate as magnesium ammonium phosphate. Some test analyses are given, showing the accuracy of the method.—J. S

F

Borax, Commercial; Valuation of. G. Heid. Zeits. f. ang. Chem. 1896, 679.

The author determines gravimetrically, by precipitation with silver nitrate, the chlorine present in—

1. The original sample (5 grms.).
2. The residue (of sodium chloride and boric acid) obtained by boiling a solution of the sample (1 grm.) with an excess of hydrochloric acid, and evaporating to complete dryness. The difference between the results (1) and (2) represents chlorine equivalent to the sodium of the sample existing as borax.

$$Cl_2 = Na_2B_4O_7.$$

—H. T. P.

Gold and Silver, Estimation of, in Auriferous Minerals. P. Truchot. Ann. Chim. anal. appliq. 1896, **1**, 365.

100—200 grms. of the finely powdered ore are carefully roasted to expel sulphur, arsenic, &c., and are then digested for 36 hours with an excess of saturated bromine water. The solution is filtered off, acidified with hydrochloric acid, concentrated to about 200 c.c., again filtered, finally precipitated warm with an acid solution of ferrous sulphate, and the separated gold collected and weighed. The silver remains behind as bromide in the residue, from which gold has been removed, and may be extracted by means of a hot solution of ammonium chloride, and determined by known methods.—H. T. P.

Lead, Volumetric Estimation of. A. Longi and L. Bonavia. Gazz. chim. ital. 1896, **26**, [1], 327.

The authors have carefully tested some of the published methods. Domonte's process—precipitation with a titrated solution of sodium sulphide—is unsatisfactory on account of the indistinctness of the final point of reaction. Hempel precipitates the lead by ammonia and an excess of oxalic acid, and determines the excess of oxalic acid remaining in solution, or the combined acid in the precipitate, by titration with permanganate. This method yields results which are either too high or too low, according as the first or second variation is adopted. Better results are obtained when oxalic acid is determined in a precipitate formed under the following conditions:—The lead solution must not be too dilute, oxalic acid must be used in large excess, precipitation being made complete by an addition of alcohol, and finally the precipitate should be washed with the same liquid.—H. T. P.

Tungsten from Titanium, Separation of. E. Defacqz. Comptes rend. 1896, **123**, 823—824.

Tungsten may be rapidly and completely separated from titanium by the following method, no matter whether the substances exist together in the free state as an alloy or in the state of oxides:—

The sample is ignited in a platinum crucible with seven or eight times its weight of a mixture of 8 parts of potassium nitrate and 2 parts of potassium carbonate at a dull red heat for 20 or 30 minutes. The fused white mass is treated with water, filtered, and washed, first by decantation and then on the filter with a solution of ammonium nitrate. The tungsten is estimated in the filtrate by precipitating as tungstate of mercury, whilst the residue, which contains the titanium, is dried, ignited, treated with potassium bisulphate, and the titanium estimated in the usual way.

In order to obtain good results, the fusion should not occupy more than one hour at the outside, and the temperature should not rise above dull redness.—J. S.

Commercial Copper, Analysis of, by the Electrolytic Method. A. Hollard. Comptes rend. **123**, 1003—1005.

The author adopts the following method for estimating, not only the copper, but also the impurities—arsenic, antimony, nickel, cobalt, &c.—in commercial copper:—

Apparatus.—A spiral of platinum wire and a truncated cone, each weighing about 20 grms., are the electrodes. The former is attached to a foot (Luckow). The latter is formed of platinum foil, autogenously soldered at its edges. Its diameter is 18 mm. above and 45 mm. below, and its side measures 63 mm A rod of hard platinum is soldered with gold to this truncated cone. Bohemian glass beakers, about 65 mm. diameter at the bottom, are used for the electrolyte.

Method: Estimation of the Copper.—10 grms. of clean filings, separated by a magnet from particles of iron, are used. These are placed in a beaker of 350—400 cc. capacity, covered with water, and 15 c.c. of sulphuric acid, followed by 40 c.c. of nitric acid of 36° B., are added. The author varies these proportions of acid, if a greater or less weight of copper is used for the analysis. The beaker is covered with an inverted funnel, and a gentle heat used when the first reaction has slackened. Refined copper yields a clear solution. Sulphur and sometimes oxygen compounds of antimony remain in unrefined copper solutions. If the latter residue be small it may be ignored. If considerable, it must be filtered off and the oxides dissolved by aqua regia. This solution is evaporated to dryness, taken up with hydrochloric acid containing tartaric acid and water, and is subsequently added to the original solution.

The copper solution is diluted to about 350 c.c. The electrodes are placed in it, the spiral connected to the + and the cone to the − poles of the battery. The distance between the lower edge of the cone and the foot of the spiral should be about 6 mm. The distance is increased or diminished, according to the amount of copper to be deposited. It must never exceed 10 mm. The upper edge of the cone must be 10—20 mm. below the surface of the solution. The vessel must be covered. The current must be 0·30 ampère. The completion of the removal of the copper is checked by withdrawal of a few c.c. of the solution and addition of ammonia solution. The author notes that the blue coloration is no longer visible, when the copper present is less than 0·017 grm. in the whole volume of the electrolyte. The current must be allowed to pass for some hours after this point is reached.

The complete deposition requires 2—3 days, with the current, and under the conditions named. The deposit is very adherent, has a smooth surface, and is of a rich red colour. The cone and spiral are removed from the electrolyte without breaking the current. They are washed by dipping successively into two beakers of distilled water. The cone is then immersed in alcohol, and, whilst still wet, placed in a drying oven. Silver is deposited with the copper. One must therefore deduct from the weight of copper, that of the silver subsequently found. If the copper used for analysis contain lead, this will be found partly in solution, and partly deposited as lead peroxide on the spiral.

In a future article, the author will deal with the exact determination of the impurities of commercial copper.

—J. B. C. K.

Lead in Water, A Simple Method of Detecting and Estimating Minute Quantities of. J. C. Berntrop. Chem. Zeit. 1896, **20**, 1020.

The author observes that reports from two chemists on a sample of water containing minute traces of lead will often be contradictory. The water may have been concentrated by one, but not by the other, before the sulphuretted hydrogen was passed through it. To secure the delicacy of the concentration method, and yet avoid its inconvenience, the author proposes to precipitate by an adequate quantity of sodium phosphate, the calcium and magnesium salts in the water. The precipitate contains also the lead. After settling for 24 hours, the supernatant liquid is siphoned off, and the precipitate is collected on a filter and dissolved in a little dilute nitric acid. The solution is concentrated to drive off excess of acid, and is then treated with sulphuretted hydrogen. The quantity of lead, if very minute, may at this stage be estimated colorimetrically, but if the precipitate be considerable, it is redissolved in nitric acid, and the lead is determined in the solution by one of the recognised methods, such as precipitation by sulphuric acid. Sulphuretted hydrogen was found to give a distinct coloration when applied according to the author's method to solutions containing 0·0001 and 0·000025 grm. of lead per litre. Two quantitative estimations in a solution containing 0·005 grm. of lead per 10 litres gave 0·0049 and 0·0041 grm. of lead. Before applying the method to very soft water, some solution of calcium chloride should be added to it.—J. A. B.

"Cap Composition," The Analysis of. F. W. Jones and F. A. Willcox. Chem. News, 1896, **74**, 283.

The authors describe a simple method for the analysis of "cap composition," which usually consists of a mixture of potassium chlorate, antimony sulphide, and mercury fulminate. It depends on the fact that the latter substance is soluble in acetone saturated with ammonia, whilst the two former are not. A tared filter paper is placed in a funnel, to the neck of which has been fitted a piece of rubber tubing provided with a clip. The paper is moistened with the solution of acetone and ammonia, the cap composition previously ground in an agate mortar (of course, with due care, and taking only small quantities at a time), weighed off directly on to the filter paper, covered with the solution of acetone and ammonia, and allowed to stand for 3 or 4 hours. It is then washed with the same solution until the washings give no coloration with ammonium sulphide, then with pure acetone until the washings give no residue on evaporation, and finally dried and weighed.

The paper is again put in the funnel, washed with water until free from potassium chlorate, and then dried and weighed.

The loss of weight after the first extraction gives the weight of mercury fulminate; the further loss after the second extraction, the weight of potassium chlorate; and the weight of the residue, that of antimony sulphide. The results of the analyses, by this method, of two mixtures of known composition, are given below :—

	A.		B.	
	Percentage taken.	Percentage found.	Percentage taken.	Percentage found.
Antimony sulphide	36·47	36·25	37·34	37·22
Potassium chlorate	33·25	33·71	46·03	46·43
Mercury fulminate	30·27	30·02	16·61	16·34

—A. S.

Nitroso-β-Naphthol, Use of, in Inorganic Analysis. R. Burgass. Zeits. f. ang. Chem. 1896, [19], 596—601.

According to the researches of the author on the action of nitrosonaphthol as a reagent with regard to the analytical separation of the most commonly occurring metals, the following results are worthy of notice :—

1. Nitrosonaphthol quantitatively precipitates, either singly or together, the following metals :—Cobalt, Copper, and Iron.

2. There remain in solution :—mercury, nickel, chromium, manganese, lead, zinc, aluminium, cadmium, magnesium, calcium, beryllium, antimony, and arsenic.

3. The following are partially precipitated, and so interfere in precipitations by means of nitrosonaphthol when they are present in a mixture :—silver, tin, and bismuth, and they must be first removed as silver chloride, stannic oxide, and bismuth oxychloride respectively.

4. In like manner, in the case of precipitations of iron, the presence of phosphoric acid is injurious, as is also that of tungstic and molybdic acid, when iron, cobalt, and copper are to be precipitated. (See this Journal, 1893, 711.)

—A. S.

ORGANIC CHEMISTRY.—QUALITATIVE.

Beer, Detection of Salicylic Acid in. J. J. La Bière, **4**, [12], 182.

The author in examining the distillate from pale and dark beers, found in every case, especially in the dark beers, that a red-violet coloration was produced with ferric chloride, and although the reaction is weaker if the distillation be performed by the aid of a paraffin bath instead of over a naked flame, and if two drops of concentrated sulphuric acid have been previously added per 100 c.c. of beer taken, it cannot be entirely eliminated. This source of error, can however, be avoided by the use of Millon's reagent (1 c.c. of mercury in 10 c.c. of concentrated nitric acid) freshly prepared, two drops producing a decided rose-coloration in presence of as little as 1 part of salicylic acid in 500,000 parts of water. The addition of the sulphuric acid also facilitates the distillation of the salicylic acid. The red-violet coloration with ferric chloride is apparently due to the presence of maltol which exists ready formed in torrefied malt and dark beers, and is probably also formed by the overheating, inevitable at the end of the distillation of the beer. (See this Journal, 1894, 670, and 1891, 165).

—C. S.

Ethylene and Benzene, Estimation of. F. Haber and H. Oechelhäuser. Ber. 1896, **29**, 2700—2705.

The authors uphold the statement made by Treadwell and Stokes (this Journal, 1889, 64), as to the utility of bromine water for the complete absorption of ethylene, and refute Winkler's conclusions (this Journal, 1889, 570), that although bromine water is rapid in its action, it is useless as an absorbent for ethylene in gas analysis. Winkler asserts that bromine water absorbs benzene incompletely; Treadwell and Stokes, however, found its action to be rapid and perfect. The authors detail experiments upon the absorption of benzene by bromine water, their object being to ascertain the extent of bromination, if any, or to determine whether the benzene is removed from the gas by mechanical means. For this purpose benzene was mixed with a measured quantity of standardised bromine water in such a manner, that the vapour of bromine was brought into intimate contact with the gas for about two minutes in diffused daylight. No loss of bromine was observed, the benzene being carried down mechanically with the bromine vapour. As a result of this work, the authors have devised a method of estimating the respective amounts of ethylene and benzene in mixtures of these gases. In the case of a mixture of ethylene, benzene vapour, and paraffins, the benzene is first separated by washing with paraffin oil, after which the bromine method can be employed.—D. B.

ORGANIC CHEMISTRY.—QUANTITATIVE.

Polarisation, Basic Nitrate of Lead as a Clarifying Agent for. F. Herles. Zeits. Zuckerind. Böhm. 1896, **21**, 189—193.

This clarifying agent, proposed seven years ago by Herles, and since then highly praised by Poupe, Stift, Petzival, Neumann, and others as having a surprising decolorising power, has the further advantage that solutions clarified with it may be used directly for inversion polarisation without further decolorising by charcoal.

For this purpose, Herles determined the inversion constant which is higher than that for solutions not clarified, being 143·5 for 0° C. The Clerget formula takes then the following form :—

$$S = \frac{100\,P - I}{143 \cdot 5 - \frac{t}{2}}.$$

For the determination of saccharose in presence of raffinose, from the polarisation at 20° C. of the inverted solution clarified as above—

$$S = \frac{0 \cdot 5124 - I}{0 \cdot 8474}$$

or for temperatures other than 20° C.—

$$S = \frac{(0 \cdot 4724 + 0 \cdot 002t)\,P - I}{0 \cdot 2074 - 0 \cdot 003t} \text{ and}$$

$$R = \frac{P - S}{1 \cdot 85},$$

where S = sugar (saccharose), P = direct polarisation, I = polarisation after inversion, t = temperature of the inverted solution, R = anhydrous raffinose.

The two solutions required are :—*Alkali.*—2 litres of water and 90 grms. of sodium hydrate. *Nitrate of Lead.*—2 litres of water and 1 kilo. of nitrate of lead. For 26·048 grms., there are to be used: for molasses and osmose waters, 15 to 18 c.c.; for raw sugar, first products, 1 to 2 c.c.; for after products, 3 to 4 c.c.; for second and third products, boiled, 12 to 15 c.c. of the nitrate of lead solution, to each c.c. of which 1 to 1·1 c.c. of the alkaline

solution is added. If more alkali is used than is necessary to form basic nitrate of lead, the polarisation is found too low, owing to the injurious action of the alkalinity. By using the proportions given, an excess of lead nitrate remains, which has no marked influence on either the direct polarisation or that after inversion, and which, on the contrary, neutralises the alkalinity of the clarified solution, and consequently prevents the error in polarisation due to this alkalinity. With the use of basic acetate of lead, which reacts alkaline, there is always risk of this error.

Solutions clarified by basic nitrate of lead, even when the nitrate has been employed in excess, may be used direct for inversion polarisation for which solutions clarified with excess of basic acetate of lead are not suitable, since, on adding the hydrochloric acid for inversion, weak acetic acid is set free, and the solution would not invert under the usual conditions of work.

Moreover, the polarisation of solutions clarified by this means gives more exact results than when basic acetate of lead is used. It is known that an excess redissolves a part of the precipitate formed, and as this may be optically active, the polarisation may be affected. (Degener, Berl. Zeits. 1885, 121.)

Neumann (Zeits. Zuckerind. Böhm. 1896—1897, 21) has found that with an excess of basic acetate of lead some molasses give lower and lower polarisations, and a darker colour of the solution was observed.

Solutions clarified by this means filter quickly and remain clear a longer time.

At the meeting of Austro-Hungarian chemists this year it was decided to use clarification by nitrate of lead for determining sugar in molasses by means of the Clerget inversion only, the solution for direct polarisation being clarified by basic acetate of lead. The author is of opinion that to obtain exact results, the nitrate should be used in both cases if his formula is to be employed.—L. J. de W.

Molasses, Contributions to the Analysis of. D. Loiseau. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 349—365.

Reichardt and Bittmann named those substances "plus sugar," which are present in beetroot molasses derived from the strontia and other processes, which remain dextro-rotatory after treatment with hydrochloric acid.

In the author's investigations a polariscope was used, requiring a normal weight of 16·19 grms. A solution of molasses containing 20 times this weight, or 323·8 grms. per litre, is treated with an amount of basic lead acetate producing the maximum defecation. If, as is often the case, the solution is too dark to read, it is filtered, and decolorised by passing through a column of animal charcoal; the application of char being based on the fact that it continues to exert decolorising influence when saturated with sugar. The author finds that 200 grms. of char per litre of defecated molasses is generally enough for this purpose. In the event of the solution of molasses being very alkaline, it is neutralised before passing through the column. The flow is regulated by a tap, and successive portions are polarised until the readings are constant. An experiment showed that a solution of pure sugar when passed through a column of char successively read 76, 89, 96, 99, 100 per cent. Therefore, if 100 to 125 c.c. of the decolorised molasses per 100 grms. of char be neglected, it will be found that the remainder contains all the sugar.

It is pointed out that the usual methods of inversion act on substances other than raffinose and cane sugar, for example, the dextrins, and, as it is necessary to estimate these, it is recommended that the inversion be carried out in the following manner:—50 c.c. of the decolorised solution are measured into a flask with an equal volume of 10 per cent. sulphuric acid. The flask is shaken, closed with a rubber stopper, immersed in a bath of water at 50° C., and kept at this temperature for $1\frac{1}{2}$ hours. Sometimes it is useful to take the reading of the defecated solution acidified with acetic acid before inversion, in order to nullify any effect arising from the use of basic lead acetate. It is advised that the tube should remain for 10 minutes at a temperature of 20° C before polarising, the results being checked before and after by reading a tube containing a solution of pure invert sugar of known strength. After inverting at 50° C. and reading at 20°, the author finds a rotation of −0·34 for cane sugar and +0·51 for raffinose per saccharimetric degree. Therefore the difference between the reading of an inverted molasses solution, and that of a solution of pure sugar, which before inversion had a rotation equal to the defecated molasses, divided by the algebraic sum (0·85) of the readings in scale divisions of raffinose and cane sugar after inversion, will give the rotation due to raffinose. For instance, the reading of a solution of molasses was 50° before inversion and −14° after. A solution of pure sugar polarising 50° when inverted would read −17°. This corresponds to a difference of 3°, and hence the rotation of the raffinose in the molasses will be $\frac{3}{0\cdot85} = 3\cdot5°$ and for sugar 50°—3·5° = 46·5°. The coefficient 0·85 was found to vary with the temperature, thus at :—

$$15° \text{ C} = 0\cdot365 + 0\cdot50 = 0\cdot865$$
$$25° \text{ C.} = 0\cdot315 + 0\cdot52 = 0\cdot835.$$

In studying the fermentation of molasses the author determines by preliminary experiment the quantity of 10 per cent. sulphuric acid necessary to invert a defecated molasses solution, under the conditions that, *firstly*, the inversion should commence at the temperature of the boiling water-bath, and *secondly* that it should be completed within five minutes. In one of the examples given, the quantity of acid necessary was 9 grms. of $H_2SO_4.H_2O$ for every 100 grms. of molasses. A volume of 400 c.c. is taken for fermentation, half of which consists of inverted molasses, and 8 grms. of top fermentation yeast; these quantities are introduced into a flask with a wide mouth, closed with a rubber stopper, and fitted with a bent tube dipping into mercury. It is kept at 24° to 25° C. for six to seven days, fermentation being considered at an end when two weighings of the flask and its contents within an interval of 24 hours are constant. The solution is filtered and the specific gravity, acidity, and content of alcohol determined. Should raffinose, dextrin, and saccharin be sought for, the following cases present themselves. 1. If the solution has no specific rotatory power, raffinose is absent. 2. If it has a dextro rotation and does not reduce Fehling's solution, raffinose is absent, but other substances such as dextrin and saccharin are present. If there should be a reduction, it arises from the presence of derivatives of noncrystallisable sugar in the molasses. 3. If the solution has a rotation of 2·5° and reduces Fehling's solution to the extent of 1 grm. of glucose to a litre of solution, raffinose may be considered to be present; but if the rotation be higher for the same reducing power, a dextrin and saccharin are also present. It is necessary to know whether the substances with rotatory power remaining in the solution after fermentation were pre-existent or due to raffinose; this can be ascertained by fermenting with bottom fermentation yeast, in which case dextrin and saccharin remain unaltered. The author carries out this determination by taking the de-alcoholised fermentation product, and making up to the original volume with water. One-third is fermented afresh with top-, the remainder with bottom-fermentation yeast. When fermentation is ended, each solution is polarised; if the reading of the latter be lower than that of the former, the difference divided by 0·785 is the rotation of the raffinose. To distinguish between saccharin and dextrin, the bottom yeast fermentation experiment is concentrated to one-half, and when cool made up to the original volume with 10 per cent. sulphuric acid, and boiled for three hours. If no glucose, is formed saccharin only is present; but should there be glucose, a fresh fermentation will reveal the existence of any other substance not attacked by acid.—J. L. B.

Molasses, The Clarification of Solutions of, for Inversion. K. C. Neumann. Zeits. Zuckerind. Böhmen, 1896, **21**, 183—188.

In the laboratory of Dr. Nevole and Neumann basic nitrate of lead has been used to clarify solutions of molasses, during the past five years, in determining the sugar by the Clerget method, and thereby perfectly clear solutions are

obtained which can be polarised without further decolorising. For the calculation, the direct polarisation is made on solutions clarified by basic acetate of lead, and the polarisation after inversion, by nitrate of lead, and Herles' constants are used.

He finds that the polarisation of the filtrate from the nitrate seldom agrees with that from basic acetate of lead, and is usually 0·6 to 0·8 per cent. higher. Finding that a number of samples of molasses from a factory working the strontian process gave a lower direct polarisation than with the acetate, he mixed the rest of the samples for further examination.

The liquid was of normal colour and smell, feebly alkaline (0·09 per cent. to litmus), and contained only 0·29 per cent. of invert sugar.

By clarifying with varying amounts of basic acetate of lead (from 2·5 to 20 c.c.), polarisations were obtained varying from 63·7 to 61·5 per cent., the usual 10 c.c. of acetate giving 62·6 per cent.

The variations with the nitrate of lead varied somewhat with the amount of alkali added.

After inversion, the dark half normal solution was treated with 6 grms. of char for half-an-hour and then gave at 20° C. in the 200 mm. tube −3°·6, −3°·8, −4°·0 or an average of −3°·8, a very uncertain reading.

The solution clarified with basic nitrate of lead was, after inversion by hydrochloric acid, very clear and easily polarised, giving −4°·50, −4°·45, and −4°·40.

Calculations were then made from the data obtained.

Result A by the basic acetate of lead, using Clerget's formula and the factor 142·7.

Result B by basic nitrate of lead, using Herles' formula and the factor 143·5.

Result C by basic acetate for the direct polarisation and basic nitrate of lead for the inversion polarisation, and the factor 143·5.

—	C.	B.	A.
	Per Cent.	Per Cent.	Per Cent.
Polarisation	62·60	62·45	62·60
Clerget	53·64	53·53	52·90
Cane-sugar......	48·49	48·41	47·29
Raffinose	7·62	7·59	8·27

The results B and C being thus in accord.—L. J. de W.

Raw Sugar Factories, Collated Methods of Analysis for the Control and Working of. Feleman, K. C. Neumann, and F. Herles. Bull de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 392—405.

This paper consists of descriptions of sample-collecting and analysis of materials dealt with in a raw-sugar manufactory, and comprising diffusion, saturation, filter-press work, evaporation, and the control of the massecuite. Diffusion juice, when collected hot, will keep for some hours; but, if it is required to be kept for a longer time, a convenient preservative is 10 c.c. of carbon bisulphide per litre of juice. The solution should be polarised immediately after filtration, as otherwise it clouds in the polarimetric tube.

The sugar in fresh and exhausted cossettes is determined by placing the required weight of pulp free from large pieces in a graduated Pellet's flask by the aid of a nickel funnel and rod. The funnel is rinsed with hot water and the flask three-quarters filled with water at 90° C. and agitated by shaking over on end. The froth is removed by alcohol; and for every 26·048 grms. of pulp, 4 or 5 c.c. of basic lead acetate are added, and the flask filled up to the containing mark with hot water. The contents of the flask are revolved, allowed to settle for 15 minutes, cooled to the temperature of the surrounding atmosphere, and filled up to the requisite volume with water or lead acetate. After being shaken vigorously, the solution is filtered and polarised, a correction being made for the volume occupied by the pulp. In the examination of exhausted cossettes, it is recommended to polarise in a 400 m.m. tube.

A convenient and sufficiently accurate method for calculating the percentage of froth in the first and second saturations, is to multiply the parts per 100 of dry lime used, by four.

In the massecuite-control, the amount of sugar indicated by analysis is not realised owing to losses occurring during the concentration and evaporation. These, which are called indeterminate losses, do not vary much in different factories. The presence of sugar in the condensation waters, occasions considerable damage to the heaters, &c., and it should be tested for at least twice a day by the magenta test.

The authors, in conclusion, point out that the control calculations should be made out every 24 hours.—J. L. B.

Massecuites, Determination of Density of. St. Tirpitz. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 372—374.

The author proposes that a temperature of 105°—107° C. should be employed to estimate the water in massecuites, which should be mixed with purified pumice, the individual grains being the size of sand.

For the determination of the direct density of the massecuite, a thick walled cylindrical stoppered glass vessel of a litre capacity is recommended, it being urged that the method of dilution so commonly practised, is incorrect.

—J. L. B.

Sugar, A New Method for the direct Estimation of, in the Beetroot by the Le Docte Apparatus. F. Sachs. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 377—382.

Of the three methods of extraction, namely, the indirect method based on the analysis of the juice, and the alcoholic and aqueous methods, the author considers the last mentioned to be the most satisfactory.

The Le Docte apparatus consists of a pipette, having at the bottom a three-way tap, connected with reservoirs of basic lead acetate and water, the third tube serving as an exit for the contents. The water and lead acetate being introduced from below, the top of the pipette is surrounded with a closed cap in case of an overflow. 5 c.c. of basic lead acetate are run in and the apparatus filled up with water to its containing mark (177 c.c.). The normal weight of pulp is directly weighed into a tinned copper capsule, the contents of the pipette added, and the capsule covered with a cap, consisting of a piece of glass surrounded with well vaselined rubber. After shaking, the cap is carefully removed by drawing it along the edge of the capsule; the contents are then filtered and polarised.—J. L. B.

Invert Sugar, the Influence of Lead Salts on the Estimation of, by the Fehling-Soxhlet Method. A. Borntrâger. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 453.

An examination of solutions of invert sugar of varying concentrations, with or without dilutions of the copper solution, has shown that the results are much lower in the presence of lead salts. This influence is considerably augmented by dilution. In the analysis of wines, the author found it useless to precipitate the excess of lead, unless more than $\frac{1}{10}$th the volume of liquid consisted of basic lead acetate. For precipitation, either phosphate or carbonate of sodium is preferable to the sulphate.—J. L. B.

Alcohol, the Estimation of. H. D. Richmond. J. Fed. Inst. Brewing, 1896, **2**, 529—535.

According to Tabarie the density of alcohol in a liquid is found by dividing the density of the liquid by that of the non-volatile portion or "extract." Blunt has shown that more correct results are obtained if the density of the extract be subtracted from that of the liquid *plus* that of water. It is generally agreed that Tabarie's method is theoretically correct; the author, however, shows from mathematical considerations, Blunt's proposition to be the true one. When the determination of the density does not yield sufficiently accurate results, the following method, based on that of Hehner, for the detection of methyl alcohol, which was derived from that of Dupré, may be used. It depends on the equation—

$$3C_2H_6O + 2K_2Cr_2O_7 + 8H_2SO_4 = 3C_2H_4O_2 + 2K_2SO_4 + 2Cr_2(SO_4)_3 + 8H_2O.$$

The liquid to be examined is distilled in the usual manner, with or without the addition of water. A quantity of the distillate, containing from 0·2 to 0·3 grm. of alcohol, is diluted to about 100 c.c., and placed in a stoppered bottle; about 1·5 grm. of potassium bichromate accurately weighed, and 5 c.c. of strong sulphuric acid are added to the liquid. The bottle is placed in water, which should be gradually raised to the boiling-point, and frequently shaken until the bichromate is dissolved. The boiling should be continued for two hours with occasional shaking; it is then allowed to cool slightly, the stopper removed, and a 7 to 8 per cent. solution of ferrous sulphate, containing 10 c.c. per litre of sulphuric acid, is run in from a burette, until a drop of the solution forms a blue coloration with potassium ferrocyanide. The excess of ferrous sulphate is titrated back with a dilute solution of potassium bichromate (5 grms. per litre). The ferrous sulphate solution should be standardised upon the dilute bichromate solution, and the quantity of ferrous sulphate solution added, calculated to its equivalent of bichromate; the amount of bichromate used during the back titration is added to the original amount, and the quantity equivalent to the ferrous sulphate solution used is subtracted from this; the difference divided by 4·278 will give the amount of alcohol.

A solution containing 42·78 grms. of potassium bichromate per litre may be used instead of weighing out the bichromate, and this diluted 10 times, can be employed for the back titration.

This method affords good results if the conditions are adhered to, the amount of sulphuric acid and time of heating being important.

As a check, the acetic acid may be estimated: the solution obtained after titration is placed in a flask and distilled down to 15 c.c., successive portions of 15 c.c. of well-boiled water are added, and 15 c.c. distilled off each time, till the distillate does not require more than two drops of $\frac{1}{10}$ N alkali to render it alkaline; the collected distillate is neutralised with caustic soda solution, to which a little baryta has been added; the amount of caustic soda used cannot, however, be taken as indicating the amount of acetic acid, since sulphuric acid is always found in the distillate; if the bulk of liquid be very large, it is advisable to concentrate it at this stage. A quantity of dilute and well boiled sulphuric acid is added to the concentrated neutralised solution, which should then be distilled to a small bulk; portions of 10 c.c. of boiled water are successively added and distilled off until the distillate is neutral. The mixed distillate should then be titrated with $\frac{1}{10}$ N alkali, using phenolpthalein as indicator. The acetic acid found is calculated to alcohol by dividing by 1·3043.

If the two determinations agree closely it may be taken as evidence that methyl alcohol is absent, since this alcohol gives no volatile fatty acid, being completely oxidised to carbon dioxide and water.

To show that higher alcohols are absent, Duclaux's method may be employed. When the distillate containing the volatile acid has been neutralised and concentrated, and there has been added a measured quantity of sulphuric acid, slightly in excess of the quantity equivalent to the alkali used for neutralisation, it is diluted to 110 c.c. This is placed in a stoppered distillation flask the upper part of which is surrounded with a jacket of compo-piping. The liquid is then heated nearly to boiling and steam passed through the coil; the liquid should then be distilled, and when 10 fractions of 10 c.c. each have been collected, portions of 10 c.c. of boiled water are added and successively distilled off. Each fraction (the whole of the distillate after the water is added should be collected and treated as one fraction) is titrated separately, and the amounts noted. It is advisable to neglect the first fraction, and add the amount of acid obtained in the other 10 together to obtain the total quantity for the purposes of calculation. Taking this as 100, the quantity of acid in each fraction should be calculated as a percentage of this. Calling the percentage distilled x, and the percentage of total acid in that quantity y, the amount in the distillate at any point is expressed by the formula $100 - y = \frac{100(100 - x)^{\frac{3}{2}}}{100^{\frac{3}{2}}}$.—J. L. B.

Critical Temperatures of Solution [Butter Analysis] in Open Tubes. L. Crismer. Bull. Assoc. Belge des Chimistes, **10**, [8], 312—316.

For butter analysis, the operation can be performed in open test-tubes surrounded by an air- or water-bath, provided absolute alcohol be employed, the critical temperature in such case (48° C.) being lower than the boiling point of the solvent. With dilute alcohol the temperature increases parallel with the proportion of water present, *viz.*, 72° C. with 4 per cent., 82·5° C. with 6 per cent., 99° C. with 9 per cent. of water, and so on.

The most suitable quantities to work with are 2 vols. of alcohol to 1 of butter (about $\frac{1}{2}$ c.c.), the temperature remaining constant between the limits of $1\frac{1}{2}$ and 3 vols. of alcohol. Heat is applied by a small flame, with agitation, and as soon as the liquid becomes homogeneous it is cooled until turbidity ensues, the temperature at this point being noted. The test is repeated as before, after addition of a small quantity of alcohol, as a check on the first determination.

The average critical temperature of a number of samples of pure butter, with alcohol of sp. gr. 0·7967 at 15·5° C. (= 0·9 per cent. of water) was 54° to 55° C., whereas margarine and adulterated butters ranged from 63·8° to 78° C., the temperatures obtained in sealed tubes, with alcohol containing 9 per cent. of water, being respectively *circa* 100° C. and 120° to 124° C.

Some pure butters give lower temperatures, but this is owing to rancidity or acidity (due to preparation from sour cream), the normal temperature being obtained after purification. It is noted as curious that the number of c.c. of $\frac{1}{10}$ N KHO required to neutralise the acidity, when added to the critical temperature of the acid butter, gives figures closely approximating to the critical temperature of the purified butter. This temperature (for pure butter) varies inversely with the degree of acidity, and therefore affords a direct indication of the latter and of the care taken in preparing the butter.—C. S.

Quinine, Note on the Estimation of. D. Howard. Pharm. J. 1896, 505.

In spite of the great difference in solubility between the sulphates of quinine and of the other alkaloids, and of the ready crystallisation from ether, of cinchonidine, cinchonine, and quinidine, the ease with which quinine and cinchonidine form more or less definite compounds when crystallising as sulphates from water or as bases from ether, makes the problem difficult. Liebig's test—shaking with ammonia and ether—will pass as pure, a sample containing 10 per cent. of cinchonidine sulphate. Kerner's ammonia test, which in its different modifications is the most widely adopted, can only give empirical results, because of the difficulty of dissolving out cinchonidine sulphate when crystallised with quinine sulphate, and this difficulty is increased by the non-homogeneity of the crystals deposited from an impure solution of quinine sulphate, the composition varying with the temperature. If the strong solution be slowly cooled, nearly pure quinine sulphate is deposited above 50° C.; below that temperature, mixtures with cinchonidine sulphate in increasing proportion separate. Thus, when the solution for the ammonia test is made by shaking with cold water, as prescribed in Ph. Ger. II., a solution of the outside of the crystals being obtained, the more rapidly the salt has been crystallised in the factory, the purer it will appear to be. To obviate this difficulty the crystals may be effloresced by heating, as in the U.S. Ph., or the sulphate may be digested with hot water and the mixture allowed to cool, with frequent shaking. Hesse and Weller have shown that 60° C. is the best temperature for this digestion; if the salt be entirely dissolved, all the difficulties of the uncertainty of the composition of the salt deposited are intensified. If a boiling solution of sulphate of quinine be saturated with the free alkaloid, on cooling, part of the latter separates with the sulphate; the solution left and the solution of the sulphate deposited will both require much more ammonia to redissolve separated quinine than the neutral salt requires. A very small quantity of ammonium or sodium sulphate greatly diminishes the solubility of quinine sulphate.

The method proposed by Kubli (this Journal, 1896, 681) has no advantages over the older method (Hesse, Arch. Pharm. **234**, 191, see following abstract; also Weller, Pharm. Zeit. 1896, No. 28).

In regard to the ether test, Prunier has shown that if a solution of impure quinine sulphate be cooled to 50° C., and kept at that temperature for some time, with frequent shaking, most of the quinine crystallises out, and if the liquid be filtered at 50° C., the solution will contain within 2 per cent. of the total cinchonidine. If this solution be evaporated to a small bulk and the magma be shaken with just enough ether and ammonia to produce momentary solution, crystals form, which in amount are in a definite ratio to the cinchonidine in the original sample, though they do not contain the whole of this alkaloid. If these crystals amount to more than 2 per cent. of the original sulphate, the process of crystallisation at 50° C. must be repeated. In the author's experience this process is on the whole more reliable than the ammonia test.

In the method of De Vrij and Hesse, which depends on crystallising the quinine as far as possible as bisulphate and estimating the cinchonidine in the mother-liquor by shaking with ether and ammonia, owing to the large amount of quinine still remaining in solution, the cinchonidine will remain to a great extent in the ethereal solution. Thus, to obtain accurate results, a sufficient quantity of the sample must be used to permit of the mother-liquor from the first batch of crystals being concentrated and again crystallised, before extraction of cinchonidine from the second mother-liquor. In a complete analysis, the impure cinchonidine obtained, is dissolved in absolute alcohol and 2·1 c.c. of 50 per cent. sulphuric acid added for each grm. of crystals; the cinchonidine crystallises out almost entirely as tetrasulphate, and, on neutralising the mother-liquor and evaporating, the quinine is obtained as sulphate. The ethereal solution containing cinchonidine should be evaporated, the alkaloid crystallised as bisulphate, and the mother-liquor again treated with ether and ammonia. These last operations are generally unnecessary in practice, for, with care, the quinine crystallising with the cinchonidine very nearly balances the cinchonidine dissolved by the ethereal quinine solution. If it be required to estimate the hydroquinine generally found to the extent of 2—4 per cent. in commercial samples, the alkaloid in the ethereal solution is dissolved in dilute sulphuric acid and oxidised at 0° C. by 4 per cent. permanganate added drop by drop till the solution when filtered is only slowly decolorised. The manganese oxide is filtered and washed, the solution treated with ether and ammonia, the alkaloid extracted from the ether by dilute sulphuric acid, the solution neutralised and crystallised. No indication of the presence of hydroquinine is given by the ether test.

The ammonia test offers great advantages as a test for pure quinine sulphate; it is, however, very doubtful if it is wise to insist on the use of the pure salt in pharmacy.

—A. C. W.

Quinine Sulphate, Analysis of. O. Hesse. Arch. der Pharm. 1896, **234**, 191.

An examination of the tests proposed by Kubli (this Journal, 1896, 681).

The Water Test.—In preparing the solution for this test the author finds that the duration of the boiling has no influence on the result; it should therefore be prolonged to complete solution. In order to obtain comparable results by this method it is necessary to use a salt completely effloresced. In place of the 10 c.c. of water required to destroy the opalescence, in the case of Russian quinine sulphate, 11·5, 15, and 16·5 c.c. were required.

The Carbonic Acid Test.—The results obtained by this test do not agree with results obtained by the official processes, which latter agree among themselves. Thus the test is wanting in accuracy, and cannot control the water test. However, the two processes may be used as a test for a pure sample, but not to give a measure of any impurity.

—A. C. W.

Theobromine in Preparations of Cocoa, Determination of. A. Eminger. Apotheker Zeit. 1896, 716.

One part of theobromine dissolves in 736·5 parts of water at 10°, in 136 at 100° C., in 5,399 parts of 90 per cent. alcohol at 18° C., in 440 on boiling, in 818 of boiling absolute alcohol, in 21,000 of ether at 17°, in 4,856 of methyl alcohol at 18°, in 5,808 of chloroform at 18°, and in 2,710 parts of boiling chloroform. Theobromine is completely insoluble in carbon tetrachloride. It cannot be left in contact without alteration with the alkaline earths, the alkalis, or hydrated oxide of lead.

The author proposes to make use of the different behaviour of caffeine and theobromine on heating, to separate the two substances; caffeine melts at 178° and sublimes at 180°, whilst theobromine sublimes at 220° C. without melting. In the following method the insolubility of theobromine in carbon tetrachloride at 18° C., is made use of. 10 grms. of the powdered substance are left in contact with 150 c.c. of petroleum ether for 12 hours, with frequent shaking; the fat extracted contains no caffeine. The residue is dried, 5 grms. are boiled with 100 grms. of 3 to 4 per cent. sulphuric acid with a reflux condenser for about half an hour, until the characteristic cocoa red is formed. After exact neutralisation with baryta water, the residue is evaporated together with sand, and the residue extracted by chloroform for five hours in a Soxhlet apparatus. After distilling off the solvent, the residue is dried at 100° for one hour, and then treated with carbon tetrachloride (not more than 100 grms.) for one hour, with frequent shaking; fat and caffeine dissolve, the solution is evaporated, the residue extracted by boiling water, and the aqueous extract evaporated in a tared dish. The theobromine remaining in the flask, and the filter through which the carbon tetrachloride has passed, are boiled with water, the liquid filtered, the residue washed, the solution evaporated, and the theobromine weighed.

In different samples of cocoa, the following amounts have been found :—

——	Theobromine.	Caffeine.
	Per Cent.	Per Cent.
Puerto Cabello	1·05	0·16
Maracaïbo	1·84	0·15
Cauca	2·03	0·36
Caracas	1·43	0·07
Ceylon	2·06	0·30
Java	2·34	0·05
Trinidad	1·98	0·09
Para	1·08	0·20
Granada	1·90	0·26
Surinam	1·83	0·20
Guayaquil ariba	1·20	0·20
„ marsala	0·88	0·20
Kameroun (Cameroons)	1·83	0·12
Saint Thomé	2·09	0·12
Bahia	2·04	0·16
Samana	1·82	0·16
Cap Haiti	2·07	0·16
Domingo	1·98	0·16

—A. C. W.

Carbon and Nitrogen in Organic Compounds, Estimation of, in the Wet Way. P. Fritsch. Annalen, 1896, **294**, 79—88.

The author has experimented with a view to combining, in the analysis of organic compounds, the Messinger wet combustion method for estimating carbon with the Kjeldahl-Krüger method of nitrogen estimation. He finds that oxidation with strong sulphuric acid and potassium bichromate, yields good carbon determinations when the apparatus is so arranged that the powdered bichromate can be added in very small quantities at a time. This is effected by the addition of a side tube on the air-inlet tube of the boiling flask, the tube containing the powdered substance being attached to this side tube by india-rubber tubing. The residue in the oxidation flask can at once be used for determining the nitrogen as ammonia by boiling with potash in the case of amides, ammonium bases, pyridine, quinoline, and indole derivatives, alkaloids, bitter principles, albuminoids, and similar substances; it cannot, however, be

satisfactorily used with nitro- or nitroso-compounds or nitrates, the methods proposed for previous reduction of the oxy-nitrogen groups all proving unsatisfactory.—L. T. T.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Glucina, Properties of Pure. P. Lebeau. Comptes rend. 1896, **123**, 818—821.

The author finds that pure glucina is fusible and volatile at the temperature of the electric furnace. When fused it does not vary sensibly in density, and retains the property of being attacked by concentrated acids. Boron, silicon, and carbon are capable of reducing glucina, giving crystalline compounds; whilst sodium, potassium, magnesium, and aluminium have no action on it.—J. S.

Nitric Oxide, Absorption by Ferrous Bromide. V. Thomas. Comptes rend. **123**, 943—945.

Ferrous salts have long been known to absorb large quantities of nitric oxide. The classic researches of Peligot proved that most probably a definite compound was formed; but he could not separate it. Recently Gay has confirmed Peligot's work; and, using the sulphate, the double ammoniacal sulphate, and the chloride of iron, he has proved that these salts absorb nitric oxide in two definite proportions, according to the temperature.

The author has undertaken a continuation of these researches, using ferrous bromide in aqueous solution, and gives, in his original communication, details of his method. He has proved that below 10° C., ferrous bromide absorbs nitric oxide, with the formation of a compound having the formula $6FeBr_2.4NO$; and that above 10° C. (15°—16°), an absorption of nitric oxide occurs, corresponding to the formula $2FeBr_2.NO$. These results confirm those of Gay. —J. B. C. K.

Nitrogen, The Fixation of, by Algæ and Bacteria. R. Bouilhac. Comptes rend. 1896, **123**, 828—830.

From experiments the author finds that *Schizothrix lardacea* and *Ulothrix flaccida* cannot grow in nutritive solutions which are free from nitrogen, even in the presence of bacteria of the soil. In the case of *Nostoc punctiforme*, however, it is otherwise. The association of this alga with bacteria allows of the simultaneous development of both species with fixation of nitrogen. According to the analyses, the amount of nitrogen fixed by this plant is comparable with the amount fixed by the Leguminosæ. As in the case of *Nostoc*, bacteria capable of fixing nitrogen can live in solutions of arsenic acid containing 1 part in 10,000.—J. S.

Jalap Tubers, Cinchona Plants, &c.; Beneficial Effect of Judicious Manuring on the Production of Essential Principles. D. Hooper. Indian Pharmacologist, 1896, **1**, 89.

For many years the jalap plant (Ipomœa purga) has been successfully cultivated in the Government cinchona plantations at Dodabetta, Nilgiris, but about two years ago it was found that the plants were in an unhealthy condition, the tubers small and containing only 12·9 per cent. of resin; some tubers transferred to a freshly manured soil contained after some months 15·3 cent., and a large tuber grown near a manure heap contained 22 per cent. of resin—the largest yield on record. The soil of the Nilgiris is deficient in lime and phosphoric acid, the addition of which has always been beneficial to the local tea, coffee, and cinchona estates. Experiments were undertaken to observe the action of these substances on the growth of jalap tubers. The results demonstrated that the application of phosphatic manures largely increases the weight of the tubers, and raises the proportion of the active principle.

The question of manuring is one of great importance in cultivating plants for the purpose of increasing their active principles. It has been proved over and over again in cinchona culture, that nitrogenous and phosphatic manures increase the amount of alkaloids in the bark; in the same way, potash is good for tobacco crops; magnesia and lime for the sugar cane. There is no doubt that better results would be obtained in growing medicinal plants if attention were directed to a proper system of applying suitable fertilising agents to the crops.—A. C. W.

Strophanthus Hispidus, Oil of the Seeds of. A. Mjoen, Arch. der Pharm. **234**, 283.

This oil, of a strong green colour, has the density 0·928; it gives on saponification:—formic, acetic, palmitic, and oleïc acids, and glycerin.—A. C. W.

New Books.

Tables for the Quantitative Estimation of the Sugars. With Explanatory Notes. By Dr. Ernst Wein. Translated, with Additions, by William Frew, Ph.D. E. and F. N. Spon, London. Spon and Chamberlain, New York. 1896. Price 6s.

8vo volume, containing preface to this, the English edition, table of contents, and text filling 128 pages. There is a frontispiece representing a Soxhlet tube (filled) drawn to scale. The little work contains the following items:—Description of Method for the Estimation of Sugars. Note on Alternative Method, and Preparation of Solutions. The tables have reference to the following subjects:—I. Estimation of Dextrose. II. Of Maltose. III. Of Milk Sugar; and IV. Of Invert Sugar (*Meissl*). V., VI., and VII. Estimation of Invert Sugar in presence of Cane Sugar (*Meissl*). VIII. Estimation of Invert Sugar in Beet Sugar (*Herzfeld*). IX. Estimation of Invert Sugar (*Lehmann*). X. Of Levulose; and XI. Of Starch. XII. Volumetric Estimation of Dextrose (*Reischauer*); and XIII. Of Maltose (*Reischauer*).

Lubricating Oils, Fats, and Greases: Their Origin, Preparation, Properties, Uses, and Analysis. By George H. Hurst. Scott, Greenwood, and Co., Ludgate Hill, London. 1896. Price 10s. 6d.

8vo volume, containing frontispiece, preface, table of contents, and subject-matter filling 301 pages, four pages of tables, and an alphabetical index. The work is illustrated with 65 wood-cut illustrations.

The text is divided into eight chapters, devoted as follows:—

I. Introductory, Oils and Fats, Fatty Oils, Hydrocarbon Oils, Uses of Oils. II. Hydrocarbon Oils. III. Scotch Shale Oils. IV. Petroleum. V. Vegetable and Animal Oils. VI. Testing and Adulteration of Oils. VII. Lubricating Greases. VIII. Lubrication.

Trade Report.

PRIZES.

Dr. Nobel's Bequest to Science.

The Paris *Figaro* of the 7th January 1897 states that the only valid will is that written and signed in Paris by Mr. Nobel on the 27th November 1895 in the presence of four of his co-patriots, and opened at Stockholm on the 30th December last, which, in addition to legacies to about 20 of his relatives, friends, and servants, contains the following provisions:—

"All the remainder of my realisable fortune shall be disposed of as follows:—

"The capital realised and invested in safe securities by the liquidators was constituted, of which the interest shall be annually distributed to those who, during the past year, shall have rendered the most eminent services to humanity. The interest shall be divided into five equal parts, and shall be distributed:—

"The *first* to the one who in the domain of physics shall have made the most important discovery or improvement:

"The *second* to the one who in the domain of chemistry shall have made the most important discovery or improvement:

"The *third* to the one who will have made the most important discovery in the domain of physiology and medicine:

"The *fourth* to the one who in the domain of literature will have produced the work highest in the ideal sense:

"The *fifth* for the one who shall have acted most and best for the fraternity of nations, for the suppression or reduction of standing armies, and the constitution and propagation of peace congresses.

"The first two prizes (physics and chemistry) shall be awarded by the Academy of Sciences of Sweden, that for physiology and medicine by the Corolin Institute of Stockholm, and the literary prize by the Swedish Academy; and that for the propagation of peace by a commission of five members elected by the Norwegian Diet (Storthing).

"It is my express wish that in distributing these prizes no consideration of nationality shall prevail, so that he who is most worthy of it shall receive the reward whether he be Scandinavian or not."

TARIFF CHANGES AND CUSTOMS REGULATIONS.

(*From the Board of Trade Journal.*)

Netherlands—Guiana.

Regulations as to Methylated Spirits of Local Manufacture.

A report has been received from H.M. Consul at Paramaribo, stating that owing to the practice amongst the natives of using methylated spirits of local manufacture as stimulants, the mixture of a mineral methylic alcohol with the methylated spirits manufactured there has now become compulsory. The authorised methylators—methylic alcohol and benzine—are now exempt from import and excise duty.

GENERAL TRADE NOTES.

Acetylene Regulations in Paris.

J. Gas Lighting, Dec. 22, 1896, **68**, 1230.

According to some provisional regulations lately issued by the Prefect of Police in Paris, manufactories of carbide of calcium and of acetylene compressed to more than $1\frac{1}{2}$ atmospheres, or liquefied, are to be placed in the first class of dangerous buildings. Manufactories of acetylene gas not compressed, or compressed to less than $1\frac{1}{2}$ atmospheres, if for public use, are to be in the first class; if for private use, in the third class.

Alcohol in Canada.

Chem. and Druggist, Jan. 2, 1897, 6.

Canadian alcohol costs about 1·15 dols. per gall. The same stuff can be obtained in the United States at 35 cents a gall. The reason for the higher price of the Canadian spirit is that the excise duty is $2·80\frac{1}{2}$ dols. per gallon, and the import duty $3·71\frac{1}{4}$ dols. The drug trade of Canada (we learn from the *Canadian Druggist*) do not like this, and have appealed to the Tariff Commissioners to adjust the matter, thinking that 20 per cent. *ad val.* duty instead of 250 per cent. is quite enough protection.

Aluminium as a Conductor of Electricity.

Eng. and Mining J., Dec. 19, 1896, 581.

The new plant of the Pittsburg Reduction Company, at Niagara Falls, known as the "Lower Works," located on the high bank of the river below the Falls, has been started. The Pittsburg Reduction Company resolved to use aluminium rods or cables instead of copper for conducting the current from the generators in the power-house down in the gorge, some 200 ft. below, to the plant on the top of the bank. These rods are each $\frac{3}{8}$ in. in diameter and 350 ft. long from the generators to the main line in the furnace room. These main line conductors, which traverse the pot room, are from 12 to 16 ins. wide and 1 in. thick, and are also of aluminium.

A Chemical Plant at Niagara.

Eng. and Mining J., Dec. 19, 1896, 581.

The new plant of the Mathieson Alkali Company, to be erected at Niagara Falls, will consist of several buildings covering 2 acres. The first section is to be completed by January 15th, 1897. The company which, as is well known, manufactures caustic soda by the Castner process, will employ 150 men and use 2,000 e.h.p. at its new works. It already has large plants in England and at Saltville, in Virginia. At the latter plant no water power is available. A very complete electric plant for the chemical process and for lighting is installed, Westinghouse apparatus being used.

The Rise of Chemical Industry in Russia.

Chem. and Druggist, Jan. 9, 1897, 56.

Some time ago the Russian Ministry of Finance had under consideration a project for reducing or abolishing the duties on all drugs and pharmaceutical preparations that are not, or cannot conveniently be, produced in Russia. This scheme, if carried into effect, would give a much-needed stimulus to our export trade with that empire, which, so far as drugs and chemicals are concerned, has long been languishing. Some portion of the export trade in crude drugs formerly done between Britain and Russia has been transferred to continental ports, another to the countries of production themselves. Heavy chemicals, such as bleaching-powder and other alkalies, for which Russia used to be one of our best customers, are now made in that country itself, to an ever-increasing extent. The result is seen in our Board of Trade statistics of alkali exports to Russia, which show dwindling figures year by year.

In Germany, the importance of Russia as a buying market is quite appreciated. The tariff war of 1892–93 seriously dislocated the business relations between the two countries, but since the conclusion of the new Russo-German commercial treaty, German exports to Russia have reached a higher figure than ever before. The value of goods sent from Germany to Russia, converted into millions of pounds, was:—

1889.	1890.	1891.	1892.	1893.	1894.	1895.
£	£	£	£	£	£	£
8·70	9·17	7·27	6·49	6·78	8·53	10·39

But there is every reason to believe that, for several years to come, Russian commercial policy will, so far as the existing treaties of commerce permit it, tend towards increased rather than relaxed protection, and in this anticipation foreign capitalists have, for some time, shown a growing inclination to establish factories in Russia. Here again, though there are many British traders, technical managers, and foremen in Russia, the German element predominates. Many years may elapse before Russia can become a leading manufacturing country, but her growth towards industrialism has recently been shown in a remarkable manner at the "All-Russian" Exhibition in Nijni-Novgorod.

This National Exhibition, the fifteenth of its kind, was patronised by the Russian Government and generally supported by the manufacturers. It has attracted little attention abroad, but Pharmacist L. J. Volpian, of St. Petersburg, gives an account of the chemical part of it in the *Pharm. Zeitschrift für Russland*, which reveals the extraordinary advance of Russian chemical industry. The imports of nitric and hydrochloric acid into Russia have fallen from 19,000 in 1885 to 313 tons in 1894; in other words, the empire is now practically independent of the foreigner for its supply of two of the principal bases of chemical industry. As for that still more important article, sulphuric acid, Russia makes all she requires. No fewer than 15 sulphuric acid factories were represented at Nijni-Novgorod. As with the acids, so it is with the closely-allied alkali industry. This branch of manufacture struck root in Russia all the quicker, because there were no old-

established interests with antiquated processes to be got rid of. The semi-foreign manufacturers who established the soda industry in Russia, the firm of Liubimov-Solvay, maintain two enormous factories in the Governments of Perm and Ekaterinoslav. Two years ago these works produced 45,406 out of the 64,450 tons of soda crystals consumed in the empire. Soda-ash and caustic soda industries have also been successfully established. The caustic soda works are mostly situated in the neighbourhood of Baku, where the product is used in the refining of petroleum derivatives, and where the abundance of raw material (Mirabolith) enables the makers to turn it out cheaply by the Leblanc process. Along with the sulphuric acid industry there has sprung up in Russia an important manufacture of chromate salts, vitriol, phosphates, lead, zinc, tin, strontium, and copper salts, mineral dyes, &c., all of which find employment in the textile, pottery, and other young industries. Platinum is almost a Russian monopoly, and no one will grudge Russia the new industry of the manufacture of platinum apparatus represented at her last National Exhibition.

In the manufacture of organic chemicals, and especially in that of pharmaceutical preparations, Russia is still dependent upon the foreigner for nine-tenths of her requirements, although the proportion grows less year by year. But since the All-Russian Exhibition of 1882, two refineries of boracic acid have been established. Chloroform, sulphuric ether, tannin, collodion, chloral hydrate, and tartrate of antimony are also made in the country, though not in sufficient quantity to cover the requirements. And tartaric acid making has been commenced. It is said that the argol from the Russian wine-growing districts serves for raw material, but the quality is confessedly poor, 10 per cent. of "tartaric acid"—or, say, about 13 per cent. of bitartrate of potash—being the average yield. We wonder that it pays for working.

In medicinal plant growing, the progress of Russia is very great. Six castor-oil factories, all working from Russian-grown seed, were represented at Nijni-Novgorod. Castor-seed growing, like other new agricultural pursuits, is assisted by the Government. An essential oil industry is also springing up, principally in the southern provinces. But, with few exceptions, it is carried on in a primitive fashion, and yields wretched products. Oils of peppermint, wormwood, caraway, fennel, anise, and pine-needles are manufactured, but only the pine-needle oil is really good. The industry, however, faulty though it be, is making its influence felt upon the seed-market, for the exportation of essential oil seeds for distillation in Britain, Germany, and elsewhere has lately received a noticeable check.

Russian wool-fat and petroleum jelly are poor in quality. Benzine-distilling, on the other hand, is a flourishing business. This is not surprising in a country which, within the past 15 years, has developed the second largest petroleum industry in the world. From 31,500 galls. in 1882, the Russian benzine output had risen to nearly 1,570,000 galls. in 1894. The firm of Nobel owns 188 miles of petroleum pipe lines, and 1,157 tank-waggons for the conveyance of its products by rail.

The industry of the dry distillation of wood in Russia is only just beginning. In Northern Russia, away from the railways, there are still many thousands of square miles under wood, and a smaller area covers portions of the Caucasus. Yet up to the present only ½ per cent. of all the rosin, and but a slightly larger proportion of the turpentine, used in Russia has been of Russian manufacture. It has generally been assumed that the Russian fir (*Pinus sylvestris*) could not be made to yield turpentine and rosin of equal quality or abundance to the French or American pines. The Russian Government gave Prof. Flaziwski, of Kasan, a slice of Crown forest to experiment on, and he reports that Russian turpentine, if collected by the French process, does not differ, chemically or physically, from the French, except that it is dextro-rotatory to the same degree that the French is levo-rotatory. Moreover, the Professor has obtained a balsam from *Abies Sibirica* that will advantageously replace Canada balsam for technical and microscopic purposes. The day of the chemical exploitation of these forests is therefore dawning, and within a few years Russia may export, instead of buy from abroad, acetic acid, wood-naphtha, acetone, wood-vinegar, and acetate of lime.

The value of the output of chemicals of all kinds in Russia, according to the statistics of the Ministry of Trade and Industry (which need not be implicitly believed), was 15,437,000 r. in 1880, and 29,822,000 r. in 1890. The importation of chemical products into the country has declined from 19,262,000 r.'s worth in 1880 to 14,402,000 r.'s worth in 1890.

Nitrate of Soda.

Shipments, Consumption, Stocks, and Prices, from 1889 *to* 1896.

——	1889.	1890.	1891.	1892.	1893.	1894.	1895.	1896.
	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.
Shipments from South American ports to all parts for the six months ending 31st December.	573,000	692,000	494,000	373,000	550,000	623,000	733,000	600,000
Do. for the 12 months ending 31st December	930,000	1,028,000	759,000	784,000	939,000	1,073,000	1,218,000	1,068,000
Afloat for Europe on 31st December	380,000	514,000	321,000	215,000	358,000	393,000	506,000	347,000
Stocks in United Kingdom ports:—	30,000	24,000	23,000	20,000	9,100	17,300	21,500	24,000
Stocks in Continental ports on 31st December	160,000	172,000	191,000	185,000	92,900	101,700	134,500	324,000
Consumption in United Kingdom for the six months ending 31st December.	32,000	28,000	30,000	33,000	29,000	29,600	36,500	24,000
Do. in Continent for the six months ending 31st December	205,000	171,000	153,000	192,000	231,000	246,500	230,500	195,000
Do. in United Kingdom for the 12 months ending 31st December.	105,000	119,000	120,000	117,000	100,000	114,500	117,500	105,000
Do. in Continent " " "	564,000	666,000	703,000	685,000	682,000	749,500	789,500	805,000
Do. in United States " " "	80,000	104,000	100,000	100,000	104,000	100,000	110,000	115,000
Do. *in the World* " " "	749,000	889,000	923,000	902,000	886,000	964,000	1,017,000	1,025,000
Visible supply on 31st December (including the quantity afloat for Europe and Stocks in United Kingdom and Continent).	570,000	710,000	535,000	420,000	460,000	512,000	662,000	695,000
Price on 31st December per Cwt.	8*s.* 1½*d.*	7*s.* 7½*d.*	9*s.* 1½*d.*	9*s.* 1½*d.*	9*s.*	8*s.* 10½*d.*	7*s.* 7½*d.*	7*s.* 10½*d.*

——	1889.	1890.	1891.	1892.	1893.	1894.	1895.	1896.
	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.
Liverpool.	11,000	10,000	7,000	9,000	2,000	4,250	8,000	10,500
London...	4,100	3,250	3,900	3,500	3,300	1,000	2,100	2,250
Out ports.	14,900	10,750	12,100	7,500	3,800	12,050	11,100	11,300

—W. Montgomery & Co.'s Circular.

UNITED STATES MINERAL AND METAL PRODUCTION IN 1896.

Engineering and Mining Journal, January 2, 1897, 3.

No.	Products.	Customary Measures.	1895. Quantity. Customary Measures.	1895. Quantity. Metric Tons.	1895. Value at Place of Production. Totals.	1895. Value at Place of Production. Per M. Ton.	1896. Quantity. Customary Measures.	1896. Quantity. Metric Tons.	1896. Value at Place of Production. Totals.	1896. Value at Place of Production. Per M. Ton.
	NON-METALLIC.									
	Abrasives—				Dols.	Dols.			Dols.	
1	Corundum and emery	Sh. T.	385	349	56,400	161·60	372	361	55,189	..
2	Garnet	,,	2,065	1,873	93,350	50·00	2,365	2,135	97,350	..
3	Grindstones	,,	36,389	33,004	290,378	8·49	31,865	30,972	282,538	..
4	Millstones	,,	..	..	8,525	..	..	..	13,452	..
5	Tripoli and infusorial earth	,,	1,788	1,622	26,049	16·00	2,012	1,955	26,207	..
6	Whetstones	,,	1,609	1,459	78,303	53·67	610	553	29,309	..
7	Alum	,,	75,000	68,025	2,225,000	32·70	75,000	72,900	2,225,000	..
8	Antimony ore	,,	1,083	982	37,905	38·60	150	145	4,750	..
	Asbestos and Talc—									
9	Asbestos	,,	1,010	916	11,400	11·35	716	696	12,670	..
10	Fibrous talc	,,	66,500	60,316	665,000	11·03	50,000	45,260	500,000	..
11	Talc and soapstone	,,	18,885	17,129	361,353	21·00	7,219	6,990	64,595	..
12	Asphalt	,,	14,300	12,970	300,000	23·13	4,500	4,374	90,000	..
13	Asphaltic limestone	,,	..	..	..	..	700	680	3,500	..
14	Bituminous rock	,,	43,778	39,707	143,456	3·61	..	..	..	..
15	Bituminous sandstone	,,	..	..	..	..	17,000	15,422	68,000	..
16	Barytes	,,	20,255	18,371	99,020	5·39	18,000	16,329	86,543	..
17	Bauxite	L. T.	14,145	14,371	56,580	4·00	15,000	15,240	..	..
18	Borax	Lbs.	13,506,356	6,126	742,850	121·09	15,181,890	..	759,094	..
19	Bromine	,,	394,854	179	102,662	573·53	550,285	..	143,074	..
20	Cement, natural hydraulic	Bbls.*a*	7,694,053	1,047,006	4,597,285	4·30	6,728,103	..	3,629,666	..
21	Cement, Portland	Bbls.*b*	749,059	135,879	1,430,089	10·53	1,684,261	..	1,790,772	..
22	Clay, refractory	Sh. T.	*i*3,750,000	3,401,250	4,500,000	1·35	3,750,000	3,401,250	4,500,000	..
23	Clay, kaolin	,,	30,910	28,035	258,431	9·22	29,056	28,242	219,732	..
24	Coal, anthracite	,,	58,362,985	52,965,538	89,948,699	1·69	51,580,928	46,794,218	79,434,629	..
25	Coal, bituminous	,,	*h*137,398,347	126,627,141	125,344,248	1·00	141,720,099	128,568,474	127,548,089	..
26	Coke	,,	9,927,348	9,006,090	15,258,935	1·69	10,372,624	9,410,044	15,973,840	..
27	Cobalt oxide	Lbs.	6,400	3	8,640	2880·00	12,825	6	16,672	..
28	Copperas	Sh. T.	14,118	12,805	69,846	5·46	11,108	10,796	53,112	..
29	Copper sulphate	Lbs.	45,000,000	20,412	1,350,000	66·13	45,000,000	20,412	1,350,000	..
30	Chrome ore	L. T.	1,450	1,473	16,795	11·39	323	293	3,670	..
31	Feldspar	,,	22,195	22,550	104,082	4·67	19,923	20,241	103,599	..
32	Fluorspar	Sh. T.	4,000	3,628	36,440	10·04	..	..	..	..
33	Graphite	Lbs.	392,008	178	17,640	9·95	450,000	..	20,250	..
34	Graphite, amorphous	Sh. T.	1,100	998	4,700	3·00	574	557	3,850	..
35	Gypsum	,,	298,572	270,804	974,219	3·60	248,869	241,900	867,071	..
36	Iron ore	L. T.	16,950,000	17,221,200	29,662,500	1·72	17,000,000	70,272,000	32,300,000	..
37	Lime	Bbls.*e*	*i*60,000,000	5,443,164	30,000,000	5·50	..	5,443,164	30,000,000	..
38	Magnesite	Sh. T.	2,200	1,995	14,700	7·39	2,067	2,592	13,435	..
39	Manganese ore	L. T.	14,883	15,121	92,044	6·12	7,303	7,419	69,585	..
40	Mica, ground	Lbs.	750,000	340	31,956	94·00	772,000	..	33,332	..
41	Mica, sheet	,,	6,200	3	6,400	2133·00	3,000	..	3,150	..
42	Mineral wool	Sh. T.	6,742	6,115	69,481	11·36	7,659	7,444	82,814	..
43	Monazite	Lbs.	1,900,000	862	114,000	132·40	..	..	..	..
44	Natural gas	..	..	..	12,000,000	..	..	..	10,000,000	..
45	Paints, mineral	Sh. T.	47,084	42,705	1,086,767	25·40	75,219	73,112	1,070,556	..
46	Paints, vermilion	,,	118	107	118,190	1105·00	96	93	94,677	..
47	Paints, white lead	,,	95,389	86,537	9,061,965	104·00	89,313	87,688	7,378,332	..
48	Paints, zinc oxide	,,	22,690	20,198	1,588,300	77·43	16,799	15,239	1,595,905	..
49	Petroleum (crude)	Bbls.*d*	50,652,025	6,420,712	42,547,701	6·60	46,795,760	5,731,920	42,116,184	..
50	Phosphate rock	L. T.	831,498	844,802	2,577,643	3·00	312,718	317,721	1,049,655	..
51	Marls	,,	217,700	221,183	587,790	2·67	155,000	157,480	418,500	..
52	Precious stones	..	..	..	250,000	..	..	..	200,000	..
53	Pyrites	L. T.	81,000	82,296	353,160	4·29	73,822	75,003	186,698	..
54	Salt, evaporated	Bbls.*e*	12,521,498	1,539,178	5,844,348	3·78	10,958,751	1,391,349	5,432,105	..
55	Salt, rock	,,	1,367,638	173,662	518,740	2·99	1,157,000	146,998	138,840	..
56	Silica, sand and quartz	L. T.	523,640	532,018	553,128	1·04	634,355	644,504	992,676	..
57	Slate, roofing	Squares	645,361	190,277	2,062,239	10·83	537,063	179,021	1,726,790	..
58	Slate, manufactures	Sq. ft.	3,786,599	11,170	369,062	33·00	2,267,424	7,558	285,803	..
59	Soda, natural	Sh. T	1,900	1,724	47,500	27·56	..	..	..	..
60	Soda, manufactured	M. T.	..	167,000	3,841,000	23·00	..	..	3,500,000	..
61	Stone, limestone (flux)	Sh. T.	3,390,000	3,444,240	2,542,509	0·74	..	..	2,500,000	..
62	Stone, marble	Cu. ft.	6,912,533	568,593	4,086,261	7·18	6,116,847	..	2,729,727	..
63	Stone, onyx	,,	800	66	10,750	163·00	500	..	5,000	..
64	Other building stones	..	..	..	33,000,000	..	..	..	30,000,000	..
65	Sulphur	L. T.	1,650	1,676	126,950	15·75	1,500	..	100,000	..
66	Estimated product unspecified	..	..	..	5,000,000	..	..	..	5,000,000	..
	Total non-metals	..	..	..	437,383,364	..	..	..	410,999,987	..

(*a*) Barrels of 300 lbs. (*b*) 400 lbs. (*c*) 200 lbs. (*d*) 42 galls. (*e*) 280 lbs. (*f*) Troy ozs. (*g*) Flasks of 76½ lbs. (*h*) Bituminous coal includes brown coal and lignite. The anthracite production is the total for Pennsylvania, Arkansas, and Colorado. (*i*) Estimated. (*j*) Kilograms.

Abbreviations: Sh. T., short tons (2,000 lbs.): L. T., long tons (2240 lbs.); M. T., metric tons (2204·6 lbs.); Squares (100 sq. ft., lapped and laid).

United States Mineral and Metal Production in 1896—*continued.*

No.	Products.	Customary Measures.	1895. Quantity. Customary Measures.	1895. Quantity. Metric Tons.	1895. Value at Place of Production. Totals.	1895. Value at Place of Production. Per M. Ton.	1896. Quantity. Customary Measures.	1896. Quantity. Metric Tons.	1896. Value at Place of Production. Totals.	1896. Value at Place of Production. Per M. Tons.
	Metals.									
67	Aluminium	Lbs.	900,000	408	495,000	1213·23	1,300,000	..	520,000	..
68	Antimony	Sh. T.	433	393	68,847	175·28	596	579	83,440	..
69	Copper	Lbs.	386,453,850	175,294	37,944,988	210·76	453,824,000	..	48,786,080	..
70	Gold	Ozs. (f)	2,265,612	(j) 70,470	46,830,200	(j) 664·60	2,757,620	85,773	57,000,000	..
71	Iron, pig	L. T.	9,446,308	9,597,449	108,632,542	10·77	8,768,869	8,909,000	87,688,690	..
72	Lead, value at New York	Sh. T.	156,854	142,298	10,132,768	71·20	175,717	159,410	10,472,733	..
73	Platinum	Ozs. (f)	150	..	2,250	..	150	..	2,250	..
74	Quicksilver	Flks. (g)	33,978	1,179	1,318,589	1114·00	33,012	..	1,222,441	..
75	Silver, commercial value	Ozs. (f)	46,331,235	(j) 1,441,087	30,254,296	20·99	45,465,173	1,414,148	30,461,665	..
76	Zinc, spelter	Sh. T.	81,858	74,245	5,942,890	80·04	77,084	74,925	6,074,219	..
	Total metals	..	..	..	240,617,370	..	..	..	242,311,481	..
	Grand total	..	..	..	678,000,734	..	..	..	653,311,468	..

(*f*) Troy ozs. (*g*) Flasks of 76½ lbs. (*j*) Kilograms.

BOARD OF TRADE RETURNS.

Summary of Imports.

Articles.	Year ending December 31st. 1895.	Year ending December 31st. 1896.
	£	£
Metals	18,656,042	20,464,786
Chemicals and dyestuffs	6,558,813	6,784,845
Oils	8,111,850	8,446,709
Raw materials for non-textile industries.	44,024,597	47,240,453
Total value of all imports	416,689,658	441,807,335

Summary of Exports.

Articles.	Year ending December 31st. 1895.	Year ending December 31st. 1896.
	£	£
Metals (other than machinery)	28,886,549	33,572,894
Chemicals and medicines	8,288,831	8,243,601
Miscellaneous articles	31,387,408	33,583,916
Total value of all exports	225,890,016	239,922,209

Imports of Metals for Year ending 31st December.

Articles.	Quantities. 1895.	Quantities. 1896.	Values. 1895.	Values. 1896.
Copper :—			£	£
Ore .... Tons	99,757	87,335	574,503	566,791
Regulus .... „	91,267	90,799	2,232,860	2,286,509
Unwrought ..... „	42,538	61,169	1,831,806	2,882,453
Iron :—				
Ore .... „	4,450,311	5,417,476	2,977,952	3,761,722
Bolt, bar, &c. ... „	67,749	71,058	549,534	570,413
Steel, unwrought .. „	10,862	17,491	95,003	158,146
Lead, pig and sheet „	162,924	167,594	1,654,073	1,853,468
Pyrites .... „	582,463	589,046	987,165	999,793
Quicksilver .... Lb.	3,724,053	3,536,928	334,558	327,729
Silver ore .... Value £	..	..	1,746,444	1,296,736
Tin .... Cwt.	832,928	767,493	2,631,038	2,289,688
Zinc .... Tons	62,525	76,663	933,894	1,245,013
Other articles ... Value £	..	..	2,107,212	2,226,325
Total value of metals	..	..	18,656,042	20,464,786

Imports of Oils for Year ending 31st December.

Articles.	Quantities. 1895.	Quantities. 1896.	Values. 1895.	Values. 1896.
			£	£
Cocoa-nut .... Cwt.	285,016	219,204	321,550	249,633
Olive .... Tuns	14,834	18,850	522,811	612,876
Palm .... Cwt.	1,262,933	1,146,391	1,320,690	1,204,679
Petroleum .... Gall.	177,146,618	189,653,941	3,368,904	3,722,056
Seed .... Tons	36,031	33,424	721,807	691,021
Train, &c. .... Tuns	24,597	21,961	406,448	366,279
Turpentine .... Cwt.	503,683	498,791	520,065	490,381
Other articles .. Value £	..	..	929,575	1,109,784
Total value of oils...	..	..	8,111,850	8,446,709

Imports of Raw Materials for Non-Textile Industries for Year ending 31st December.

Articles.	Quantities. 1895.	Quantities. 1896.	Values. 1895.	Values. 1896.
			£	£
Bark, Peruvian .. Cwt.	32,206	35,291	58,804	61,578
Bristles .... Lb.	3,964,750	4,056,279	556,204	555,888
Caoutchouc ...... Cwt.	341,553	431,164	3,760,178	4,993,186
Gum :—				
Arabic .... „	68,351	80,506	169,282	195,752
Lac, &c. .... „	114,122	157,352	629,459	776,117
Gutta-percha .... „	48,077	43,805	389,258	401,490
Hides, raw :—				
Dry .... „	491,547	369,063	1,153,757	905,427
Wet .... „	771,133	604,728	1,650,369	1,319,501
Ivory .... „	10,911	10,907	467,976	458,961
Manure :—				
Guano .... Tons	49,842	20,214	392,309	104,554
Bones .... „	74,056	66,681	320,051	251,866
Nitrate of soda... „	122,687	106,445	988,897	836,552
Phosphate of lime „	359,659	291,263	633,314	465,931
Paraffin .... Cwt.	720,804	725,398	755,790	711,050
Linen rags .... Tons	25,033	21,055	229,645	206,772
Esparto .... „	186,408	187,279	791,236	796,683
Pulp of wood .... „	297,095	328,080	1,574,302	1,684,527
Rosin .... Cwt.	1,427,569	1,655,374	346,798	419,563
Tallow and stearin „	2,175,822	2,049,749	2,575,071	2,178,652
Tar .... Barrels	121,591	115,959	85,865	74,080
Wood :—				
Hewn .... Loads	2,260,761	2,433,795	4,181,436	4,889,393
Sawn .... „	5,061,986	6,032,222	10,695,916	13,380,660
Staves .... „	144,751	138,399	594,615	655,243
Mahogany ..... Tons	31,848	53,772	270,712	477,265
Other articles .... Value £	..	..	10,743,353	10,440,249
Total value	..	..	44,024,597	47,240,940

Besides the above, drugs to the value of 936,106*l.* were imported, as against 1,012,466*l.* in 1895.

Imports of Chemicals and Dyestuffs for Year ending 31st December.

Articles.	Quantities. 1895.	Quantities. 1896.	Values. 1895.	Values. 1896.
			£	£
Alkali............ Cwt.	184,114	176,156	114,586	95,930
Bark (tanners', &c.) „	384,959	309,814	146,491	114,969
Brimstone........ „	498,386	452,565	99,367	94,012
Chemicals...... Value £	..	..	1,276,670	1,391,608
Cochineal......... Cwt.	5,813	5,697	38,411	36,658
Cutch and gambier Tons	25,545	26,844	556,120	549,256
Dyes:—				
Alizarin ....Value£	..	..	302,218	256,344
Anilin and other „	..	..	407,775	483,002
Indigo........... Cwt.	83,071	89,958	1,392,534	1,553,722
Nitrate of potash . „	228,477	340,750	213,653	278,484
Valonia........... Tons	35,605	31,605	395,943	319,798
Other articles... Value £	..	..	1,615,045	1,631,061
Total value of chemicals	..	..	6,558,813	6,784,845

Exports of Drugs and Chemicals for Year ending 31st December.

Articles.	Quantities. 1895.	Quantities. 1896.	Values. 1895.	Values. 1896.
			£	£
Alkali............ Cwt.	6,248,800	4,841,079	1,557,593	1,242,028
Bleaching materials „	1,412,300	1,188,220	500,717	406,546
Chemical manures Tons	328,553	353,460	1,949,049	1,809,939
Medicines....... Value £	..	..	1,048,310	1,123,011
Other articles... „	..	..	3,233,222	3,662,077
Total value..........	..	..	8,288,831	8,243,601

Exports of Miscellaneous Articles for Year ending 31st December.

Articles.	Quantities. 1895.	Quantities. 1896.	Values. 1895.	Values. 1896.
			£	£
Gunpowder...... Lb.	8,046,800	8,166,400	181,507	191,096
Military stores.. Value £	..	..	2,074,358	1,829,011
Candles.......... Lb.	23,380,400	23,555,100	385,703	357,021
Caoutchouc..... Value £	..	..	1,190,885	1,262,981
Cement.......... Tons	395,184	353,863	641,551	580,518
Products of coal Value £	..	..	1,573,731	1,806,214
Earthenware... „	..	..	1,839,318	1,779,134
Stoneware...... „	..	..	153,015	189,032
Glass:—				
Plate........... Sq. Ft.	1,446,000	1,802,778	79,715	102,721
Flint........... Cwt.	94,211	100,210	214,334	227,492
Bottles......... „	680,650	775,986	324,901	370,845
Other kinds.... „	224,836	242,258	170,758	190,450
Leather:—				
Unwrought.... „	159,037	138,069	1,422,747	1,279,702
Wrought..... Value £	..	..	355,912	394,504
Seed oil.......... Tons	49,154	58,293	990,311	1,088,374
Floorcloth ..... Sq. Yds.	22,483,200	23,415,400	857,430	954,659
Painters' materials Val. £	..	..	1,556,579	1,657,808
Paper........... Cwt.	906,842	1,060,620	1,436,690	1,629,814
Rags............ Tons	49,239	52,977	293,821	286,883
Soap............ Cwt.	728,398	719,651	756,704	744,848
Total value..........	..	..	31,387,408	33,583,916

Exports of Metals (other than Machinery) for Year ending 31st December.

Articles.	Quantities. 1895.	Quantities. 1896.	Values. 1895.	Values. 1896.
			£	£
Brass........... Cwt.	107,182	119,212	418,601	487,907
Copper:—				
Unwrought.... „	590,927	466,626	1,344,913	1,139,690
Wrought...... „	313,601	308,052	844,031	892,040
Mixed metal... „	298,233	225,791	629,906	517,159
Hardware...... Value £	..	..	1,856,532	2,121,268
Implements..... „	..	..	1,240,327	1,413,681
Iron and steel... Tons	2,835,541	3,552,286	19,680,923	23,813,289
Lead............ „	41,705	41,247	493,196	523,228
Plated wares... Value £	..	..	345,714	389,705
Telegraph wires. „	..	..	782,375	866,996
Tin .... Cwt.	113,412	124,354	383,604	398,472
Zinc............ „	197,568	189,301	130,385	134,762
Other articles .. Value £	..	..	736,042	874,738
Total value.......	..	..	28,886,549	33,572,894

Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—PLANT, APPARATUS, and MACHINERY.

Applications.

28,691. G. Morriss. Improvements in muffles for annealing and like purposes. Dec. 15.

28,718. T. Hennessy. Improvements in and relating to gas generators. Complete Specification. Dec. 15.

28,728. P. E. Placet. Improvements in the lining of crucibles and furnaces. Filed Dec. 15. Date applied for July 24, 1896, being date of application in France.

28,742. H. B. Wright. Improvements in mechanical water purifiers. Complete Specification. Dec. 15.

28,763. J. Y. Johnson.—From The Electric Rectifying and Refining Co. *See* Class XVII.

28,893. G. Nayler and A. Nayler. An improved fire retort. Dec. 16.

29,000. P. J. E. E. Chambost. Improvements in apparatus for heating or evaporating by means of hydrocarbon liquids of any density. Dec. 17.

29,318. W. Stagg, R. T. Glover, and J. G. Glover. Improvements in and connected with pressure or vacuum gauges. Dec. 21.

29,377. J. Hill. Improvements in filter presses. Dec. 22.

29,491. J. V. Forns. Improvements in tele-thermometers. Complete Specification. Dec. 22.

29,576. W. W. Hughes. Improvements in or relating to electric furnaces. Dec. 23.

29,587. E. Gauhe and Gockel. Improvements in mixing drums. Complete Specification. Dec. 23.

29,757. R. H. Yeomans. Improvements in furnaces for evaporating pans and other purposes. Dec. 28.

29,878. Starkey Bros., Ltd., and F. Hinchliffe. Improvements in the means or method for the consumption of smoke in furnaces. Dec. 29.

29,884. T. G. Webb. Improvements in and connected with apparatus for concentrating sulphuric acid and other liquids. Dec. 29.

1897.

38. S. A. Everett. Furnace for burning powdered fuel. Jan. 1.

81. W. Glover. Improvements in apparatus for heating air for drying purposes. Jan. 1.

154. S. Shail. An improved muffle furnace. Jan. 4.

189. W. L. Gilder. Improvements in or relating to furnaces. Complete Specification. Jan 4.

446. T. Morton and W. S. McLennan. Improvements in washing apparatus. Jan. 7.

746. C. Gautier and X. Wehrle. Improvements in carburetting apparatus. Complete Specification. Jan. 11.

852. T. B. Lightfoot.—From The Gesellschaft für Linde's Eismaschinen. Apparatus for the production of ice and for cooling liquids by the expansion of compressed air. Complete Specification. Jan. 12.

853. T. B. Lightfoot.—From The Gesellschaft für Linde's Eismaschinen. Safety and regulating appliance for apparatus for compressing vapours of ammonia and other volatile liquids. Complete Specification. Jan. 12.

882. C. Guattari. An improved condenser. Jan. 12.

1138. H. H. Lake.—From D. H. Burrell and E. J. Burrell. Improvements in centrifugal liquid separators. Complete Specification. Jan. 15.

Complete Specifications Accepted.*

1895.

20,770. W. Busche. *See* Class IX.

1896.

424. G. E. Sherwin. Appliances for cooling and heating air and liquids. Jan. 13.

1228. D. M. Hawes. *See* Class XI.

2859. J. G. Lorrain. Apparatus for filtering and purifying liquids. Jan. 13.

4266. H. J. Fraser. Evaporative and other condensers, evaporators, and the like. Jan. 20.

6785. H. L. Doulton. Condensing worms constructed of pottery. Jan. 13.

11,345. A. Gates. Apparatus for mixing liquids. Dec. 23.

23,031. P. Funfak. Vessels for holding corrosive substances or the like. Jan. 13.

II.—FUEL, GAS, and LIGHT.

Applications.

28,518. J. Bland. Fuel cartridges. Dec. 14.

28,588. L. Mond. Improvements in or relating to the production of a mixture of air and water-vapour for gas producers. Dec. 14.

28,692. E. Bert. Improvements in the production of ozone. Dec. 15.

28,721. R. G. Leventhorpe. Improvements in the production of hydrogen gas and in apparatus therefor. Dec. 15.

28,722. H. R. Bean and H. Ringwood. Improvements in generators and holders for acetylene and other gases. Dec. 15.

28,776. A. G. Smith and A. J. Smith. A floating acetylene gas generator for the illumination of harbour works, floating buoys, ponds, rivers, or such like. Dec. 16.

28,853. W. P. Thompson.—From The Continental-Gas Gluhlicht-Actien-Gesellschaft "Meteor" vormals Kroll, Berger, and Co. Improvements in oil burners applicable for incandescent lighting. Dec. 16.

29,057. J. Edwards. Improved method of treating the mantles of incandescent gas burners to render them more refractory, the same being also applicable as a preservative of india-rubber. Dec. 18.

29,168. E. Richard-Lagerie. Apparatus for producing acetylene gas. Dec. 19.

29,188. W. P. Thompson.—From L. L. H. Gerard. Improvements in or connected with apparatus for producing acetylene gas. Dec. 19.

29,202. D. Nagy. A new or improved process for making brown coal and lignite coke. Complete Specification. Dec. 19.

29,246. J. Cole and J. E. Zealey. Improvements in and connected with the burning of oils for illuminating and heating purposes. Dec. 21.

29,301. P. de Resener and H. Luchaire. An improved process for the manufacture and purification of acetylene. Filed Dec. 21. Date applied for Aug. 21, 1896, being date of application in France.

29,320. P. de Resener and H. Luchaire. Improvements in apparatus for the manufacture of acetylene. Filed Dec. 21. Date applied for Aug. 21, 1896, being date of application in France.

29,422. P. Cannell-Bunn. Improved mantles for gas and other burners. Dec. 22.

29,435. D. Pemberton and J. Lewis. Improvements in acetylene and other gas generators. Dec. 22.

29,495. C. Guattari. Improvements in acetylene gas generating apparatus. Dec. 22.

29,500. E. Quelle. Improvements in apparatus for generating acetylene gas. Complete Specification. Dec. 22.

29,554. H. K. Spence, A. Beveridge, W. G. Beveridge, and S. M. Beveridge. Improvements in acetylene gas generators. Dec. 23.

29,596. J. O. O'Brien.—From P. P. H. Mace. Improvements in feed governors for apparatus for producing acetylene gas. Dec. 23.

29,597. A. Bandsept. Improvements in incandescent burners. Dec. 23.

29,603. J. Spurge. Improvements in gas-cleansing apparatus. Dec. 23.

29,613. A. Rommoser and A. von Hake. Improvements in and relating to incandescent lights. Complete Specification. Dec. 23.

29,736. C. Colas. Improvements in means for incandescent lighting. Complete Specification. Dec. 24.

29,750. G. Claude and A. Hess. An improved method of storing acetylene for lighting and other purposes. Dec. 24. Date applied for June 6, being date of application in France.

29,768. W. H. Dargue. An improved apparatus for the production, drying, purifying, and storage of acetylene gas. Dec. 28.

29,780. A. Dorst. A new incandescent light. Dec. 28.

29,852. P. de Resener and H. Luchaire. Improvements relating to burners for use with acetylene. Dec. 28. Date applied for June 30, 1896, being date of application in France.

29,858. F. Altmann. Improved method and apparatus for burning liquid fuel. Complete Specification. Dec. 28.

29,863. C. Dellwik. Improvements in the production of water-gas and in apparatus therefor. Complete Specification. Dec. 28.

29,966. J. Y. Johnson.—From The Electric Rectifying and Refining Co. Improvements in and apparatus for producing ozone. Complete Specification. Dec. 29.

29,991. J. H. Dunn. Improvements in the generation of gas by means of electricity from coal, oil, or other carboniferous matter. Dec. 30.

30,037. P. de Resener. An improved apparatus for manufacturing acetylene. Dec. 30.

30,134. J. Schumacher. Improvements in apparatus for use in generating gas. Complete Specification. Dec. 31.

1897.

6. T. J. Meek, H. Davison, and J. Southam. An improvement in ascension pipes used in the manufacture of coal-gas. Jan. 1.

139. F. Trendel and J. Mücke. Process and apparatus for producing acetylene gas. Complete Specification. Jan. 2.

160. W. J. Holme. The lime-light incandescent mantle. Jan. 4.

187. G. Oppenheimer. Improvements in mantles for use in incandescent lighting. Jan. 4.

262. W. M. Ward. *See* Class XVIII. B.

* *See* Note (*) on previous page.

294. O. Kern. Improvements in incandescent gas burners. Jan. 5.

298. G. Hervieu and L. Macke. Improvements in apparatus for generating acetylene and other similar gases. Jan. 5.

328. J. Zimmermann. Improvements in processes of and apparatus for forming and burning acetylene and similar gases. Complete Specification. Jan. 5.

330. D. W. Sugg. Improvements in and relating to incandescent gas burners. Jan. 5.

409. A. A. Humphrey. Improvements in the method of and apparatus for burning liquid fuel. Jan. 6.

501. R. Langhans. Improvements in the manufacture of incandescing media for use in incandescent gas lighting. Jan. 7.

512. G. Lebrun and F. Cornaille. Apparatus for producing acetylene gas. Filed Jan. 7. Date applied for June 17, 1896, being date of application in France.

658. L. Tėlé. Improved apparatus for the production of acetylene. Jan. 9.

885. T. Holliday. Improvements in means or apparatus for use in the production, storing, and application of acetylene gas. Complete Specification. Jan. 12.

911. V. Daix. Improvements in or relating to the production of acetylene gas. Complete Specification. Jan. 13.

955. G. Grossard and E. Peychiers. Apparatus for the production of acetylene gas. Jan. 13.

1153. E. H. F. des Essards. An improved generator for acetylene and certain other gases. Complete Specification. Jan. 15.

1194. H. Simon. Improvement in by-product coke ovens. Jan. 16.

1240. F. E. Ross and A. J. Gibson. Improvement in apparatus for the production of combustible gas from towns' refuse and sewage sludge. Jan. 16.

Complete Specifications Accepted.

1896.

317. G. Love. Apparatus applicable to coke ovens. Jan. 6

682. H. C. B. Forester. Manufacture of artificial fuel. Jan. 20.

841. G. W. Breffit and F. G. Treharne. A new furnace for producing coke, with or without suitable apparatus for collecting the by-products. Jan. 20.

858. A. Devis. Process and apparatus for carburetting air. Jan. 20.

873. W. Raydt. Obtaining carbonic acid from gaseous mixtures. Jan. 20.

1332. C. G. Luis. Apparatus for use in the production of acetylene gas. Jan. 13.

1576. L. Denayrouze. Incandescent lighting with an intimate mixture of gas and air. Dec. 23.

1681. W. H. Wheatley.—From F. Woernle. Manufacture of incandescence bodies for illuminating purposes. Dec. 31.

1682. W. H. Wheatley.—From C. Schmid. Manufacture of incandescence bodies for illuminating purposes. Dec. 31.

3219. E. B. Pym and J. Gore. Apparatus for use in the manufacture of acetylene gas. Jan. 13.

3321. C. M. Schnauder. Manufacture of fuel blocks. Jan. 13.

3681. W. Mackean and the Incandescent Gas Light Company, Ltd. Manufacture of mantles for incandescent gas lighting. Dec. 23.

4000. W. Nicholls. Manufacture of incandescence bodies for illuminating purposes. Dec. 31.

4285. J. R. Wigham. A method of burning acetylene gas or other very rich hydrocarbon gas. Jan. 13.

5916. F. A. MacKinnon. A new or improved method or process for the treatment of coke so as to render it suitable for transport. Jan. 6.

21,098. S. Katz. Manufacture of charcoal fuel blocks. Jan. 20.

22,646. A. G. Smith. Acetylene gas generator, purifier, and holder. Jan. 13.

22,647. A. J. Smith and A. G. Smith. A method of adapting acetylene gas for use on cycles, carriages, or other vehicles. Jan. 20.

23,154. F. G. Bartlett. Burners for incandescent gas lighting. Jan. 13.

23,595. E. Theisen. Purifying gases or vapours and gaining by-products therefrom, and in subjecting gases or vapours to the action of solid or liquid substances, and the converse by means of centrifugal action, and apparatus therefor. Jan. 13.

24,274. R. Turr. Apparatus for the production and combustion of acetylene gas. Jan. 20.

27,085. S. D. Gillet, G. Forest, and J. E. O. Bocande. A new or improved arrangement of apparatus for lighting by acetylene gas. Jan. 13.

IV.—COLOURING MATTERS and DYES.

Applications.

28,557. W. H. Claus and A. Ree. Manufacture of brown colouring matters containing sulphur. Dec. 14.

28,810. W. E. Heyes.—From The Chemical Works, Sandoz. Production of substantive trisazo colouring matters. Complete Specification. Dec. 16.

29,706. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of dyestuffs of the series known as patent blue. Dec. 24.

29,717. G. B. Ellis.—From La Société Chimique des Usines du Rhone, anciennement G. P. Monnet et Cartier. Improvements in the manufacture of chlorine derivatives of toluene. Dec. 24.

29,827. P. A. Newton. — From The Farbenfabriken vormals F. Bayer and Co. Improvements in the production of dyestuffs on fibre. Dec. 28.

29,828. S. Pitt.—From L. Cassella and Co. Production of a brown colouring matter. Dec. 28.

30,015. W. E. Heys. — From The Chemical Works, Sandoz. Production of violet and blue colouring matters of the triphenylmethane group. Complete Specification. Dec. 30.

1897.

304. R. W. James.—From W. J. Matheson and Co., Ltd. Process for the manufacture of aceto-derivatives of the simpler aromatic amines. Complete Specification. Jan. 5.

765. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Improvements in the manufacture of colouring matters and of new intermediate products. Jan. 11.

1145. H. Imray.—From F. Peterson and Co. Manufacture of new colouring matters from benzidin sulpho-acids. Complete Specification. Jan. 15.

Complete Specifications Accepted.

1896.

2946. Levinstein, Ltd., and I. Levinstein. Production of new sulpho-acid and of colours derived therefrom. Jan. 6.

2999. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of sulphonic acids of the meta-amidophenolphthaleines and their alkyl derivatives. Jan. 13.

3966. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of new phthaleine colouring matter. Jan. 6.

5068. T. R. Shillito. — From J. R. Geigy and Co. Manufacture of greenish-blue colouring matters or dyes of the malachite green series. Dec. 31.

5090. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of safranine azo dyestuffs soluble in water. Jan. 13.

5128. O. Imray.—From The Society of Chemical Industry in Basle. Manufacture of new colouring matters dyeing from blue-green to blue, and of new chloro-derivatives and sulphonic derivatives of benzoic aldehyde therefor. Dec. 31.

V.—TEXTILES, COTTON, WOOL, SILK, Etc.

Applications.

28,613. D. M. Sutherland and W. McLaren. Improvements in treating nitro-cellulose for the purpose of coating or impregnating textile and other fabrics. Dec. 11.

28,870. O. Seyfert. A process for giving a silk appearance to cotton. Dec. 16.

29,832. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. *See* Class VI.

1897.

90. H. W. Godfrey. Improvements in the manufacture of floorcloth and the like. Jan. 1.

198. F. Walton. Improvements in apparatus for manufacturing mosaic floorcloth. Complete Specification. Jan. 4.

Complete Specifications Accepted.

1896.

1104. J. L. Bottomley. Manufacture of yarn. Jan. 13.

3517. J. R. Hoyle and H. Colburn. Back-washing machines for use in the treatment of wool or other fibrous substances. Jan. 13.

8235. J. Weiss. A chemical process for the production of white or coloured crape-like designs on cotton, linen, or other fabrics or yarns of vegetable origin. Jan. 20.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

Applications.

28,552. W. Hadfield, J. J. Sumner, and H. Hadfield. Improvements in apparatus for washing and treating woven goods in the piece. Dec. 14.

28,553. W. Hadfield, J. J. Sumner, and H. Hadfield. Improvements in apparatus for use in the continuous bleaching and washing of woven goods in the piece, applicable also in the washing and clearing of printed or dyed goods. Dec. 14.

28,840. A. J. Inston and E. Ramsden. Improvements in or connected with apparatus for the settling and filtration of suds, dye water, sewage, and the like, and obtaining useful products therefrom. Dec. 16.

28,984. J. C. Hamer and J. Haslam. Improvements in or connected with the dyeing of aniline black. Dec. 17.

29,150. F. A. Gatty and Co., Ltd., and V. H. Gatty. Improvements in dyeing wool or other animal fibres in either the raw or manufactured state. Dec. 19.

29,154. T. Russell. Improvements in and relating to the printing of calico and like wet fabrics. Dec. 19.

29,645. W. Hadfield, J. J. Sumner, and H. Hadfield. Improvements in apparatus for use in the continuous bleaching and washing of woven goods in the piece, applicable also in the washing and clearing of printed or dyed goods. Dec. 24.

29,832. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Production of local durable silk-like gloss-effects on cotton or linen stuffs by means of printing. Dec. 28.

Complete Specifications Accepted.

1896.

1407. J. Y. Johnson.—From E. G. May, Söhne, and Co. Improvements in the production of coloured figures or designs and ornamental effects. Dec. 9.

2840. T. Halliwell and J. Stones. Apparatus for dyeing, bleaching, or scouring fibrous materials. Jan. 13.

5712. J. Barnes. The production of fast colours on animal fibre by the aid of oxide of titanium. Jan. 20.

26,495. A. Vogelsang. Process for treating dry materials (more particularly fibrous substances) with liquid. Dec. 31.

VII.—ACIDS, ALKALIS, and SALTS.

Applications.

28,891. G. Kynoch and Co., Ltd., and A. T. Cocking. Improvements in purifying and concentrating sulphuric acid, in part applicable to the manufacture of nitric acid. Dec. 16.

29,066. E. J. Hunt and E. F. Watson. Improvements in the manufacture of chlorine. Dec. 18.

29,455. H. H. Lake.—From C. Pieper. A method of and apparatus for the continuous manufacture of ammonia from nitrogenous materials. Dec. 22.

29,884. T. G. Webb. *See* Class I.

29,922. J. D. Gilmour. Method of drying alkaline cyanides. Dec. 29.

1897.

28. E. J. Ball. Improvements in the treatment of substances containing potassium. Jan. 1.

204. G. Cipri. Improvements in the manufacture of sulphuric acid. Jan. 4.

381. A. K. McCosh. *See* Class X.

422. A. James. Improvements in the treatment of seaweed and the like for the production of iodine and other useful products. Jan. 6.

591. P. J. Worsley and The United Alkali Co., Ltd. Improvements in the manufacture of caustic soda or caustic potash from carbonate of soda or carbonate of potash. Jan. 8.

1248. H. H. Lake.—From J. S. Levi and Co. An improved process for obtaining barium carbonate from native sulphate of barium. Jan. 16.

Complete Specifications Accepted.

1896.

928. J. Foster. Improvements in and relating to the discharging of salts and the like from evaporators. Dec. 23.

3869. J. T. Conroy, F. Hurter, and J. Brock. Manufacture of ferrocyanides from sulphocyanides. Dec. 23.

4743. L. G. Paul. Manufacture of nitrites of soda and potash. Jan. 6.

25,100. A. Schmidt. Process for purifying crude acetic acid. Jan. 20.

VIII.—GLASS, POTTERY, and ENAMELS.

Applications.

28,789. C. F. Ravenhill. Improved glaze for clay tobacco pipes. Dec. 16.

29,315. J. P. Guy. Improvements in or relating to machines for pressing and forming saggars or other earthenware articles. Dec. 22.

29,316. J. P. Guy. Improvements in hollow earthenware vessels, such as potters' saggars. Dec. 21.

29,386. R. F. Copeland. Improved construction of ovens for use in firing pottery-ware. Dec. 22.

29,589. F. Joynson, W. Harrison, and G. T. Joynson. Improved machine for the manufacture of glass bottles, jars, and the like. Complete Specification. Dec. 23.

29,844. P. Rataud. Process for the decoration of china, earthenware, glass, and the like by means of chromotypography and photochromy. Dec. 28.

1897.

334. G. G. M. Hardingham.—From G. W. Blair. An improvement in the manufacture of glass articles. Complete Specification. Jan. 5.

Complete Specification Accepted.

1896.

1834. J. Jacques and W. E. McCalla. A new or improved manufacture of articles in glass, tile, pottery, metals, and like substances by a process for producing thereon letters, figures, or designs in permanent colours. Jan. 13.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Applications.

29,109. W. H. Coward. Improvements in the manufacture of composition and other compressed blocks for paving. Dec. 18.

29,212. Petrifite, Ltd.—From J. Steiger. An improvement in the manufacture of cement. Dec. 19.

29,651. O. Kraner. Process for the production of a material for making floors, plasterings, ceilings, walls, roofs, and the like without joints or chinks. Complete Specification. Dec. 24.

28,864. A. Zboril and A. Schenk. Improved plates, slabs, or the like for floors, pavements, fireplaces, and similar purposes, and method of manufacturing the same. Dec. 28.

30,117. A. Gutensohn. Improvement in the manufacture of fire-proof materials. Dec. 31.

1897.

322. A. Calladine. A new or improved cement. Jan. 5.

Complete Specifications Accepted

1895.

20,770. W. Busche. Brick-burning and other kilns. Jan. 13.

1896.

2223. F. Turner. Manufacture of cement or artificial stone. Jan. 6.

3993. W. F. Williams. Artificial stone for building purposes. Jan. 20.

23,105. J. A. Just. Manufacture of compositions for paving and like purposes. Jan. 13.

24,242. A. Clery. Manufacture of artificial stone. Jan. 6.

24,902. A. Gries. Production of slabs or such like for building and other purposes. Dec. 23.

X.—METALLURGY, MINING, Etc.

Applications.

28,714. J. B. Torres. Improvements in or relating to the extraction of gold, silver, and other metals from ores and the like. Dec. 15.

28,727. P. E. Placet. Improvements in the purification of aluminium and apparatus therefor. Dec. 15. Date applied for July 24, 1896, being date of application in France.

28,836. J. W. Spencer. Improvements in the manufacture of steel. Dec. 16.

28,880. C. de E. Llatas. Improvements in the manufacture of steel. Dec. 16.

29,080. J. W. Spencer. Improvements in the manufacture of steel in regenerative furnaces and the apparatus connected therewith. Dec. 18.

29,090. T. T. Sill. Improvements in the manufacture of red oxide from the waste liquors from galvanising works, tin-plate works, wet copper process, or other residual liquors containing iron in the form of chloride. Dec. 18.

29,251. R. D. Burnie and W. T. Lougher. An improved process for covering or coating steel and iron with copper or alloy of copper. Dec. 21.

29,284. J. B. Torres. Improvements in extracting gold, silver, and other metals from ores and the like, and in the apparatus therefor. Dec. 21.

29,317. P. E. Serrant, A. E. Ragetly, and F. G. H. Marvelle. An improved process of extracting gold from auriferous ores and tailings or residues. Dec. 21.

29,388. C. Wilcox and B. J. Hammersley. Certain improvements in annealing muffles for annealing cold-drawn steel and other metal tubes, metal sheets, and other metal articles. Dec. 22.

29,709. W. Kirkham. Improvements in crucible melting furnaces. Dec. 24.

29,795. C. W. Robinson. A new tool steel and means for producing the same. Dec. 28.

29,833. J. B. Clive. A new or improved appliance or apparatus to be used in the casting of metals and metallic alloys. Dec. 28.

29,846. S. Danner. Improvements in the manufacture of crucible steel. Dec. 28.

30,022. H. R. Angel. Improvements in the reduction of zinc blend and other ores. Dec. 30.

30,077. J. Burgess and W. A. Brodie. Improvements for use in producing castings in malleable iron, mild steel and other metals. Dec. 31.

30,141. A. F. Cothias. An improved process of casting for reducing the density of cast articles. Complete Specification. Dec. 31.

30,152. A. M. Clark.—From R. H. T. Biewend and The Actien Gesellschaft für Zinc Industrie vormals W. Grillo. Process of recovering zinc from zinciferous gases in the distillation and smelting of zinc. Dec. 31.

1896.

91. J. D. Grey and T. Gwynne. Improvements in the treatment of sheet iron and steel in the manufacture of polished sheets. Complete Specification. Jan. 1.

290. W. E. May. Improvements relating to the casting of metals and to apparatus for use in connection therewith. Jan. 5.

292. H. R. Angel. Improvements in the reduction of zinc blend refractory ores, and recovery of the lead and precious metals therefrom. Jan. 5.

381. A. K. McCosh. Improvements in obtaining cyanides and other products from blast furnaces. Jan. 6.

513. W. S. Simpson. Improved means for effecting close or different texture of metallic particles or molecules during the process of casting. Jan. 7.

516. A. S. F. Robinson. Improved method and machinery or apparatus for extracting gold or other valuable metal from crushed ore or material containing it. Jan. 7.

531. S. Shaw. Improvements in the recovery of zinc or spelter from alloys or waste materials containing metallic zinc. Jan. 8.

560. S. Shaw. Improvements in the treatment of argentiferous lead. Jan. 8.

662. H. R. Angel. Improvements in the reduction of zinc blend and other ores and recovery of the lead and other metals therefrom. Jan. 9.

880. A. Etard. Improvement in the treatment of ores or other matters containing gold, silver, or copper, for the obtainment of the metal therefrom. Jan. 12.

918. G. A. Webb and W. A. Thoms. An improvement in alloys. Jan. 13.

990. G. F. Redfern.—From J. L. Gauharou. Improvements in the treatment of metals and alloys. Jan. 13.

1034. H. R. Angel. Improvements in the reduction of zinc blend and other ores, and recovery of the lead with other metals therefrom. Jan. 14.

1062. C. T. J. Vautin. An improved process for the reduction of argentiferous or non-argentiferous complex sulphide ores of lead and zinc. Jan. 14.

Complete Specifications Accepted.

1895.

23,152. E. J. Post. Process and apparatus for plating articles or covering the same with metal. Jan. 13.

24,703. R. Heathfield and W. S. Rawson. An improvement in galvanising. Dec. 23.

1896.

203. E. Placet. Treatment of iron and steel. Dec. 31.

303. C. C. Longridge and G. T. Holloway. Improvements in plant and process for smelting auriferous antimony ores. Jan. 6.

1055. A. J. Boult.—From C. Prioux and C. Hugot. Soldering metals. Jan. 20.

1316. A. J. Boult.—From C. J. L. Otto. Manufacture of iron and steel. Jan. 13.

1970. R. B. Boulton. The application of inlaying of metal or alloy or amalgams, when in a softened condition, in or upon any hard substance devoid of life, and which has been formed into any pattern, lines, letters, or designs. Jan. 20.

2327. T. M. Ash and H. N. Weldon. An improved process for plating or coating non-metallic articles with metals. Jan. 20.

18,276. H. H. Lake.—From H. Schweitzer and E. E. Lungwitz. Production of steel and the refining of iron ore. Dec. 31.

23,954. J. Y. Johnson.—From J. J. Deeble. Apparatus for use in the extraction of gold from auriferous material by the aid of chemical solvents. Dec. 31.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

Applications.

28,723. J. Schuil. Improvement in secondary batteries for electricity. Dec. 15.

28,764. F. Hurter and the United Alkali Co., Ltd. Improvements in apparatus for use in electrolysis, more especially intended for use in the electrolysis of zinc chloride. Dec. 15.

29,115. F. King. Improvements in electric storage or secondary batteries. Dec. 18.

29,306. A. Heil. Improvements in secondary batteries. Complete Specification. Dec. 21.

29,307. A. Heil. Improvements in peroxide of manganese and carbon electrodes, and in the method of producing the same. Complete Specification. Dec. 21.

29,362. W. H. Akester and M. Miroy. An improvement in accumulators. Dec. 22.

29,363. W. H. Akester and M. Miroy. Improvements in or relating to accumulators. Dec. 22.

29,574. C. D. Abel.—From C. N. Seidneff. A new galvanic element and exciting and depolarising liquids therefor. Complete Specification. Dec. 23.

29,907. C. Brault. Improvements in the manufacture and production of elements or plates for electrical storage batteries. Complete Specification. Dec. 29.

29,981. A. W. Turner and A. Hiorns, jun. Improvements in and relating to electric accumulators. Dec. 30.

30,024. F. Wüste. Improved composition of filling material for electrolytical and galvanic purposes. Dec. 30.

1897.

251. G. W. Harris and R. J. Holland. Improvements in secondary batteries and process of forming same. Complete Specification. Jan. 5.

423. D. G. Fitzgerald. Improvements in primary and secondary voltaic batteries. Jan. 6.

425. F. King. Improvements in or connected with secondary batteries. Jan. 6.

490. W. Wright. Improvements relating to the electric deposition of metal on pipes, tubes, and other articles. Jan. 7.

498. A. Koch. Improvements in apparatus for the electrolytic treatment of alkaline salts. Jan. 7.

615. E. Bohn. Improvements in and applicable to means for electrically obtaining ozone, and employing the same for curative and other purposes. Jan. 9.

631. W. P. Freeman. Improvements in primary batteries. Complete Specification. Jan. 9.

652. D. W. B. E. Thomson and H. C. Bull. Improvements in or connected with primary electric batteries. Jan. 9.

859. Aschermann. Process for the extraction of metallic alloys and pure metals by electric heat. Complete Specification. Jan. 12.

995. W. S. Doe. Improvements in galvanic batteries. Jan. 13.

996. W. Majert. Improvements in and relating to accumulator plates. Jan. 13.

1026. J. E. Hartley.—From F. W. Zingsen. Improvements in apparatus for the electro-deposition of metals. Jan. 14.

1038. W. H. Graham. Process and apparatus for electro-thermally treating materials. Jan. 14.

1099. W. Rowbotham. Improvements in and relating to accumulators or storage batteries. Jan. 15.

1203. H. W. Hancock and A. H. Dykes. An improved secondary battery plate. Jan. 16.

1204. H. W. Hancock and A. H. Dykes. An improved method of preparing positive plates for secondary batteries. Jan. 16.

Complete Specifications Accepted.

1895.

25,002. R. Heathfield and W. S. Rawson. Electrolytical apparatus. Dec. 31.

1896.

535. H. W. Headland. Primary and secondary batteries. Jan. 6.

778. H. W. Headland. Secondary batteries. Jan. 13.

842. S. O. Cowper-Coles. Manufacture of electric conductors. Dec. 31.

1069. C. Pollak. Electrical condensers. Jan. 20.

1228. D. M. Hawes. Manufacture of battery cells or vessels to contain acids or other chemicals. Jan. 20.

1669. W. Turner. Electric batteries. Jan. 13.

13,520. C. Pollak. Plates for accumulators or secondary batteries. Jan. 20.

16,268. W. Rowbotham. Electric batteries. Jan. 13.

26,281. K. Klic. Manufucture of metal sheets or strips by electro-deposition. Jan. 6.

XII.—FATS, OILS, AND SOAP.

Applications.

28,682. J. L. Garle and C. C. Frye. A new or improved process for the decolorisation of palm oil. Dec. 15.

28,781. H. A. Wild. An improved laundry-cream composition. Dec. 16.

1897.

112. Read, Holliday, and Sons, Ltd., and T. Holliday. Improvements in the manufacture of soap. Jan. 2.

123. G. Rezek. An improved washing material and process for making the same. Complete Specification. Jan. 2.

751. J. T. Potts. Improvements in the manufacture of soap. Jan. 11.

1067. J. Mendess. Improvements in compounds for cleansing and dyeing purposes. Jan. 14.

1208. W. P. Thompson.—From J. Van de Bücken and S. Gattfeld. Improved soap for carpets. Complete Specification. Jan. 16.

Complete Specifications Accepted.

1896.

3329. G. D. Macdougald and The London and Dundee Syndicate, Ltd. Improved means and apparatus connected with and for the marking of soap tablets, cakes, or bars with indelible colour. Jan. 6.

24,909. G. W. Hansen. Manufacture of waterproof dubbing for leather and leather goods. Dec. 23.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

28,598. L. Greening. Improvements in the manufacture of material suitable for use as a substitute for india-rubber, gutta-percha, ivory, horn, vulcanite, and the like; also for waterproofing paper, leather, and other fabrics; and as a varnish. Dec. 14.

28,698. H. Sutton. Imitation stained glass enamel. Dec. 15.

28,950. F. Beindorff. A process for making colours for water-colour painting. Complete Specification. Dec. 17.

29,805. F. Ellershausen. Improved method of preparing rubber substitute. Dec. 28.

30,017. P. A. Duprez. Process and apparatus for devulcanising or desulphurising vulcanised caoutchouc waste. Dec. 30.

1897.

1049. P. H. King, V. M. H. Beever, and C. T. Sidgwick. Manufacture of synthetic caoutchouc and analogous compounds. Jan. 14.

Complete Specifications Accepted.

1896.

2568. J. L. Pollock. Improvements in air- and waterproofing varnishes or compositions. Dec. 23.

3906. W. Hampe and C. Schnabel. Manufacture of zinc oxide. Jan. 6.

26,015. A. Schmidt. Process for obtaining turpentine from wood and wood waste. Jan. 13.

28,283. W. A. Hall. Water-paints. Jan. 20.

XIV.—TANNING, LEATHER, GLUE, and SIZE.

Applications.

29,885. W. E. Heys.—From G. Wendler. Improvements in and relating to farinaceous and resinous solutions and mixtures for sizing and finishing textile fabrics, paper, and the like. Dec. 29.

1897.

655. E. S. Cook. Improved method of preparing raw hides and the manufacture of travelling trunks, portmanteaus, and the like therefrom. Jan. 9.

Complete Specifications Accepted.

1896.

1587. M. Hocquet. The manufacture of new compounds or composition from cork and other materials for use as substitutes for wood, leather, and other substances, or for other purposes to which same may be applicable. Jan. 20.

2572. H. L. J. Roy. Process and apparatus for the rapid tanning of hides, applicable also for dyeing and otherwise treating leather. Dec. 31.

16,764. J. Blunck. Process of tanning hides, skins, or the like. Jan. 6.

XVI.—SUGARS, STARCHES, GUMS, Etc.

Applications.

29,319. J. Brown and W. J. Brown. Improvement in the manufacture of artificial gum. Dec. 21.

1897.

24. E. L. Newsome. Improved appliance for supplying glucose, syrup, or other material to preserving pans when boiling. Jan. 1.

586. R. Ansorge. An improved starch preparation and process of making the same. Complete Specification. Jan. 8.

742. H. W. Hellon and R. G. Whitford. Improvements in vegetable syrups. Jan. 11.

1141. A. Classen. Manufacture of chemical compounds of starch and dextrine or equivalent bodies with formaldehyde or its equivalents. Jan. 15.

1155. A. Collette, jun., and A. Boidin. A process for utilising moulds or microscopic fungi for extracting from residues, particularly those arising from the treatment of amylaceous and sugary substances, alcohol, and mucidines capable of being used in panification, distillation, saccharification, and the like. Jan. 15.

Complete Specifications Accepted.

1896.

1960. E. Shaw. Boiling sugar and other syrups. Jan. 20.

4560. D. N. Bertram. Strainers for treating paper pulp, separating fibrous materials from starch, and like purposes. Jan. 13.

6508. W. Feld. Process for the extraction of sugar from molasses and other saccharine liquors by means of barium hydroxysulphide, and the regeneration of the latter from the by-products produced. Jan. 13.

24,038. F. W. Golby.—From J. Hensel. See Class XVIII. A.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

28,713. C. Killing. Process and apparatus for purifying crude alcohol and other alcoholic mixtures. Complete Specification. Dec. 15.

28,763. J. Y. Johnson.—From The Electric Rectifying and Refining Co. Improvements in and apparatus for purifying and decolorisng saccharine solutions and other liquids. Complete Specification. Dec. 15.

29,965. J. Y. Johnson.—From The Electric Rectifying and Refining Co. Improvements in and apparatus for treating or purifying, rectifying, ageing, deodorising, and decolorising alcoholic and other liquids or liquified matter. Complete Specification. Dec. 29.

1897.

275. J. Jaquemin, W. A. Owston, and G. H. Newman. An improvement in the production of pure alcohol by the fermentation and distillation of dried fruit. Jan. 5.

284. J. W. L. Hepton, A. E. L. Hepton, and T. Clarke. Improvements in unions applicable for spargers and other purposes. Jan. 5.

978. H. Muller-Thurgau. Process for the production of non-alcoholic or feebly alcoholic fruit juices (fruit and grape wines) which may be kept or preserved without change. Jan. 13.

1162. F. C. Haeck. A new or improved process and apparatus for rapidly and economically ageing or maturing alcoholic beverages. Complete Specification. Jan. 15.

Complete Specification Accepted.

1896.

24,038. F. W. Goldby.—From J. Hensel. Process for the production of a food of a high nutritive value from the molasses of raw beet-sugar. Jan. 20.

XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

Applications.

A.—Foods.

28,708. A. J. C. Rayson. The manufacture of a substitute for coffee. Complete Specification. Dec. 15.

1897.

899. J. J. Hazard. Improvements in the preservation of organic matters and in apparatus for use in connection therewith. Jan. 12.

B.—Sanitation.

28,840. A. J. Inston and E. Ramsden. *See* Class VI.

28,918. A. P. I. Cotterell. An appliance for facilitating the deposition or precipitation of sewage in sewage tanks, applicable also to clarifying water and other liquids. Dec. 17.

29,009. J. F. G. de Bonardi (Marquis du Menil). Method and furnace for the incineration of so-called garbage and the recovery of ammonia therefrom. Dec. 17.

29,223. I. S. McDougall. Improvements in the methods of, and apparatus for water purification. Complete Specification. Dec. 19.

1897.

107. W. Bagshaw. Improvements in filters and clarifiers for the treatment and purification of water, sewage, waste liquids, and the like. Jan. 2.

262. W. M. Ward. Improvements in the mode, means, and apparatus for treating smoke for the extraction of the carbon and other residual products resulting from imperfect combustion. Jan. 5.

654. W. M. Ducat. Improvement in the method of, and apparatus for purifying water sewage and other foul liquids. Jan. 9.

C.—Disinfectants.

29,288. W. B. Bottomley. Improvements in and relating to material for use in the treatment and purification of sewage and other impure waters. Dec. 21.

29,484. J. Startin. Improvements in or relating to disinfectants, antiseptics, and the like. Dec. 22.

29,591. J. Hardwick. Improvements in deodorising, disinfecting, and antiseptic compounds. Dec. 23.

COMPLETE SPECIFICATIONS ACCEPTED.

A.—Foods.

1896.

3260. T. Hyatt. Alimentary substances, their manufacture, and putting up or conditioning them for domestic use. Jan. 6.

3833. J. Dunn and W. Dunn. Treating lentils, peas, and other leguminous vegetables to obtain new alimentary products. Dec. 31.

26,498. W. F. E. Casse. Casse freezing process for preserving milk and cream. Jan. 13.

26,635. P. M. Justice.—From M. W. Marsden. An improved product obtained from corn or maize stalks, and a process of, and apparatus for making the same. Jan. 20.

B.—Sanitation.

821. J. H. Williams. Apparatus and process for purication, softening, or hardening of water for domestic, manufacturing, or other purposes. Dec. 23.

1038. R. E. von Lengerke. Filtration of sewage. Jan. 20.

XIX.—PAPER, PASTEBOARD, Etc.

APPLICATIONS.

29,166. C. Deselle. A process for the manufacture of artificial ivory articles. Dec. 22.

29,829. C. D. Abel.—From Farbwerke Friedrichsfeld, P. Remy. Manufacture of waterproof paper and similar fabrics, and the production of compounds therefor. Dec. 28.

COMPLETE SPECIFICATIONS ACCEPTED.

1895.

23,598. C. Brodbeck. Manufacture of fabrics from chemical cellulose. Jan. 20.

1896.

4560. D. N. Bertram. See Class XVI.

4713. C. F. Cross. Manufacture of alkali cellulose. Jan. 13.

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29,322. J. Rosenberg. An improved process of producing tri-bromsalol. Dec. 21.

29,446. O. Imray.—From F. Hoffmann-La Roche and Co. Production of certain organic therapeutic preparations. Dec. 22.

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445. R. Mackill. Improvements in obtaining nicotine from tobacco. Jan. 7.

656. J. Y. Johnson.—From C. F. Boehringer and Soehne. Improvements in the preparation of alkylated uric acids. Jan. 9.

1075. A. J. Boult. From F. Fritzsche and Co. Improved process for production of iso-eugenol from oil of cloves, with the simultaneous extraction of terpene. Jan. 14.

1,164. W. L. Wise.—From The Chemische Fab. von Heyden Gesellschaft mit beschrankter Haftung. Improvements in the manufacture of *o*-sulphamin benzoic acid alkylesters and of anhydro-*o*-sulphamin benzoic acid. Jan. 15.

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NOTICES.

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In reply to numerous inquiries, the compilation of the Collective Index is in active progress, part of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

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Button, Herbert, c/o Solvay Process Co., Syracuse, N.Y., U.S.A., Chemist.

Cunliffe, Albert J., Kern Mill Printworks, Whittle-le-Woods, Lancashire, Calico Printer.

Davies, Saml. H., c/o H. S. Rowntree and Co., Cocoa Works, York, Research Chemist.

Felix, Dr. Lorenz, 23, Arpley Street, Warrington, Technical Chemist.

Garfield, Jos., 163, Penn Road, Wolverhampton, Civil Engineer.

Graesser, Franz A., The Wrexham Lager Beer Co., Wapping Station, Liverpool, Manager.

Hewitt, T. Lacey, Beechfield, St. John's, Higher Broughton, Manchester, Analytical Chemist.

Hodgson, Matthew, 2, Leitrim Place, Wicklow, Ireland, Technical Chemist.

Jacqué, Maurice, Fabrica de Dinamita, Galdacano, cerca Bilbao, Spain, Chemical Engineer.

Kirkby, Wm., 14, Ducie Avenue, Oxford Road, Manchester, Chemist.

Le Bosquet, Maurice, 328, East Eagle Street, East Boston, Mass., U.S.A., Technical Chemist.

MacDonald, G. W., Fulwich Farm, Dartford, Kent, Explosives Chemist.

Maddox, Arnold, 184, Malpas Road, Brockley, S.E., Chemical Lecturer.

Meslans, Prof. M., 12, Rue de la Grande Ile, Meaux (Seine et Marne), France, Professor of Chemistry.

Millard, Edgar J., 40—42, Charlotte Street, London, E.C., Chemist and Manager.

Merton, Jno., 50, North Road, St. Helens, Lancashire, Analytical Chemist.

Murch, D. Wilshin, Church Rd., Lostock Gralam, Cheshire, Chemist.

Nairn, Michael, Kirkcaldy, N.B., Linoleum Manufacturer.

Nicholson, Wm. J., 27, Dundonald Road, Kelvinside, Glasgow, Chemist.

Palmer, J. Howard, Apartado 48, Hermosillo, Sonora, Mexico, Assistant Manager, Gold and Silver Recovery Company.

Poyser, Jos. C., Woodlands, The Priory, Higher Broughton, Manchester, Manufacturing Chemist.

Quirk, Jno. S., Lead Smelting Works, St. Helens, Lancashire, Manager.

Reid, Jno., Garnockside, Glengarnock, Ayrshire, Metallurgical Chemist.

Stein, Sigmund, 323, Vauxhall Road, Liverpool, Sugar Refinery Manager.

Stones, Wm., Northwood, Seymour Grove, Old Trafford, Manchester, Chemist.

Walker, Jas. W., Messrs. Alex. Walker and Co., Irvine, N.B., Chemical Manufacturer.

Wielandt, Dr. Wm., Alleestrasse, Gelsenkirchen, Westfalia, Germany, Chemist.

Wilson, Ellwood, 101, Harrison Street, East Orange, N.J., U.S.A., Chemist.

Zacharias, Dr. P. D., Philhellinon Street 22, Athens, Greece, Industrial Chemist.

Zahorski, Dr. Boleslas, 22, Oxford Street, Liverpool, Technical Chemist.

CHANGES OF ADDRESS.

Adriance, J. S., l/o E. 34th Street; 321, Lexington Avenue, New York, U.S.A.

Appleton, H. A., l/o Leeds; 33, Upper Rushton Road, Thornbury, Bradford, Yorks.

Barraclough, W. H., l/o Chapeltown; Mortomley, near Sheffield.

Boot, J. C., l/o Brooklyn; American Sugar Refining Co., Jersey City, N.J., U.S.A.

Brothers, Wm., l/o Bury; Beechwood, Prestwich, near Manchester.

Buchanan, Josh., l/o Buffelsdoom; c/o New Transvaal Chemical Co., Ltd., Box 1829, Johannesburg, S.A.R.

"Chemical News," l/o Boy Court; 6 and 7, Creed Lane, Ludgate Hill, E.C.

Cowburn, W. H., l/o Ashley Road; Fernroyd, St. Margaret's Road, Bowdon.

Duttson, W. H., l/o Brockley; Southwood, Silverdale, Sydenham, S.E.

Ede, H. E., l/o Sheffield; c/o New Explosives Co., Ltd., Stowmarket, Suffolk.

Gordon, J. G., l/o Cannon Street; Queen Anne's Mansions, Westminster, S.W.

Haller, H. Loft, l/o Hull; 109, Tennyson Place, Bradford, Yorks.

Hays, Jos. A., l/o New York; 147, South 18th Street, Pittsburg, Pa., U.S.A.

Hodges, H. B., l/o Washington; Long Island R. R. Co., Long Island City, N.Y., U.S.A.

Hunt, Bertram, l/o Denver; 23, Stevenson Street, San Francisco, Cal., U.S.A.

Hurry, E. H., l/o New York; Bethlehem, Pa., U.S.A.

Kawakita, M., l/o Berlin; 14, Richmond Terrace, Clapham, S.W.

King, J. Falconer, l/o Russell Place; 43, Stirling Road, Edinburgh.

Lee, T. H.; Journals to Hampden House, Phœnix Street, N.W.

Lishman, G. P., l/o Success; Bunker Hill, Fence Houses, Co. Durham.

Lovejoy, F. W., l/o Cambridgeport; 121, Lake Avenue, Rochester, N.Y., U.S.A.

Lowe, Jas. S., l/o Belle Rive Estate; Beauchamp Estate, Grand Rivière S.E., Mauritius.

McCann, Owen, l/o 62; 80, Studley Road, Forest Gate, Essex.

McGhie, J. Burns; Journals to c/o Quirk, Barton, and Co., Normandy Wharf, Rotherhithe, S.E.

Maxwell, T., l/o Braeside Avenue; 2, Wardlaw Avenue, Rutherglen, Glasgow.

Ogata, S., l/o Imperial Mint; Zohei Shikyoku, Okurasho, Tokio, Japan.

Playfair, D. J., l/o Woodside Terrace; Tramillan, Gowan, Glasgow.

Quinn, J. C.; Journals to Rubber Tyre Manufacturing Co., Ltd., Aston Cross, Birmingham.

Reid, Walter C., l/o Tynemouth; 12, Wentworth Place, Newcastle-on-Tyne.

Roscow, J. F., l o Lowell; Norwich Bleachery, Norwich, Conn., U.S.A.
Rosell, Dr. C. A. O., l o East 17th Street; 42, West 26th Street, New York City, U.S.A.
Schiller, L. J., l o Boston; c/o Arbuckle Bros., 111, Front Street, New York, U.S.A.
Smith, Francis P., l o New York; 77, Woodland Avenue, New Rochelle, N.Y., U.S.A.
Smith, Harry, l o St. Thomas' Terrace; 30, Falconar Street, Newcastle-on-Tyne.
Sowter, Alf.; Journals to Assay Office, Sylvester Street, Coolgardie, W. Australia.
Tinsley, J. D., l o Virginia; College of Agriculture, Mesilla Park, N. Mexico, U.S.A.
Wetter, J., l o Strand; 39, Essex Steeet, Strand, W.C.

MEMBERS OMITTED FROM LIST.

Eastwood, Edw., Tunnel Soap Works, Wapping, E., Soapmaker.
Hills, Henry, Chemical Works, Deptford, S.E., Chemical Manufacturer.
Small, Evan W., Sunny Side, West Park, Newport, Mon., Director of Technical Instruction.
White, G., Rantoul, Exeter, New Hampshire, U.S.A., Chemist.

Deaths.

Demuth, Dr. L., Wharfedale, Edgbaston, Birmingham.
Fulton, Robt., jun.; accidentally killed at Ardeer Works, Stevenston, N.B.
Martyn, Wm., Afton House, Hebburn-on-Tyne. Feb. 12.
Tyler, Arthur J.; accidentally drowned at Marburg, Germany.

London Section.

THE CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE, W.

Chairman: R. Messel.

Vice-Chairman: Boverton Redwood.

Committee:

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W. J. Dibdin.	S. Rideal.
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S. Hall.	F. Napier Sutton.
D. Howard.	Wm. Thorp.
E. Grant Hooper.	T. Tyrer.

Hon. Local Secretary:
John Heron, 110, Fenchurch Street, E.C.

SESSION 1896–97.

March 1st:—
(1.) Mr. J. W. Lovibond. "Relation of Colour to Quality in Malt."
(2.) Mr. J. H. B. Jenkins. "Hehner's Bromine Tests for Oils."
(3.) Mr. J. H. Coste. "Note on the Analysis of Superphosphates."

N.B.—The Election of Officers and Five Members to the Local Committee will take place at the Meeting on April 5th. Nominations must be signed by Ten Members and sent to the Hon. Local Secretary not later than March 22nd. Members can only sign one Nomination. (See Bye Laws of Section.)

Meeting held on Monday, January 18th, 1897.

DR. R. MESSEL IN THE CHAIR.

ON THE BROMINE ABSORPTION OF FATS AND OILS, GRAVIMETRICALLY AND THERMOMETRICALLY.

BY OTTO HEHNER.

In the Journal of this Society for December 1896, p. 859, Dr. Lewkowitsch publishes a short paper on the gravimetric determination of the bromine value of fats, and therein attempts to disprove the accuracy of certain results and observations on this subject contained in papers by myself (Analyst, March 1896, p. 49), and by myself and Mitchell (Analyst, July 1895, p. 146).

On adding bromine to unsaturated fatty compounds, suitably diluted with a solvent like chloroform or carbon tetrachloride, the bromine immediately enters into additive combination with the fat, under more or less considerable evolution of heat. Little or no substitution of hydrogen, as a rule, takes place, at least not under the conditions laid down in the first of the two papers above referred to, in which it was sought to ascertain whether the weight of the bromo-addition compound formed stood in any definite relation to the iodine number as determined by Hübl's method, while in the second paper, by myself and C. A. Mitchell, relations are looked for, and conclusively shown to exist, between the iodine number and the heat evolved on bromination.

It was found that in a number of fats the percentage of bromine fixed by them was in molecular proportion to the Hübl number, for on calculating the bromine into its equivalent of iodine, figures closely approaching the Hübl numbers were obtained, as is seen from the following figures:—

Kind of Oil.	Hübl Number.	Bromine Addition multiplied by 1·587.
Olive oil	80·3	81·5
"	80·2	79·9
"	80·6	80·7
Lard	65·7	64·4
"	65·7	64·6
"	63·2	64·1
"	60·1	61·4
Maize oil	122·0	123·2
Butter-fat	34·0	34·3
Tallow-fatty acids	48·1	47·8

In the case of castor oil and of boiled linseed oil no close agreement could be observed. A sample of the former, with a Hübl number of 83·0 showed only 69·5 gravimetrically, and one of boiled linseed oil (Hübl 132·5) gave 159·5.

Dr. Lewkowitsch's numbers, as given on page 859 of the Journal do not agree with the above. I do not propose to examine them all in detail, but I will show, in the case of linseed oil, that Dr. Lewkowitsch is wrong.

Linseed oil has an iodine absorption of from 154 to 190, the great variation being largely due to modifications in the method of determination adopted by different workers. Unless the excess of iodine used is very large and the time of its action very prolonged, the iodine number is found too low.

Dr. Lewkowitsch finds two samples of linseed oil to combine with 18·68 and 10·4 per cent. of bromine, gravimetrically determined, corresponding to an iodine absorption of 29·65 and 16·5 respectively.

Now, by adding to linseed oil, or to the fatty acids obtained from linseed oil, dissolved in a suitable solvent, bromine in excess, a violent reaction ensues, with great evolution of heat. A little hydrobromic acid is formed, showing that some hydrogen of the fatty acid has been substituted, and at the same time an insoluble bromo-compound, found by analysis to be the hexabromo-compound of linolenic acid, separates. From the mother liquor of this large amounts of tetrabromo-linolic acid can readily be obtained. I have, together with C. A. Mitchell, made many determinations of the insoluble hexabromo-compound obtained, and find that the weight of this alone invariably corresponds to more than the total bromine absorption found by Lewkowitsch, not to speak of the other bromo-compounds at the same time abundantly formed. Apart from this the strongest corroborative evidence exists, partly contained in a paper published by Rowland Williams (Analyst, December 1895, p. 277), who, as chemist to a very large firm of linoleum manufacturers, is in an excep-

tionally good position to investigate the subject. Williams's published results are as follow :

Kind of Oil.	Hubl Number.	Bromine gravimetrically determined.	Iodine corresponding to Bromine.
Raw linseed oil	183·2	114·2	181·2
" "	192·9	120·7	191·5
" "	185·2	115·1	182·7
" "	195·5	119·2	189·2
" "	194·8	119·6	189·8
" "	195·1	119·4	189·5
Boiled linseed oil, thin.......	175·1	111·3	176·6
" " "	163·0	112·4	178·4
" " stout......	99·5	65·6	104·1
" " very stout.	95·9	59·9	95·1

That is to say, R. Williams finds with me 10 times more bromine absorbed and combined with linseed oil than does Lewkowitsch. Williams states: "Having convinced myself of the accuracy of the gravimetric method, especially as regards raw linseed oil. I can confidently recommend it as being in many cases more convenient and more reliable than the volumetric iodine estimation." Since December 1895, Williams has continued to work on the subject and has embodied his results in a letter which he has authorised me to bring before the meeting. I quote now from his letter: "A large proportion of my time during the last five years has been occupied in the analysis of linseed oil, both in its raw state and also boiled; my opportunities in this direction having been, as you are no doubt aware, quite exceptional. My remarks in this communication are intended to refer entirely to raw linseed oil.

"During the last 18 months or so I have made about 200 gravimetric determinations of the bromine absorption of linseed oil, using essentially the method described by you in 'The Analyst,' Vol. XX., p. 50. With rare exceptions the figures obtained have been between 110 and 120 per cent. The average of 21 successive determinations, just taken from my note book, is 113·1, the highest being 122·5 and the lowest 101·0. Two samples which I examined this week gave respectively 105·1 and 111·3. The average of all my figures, since I began to use this process is 112·2 per cent.

"I have on two occasions obtained very low results, *viz.*, 71·5 and 62·2 per cent. respectively. I am unable to offer a satisfactory explanation of the abnormal figures, as pressure of other work prevented my investigating the matter at the time, but I have no reason to doubt the genuine character of these particular samples, judging from the other results obtained in their analysis. However, I think the results of more than 200 determinations, all fairly concordant, with the two exceptions just mentioned, should be sufficient to convince anyone that the figures (18·68 and 10·4 respectively) given by Dr. Lewkowitsch in the Journal of the Society, Vol. XV., p. 859, are erroneous; that is, if they refer to raw linseed oil."

Here I may leave Dr. Lewkowitsch, in order that I may refer to a means of ascertaining the iodine absorption of oils—the most important factor in the analysis of oils—with sufficient practical accuracy, with the minimum expenditure of labour and of time. After I had shown that in the case of most fats, at least, a definite ratio existed between the Hübl number and the gravimetrically ascertained bromine absorption, I investigated, together with C. A. Mitchell, the relation between the heat evolved on bromination and the Hübl number.

On dissolving 1 grm. of the fat or oil to be examined in 10 c.c. of chloroform, either in a test tube packed with cotton-wool in a beaker to prevent loss of heat, or, better, in a vacuum jacketed Dewar tube, and on adding a more than sufficient amount (1 c.c.) of bromine to combine with the oil, a distinct rise of temperature is observed. This rise of temperature is very small with cocoanut oil, which has the lowest iodine absorption of natural fats, and very high with linseed oil, which has the highest iodine absorption. It was soon found that a very definite relation existed between the rise of temperature and the iodine absorption. Under the conditions described and with the tubes used by us, the rise of 1° C. corresponds to 5·5 per cent. of iodine absorbed from Hübl solution. This is seen from the following table, reproduced from the "Analyst":—

Oil or Fat.	Rise of Temperature with Bromine.	Hübl Figure.	Calculated I. No.
1. Lard........................	10·6	57·15	58·3
2. "	10·4	57·13	57·2
3. "	11·2	63·11	61·6
4. "	11·2	61·49	61·6
5. "	11·8	54·69	64·9
6. "	11·8	63·96	64·9
7. "	10·2	57·15	56·1
8. "	10·4	57·80	57·2
9. "	9·0	50·38	49·5
10. "	11·0	58·84	60·5
11. Lard + 10 per cent. cotton oil	11·6	64·13	63·8
12. Lard fatty acids	10·4	59·60	57·2
13. " "	11·0	59·15	60·5
14. Mutton fat (kidney)........	8·1	44·48	44·5
15. " (flare)...........	7·6	39·70	41·8
16. Butter......................	6·6	37·07	36·3
17. "	7·0	38·60	38·5
18. " (fatty acids)...........	6·2	36·50	34·1
19. Almond oil..................	17·6	96·64	96·68
20. Olive oil	15·0	80·76	82·5
21. Maize oil..................	21·5	122·00	118·2
22. Cotton oil..................	19·4	107·13	106·7
23. Castor oil	15·0	83·77	82·5
24. Linseed oil..................	30·4	160·70	167·2
25. "	31·3	154·90	172·0
26. Rape oil	18·4	88·33	101·2
27. "	17·6	77·20	96·8
28. Cod-liver oil..................	28·0	144·03	140·0
29. Oil sent as olive oil..........	19·0	108·50	104·5
30. " " "	19·2	105·70	105·6
31. " " "	18·9	105·70	103·9

It is obvious that if there were no definite relation between the amount of bromine entering into combination with the fat, in other words, between the weight of brominated fat obtainable and the degree of non-saturation, otherwise the Hübl number, nothing like regularity as shown to exist by the above table could have been observed. I may say, that since the publication by Mitchell and myself of the above numbers, I have employed the method almost daily in my laboratory, mainly upon edible fats, and have checked on very many occasions the numbers obtained by the Hübl method, never finding a greater difference than 3 to 4 per cent., calculated as iodine. I have thus made some hundreds of determinations, and am quite satisfied with the results. I need not trouble to insert them, as they wholly confirm those contained in the table. Wilson (Chem. News, Feb. 21, 1896, p. 87) corroborates and Dr. H. Wiley (Journal of the American Chem. Society, April 1896, p. 378), after thoroughly investigating the matter, testifies to the value of the method in analytical operations. At the same time Wiley makes a slight modification in a proper direction, by using instead of pure bromine a solution of bromine in chloroform.

I use the method solely as a sorting test, while for all important purposes I have made it a rule to determine the Hübl number also in the orthodox manner. The latter is the standard method, by which all modifications having the same object in view must be judged, and I heartily agree with Dr. Lewkowitsch that modifications of a standard method should not be lightly introduced. But when the many difficulties besetting the Hübl method are taken into consideration, it will be conceded that a mode of working which takes less than as many minutes as the Hübl method takes hours, and for practical purposes, in many cases at least, gives as good results, has some justification for its existence. As Dr. Lewkowitsch was cognisant of the thermometric method (by his reference to it in his paper), yet involved it in his most unfavourable criticism of the gravimetric study which led up to it, I need not apologise for having referred to a method already brought before another professional audience and which I know to have been tested and adopted by a number of my professional brethren.

Discussion.

Dr. J. Lewkowitsch said that his remarks on the iodine method did not refer at all to Mr. Hehner. Indeed,

he was glad to congratulate Mr. Hehner on the fact that he was one of the few chemists who did not come under the stricture of rushing into print with a trifling modification of the iodine method. Mr. Hehner's criticism of his paper was unfortunately mixed up with matter which was beyond its scope. For instance, he spoke about the heat test, whereas he (the speaker) had not said a word about it or anything else touched on in Mr. Hehner's further paper. Mr. Hehner had in his later paper made the positive statement that the action of bromine on unsaturated fatty substances is complete and quantitative. Against this statement he had put a series of figures, some of which agreed with Mr. Hehner's and some of which widely disagreed. He held that his figures contradicted in the most positive manner the usefulness of the gravimetric bromine process as a general process. Referring to his own experiments he admitted that in the case of fish and linseed oils they had been purposely pushed to the extreme in order to show that there was no knowing when to stop drying at 125° C. They had been carried out by three assistants who had had considerable experience in such work. With this method, however, they were helpless in the case of the last two oils and could not determine when the decrease ended. If it were desired to uphold this method as reliable, whilst his own experiments showed figures which did not agree with the author's, surely the only way out of the difficulty was a new series of experiments and figures. What he would have expected, would be a number of oleic acid figures agreeing with the Hübl method or some further notes as to how to work the method which seemed simple enough, but they had had nothing of the sort. He was not concerned to show why he agreed with Mr. Hehner in some cases. His case was that he totally disagreed in others. He could have gone further, but thought that he had done enough to show that the method was not a workable one.

Mr. C. A. Mitchell said that it was difficult to find a reason for Dr. Lewkowitsch having obtained such good results with certain oils, whilst with others not even an approximate agreement had been obtained. One might have expected this in the case of linseed oil, which contained a large proportion of linolenic acid, but it was strange that the fatty acids from soap, which would certainly not contain much of the more unsaturated fatty acids should not have given more satisfactory figures. With regard to linseed oil, the long experience of Mr. Williams seemed very conclusive. Moreover, he (Mr. Mitchell) had recently made some determinations on linolenic acid prepared from linseed oil. By the Hübl process, this gave an iodine number of 241, as compared with 235·8 by the gravimetric process, and it was noteworthy in the case of the latter that the bromine compounds at the last began to gain in weight instead of losing. He considered that granting that Dr. Lewkowitsch's experiments were correct, his conclusions ought to have been stated in a more modified form, and that every chemist who examined all the evidence would come to the conclusion that the bromine absorption as determined in some cases by the gravimetric method and in others by the bromine heat value should go hand-in-hand with the Hübl method, though not attempting to displace it.

Mr. Cassal deprecated any public discussion of the differences between two analysts of such high reputation as Mr. Hehner and Dr. Lewkowitsch. The results adduced by these two gentlemen were so widely different that experimental errors could not possibly be suggested as the cause. There was no doubt some fundamental difference in the method of working, and he felt sure that by private correspondence or discussion between themselves the true reason could be discovered and the matter dealt with satisfactorily. It was impossible that either of them could be so completely wrong as each seemed to suppose the other to be at the present moment. His own experience led him to the conclusion that Mr. Hehner's results correctly represented what took place on the application of the process, as he understood it.

Mr. Hehner, in reply, said that he had no wish to add to arguments which had been brought before the meeting. Facts would speak for themselves. He had before the meeting, large samples of the bodies actually formed in the experiments to which he had referred, and he felt sure that if Dr. Lewkowitsch would repeat his experiments he would find results which would agree with his own.

Meeting held on Monday, February 1st, 1897.

DR. R. MESSEL IN THE CHAIR.

OZONE, ITS COMMERCIAL PRODUCTION AND APPLICATIONS.

BY E. ANDREOLI.

In a paper read before the Academie des Sciences, Fremy and Becquerel had confirmed Marignac and De la Rive in stating that ozone was oxygen condensed by electrification. Andrews, in a paper read before the Royal Society in 1856, held that ozone was simply an allotropic modification of oxygen. Soon afterwards Andrews and Tait published the results of researches on the density of ozone, and stated that only a minute percentage of oxygen could be converted into this substance, that electrolysis yielded less ozone than could be produced by electric machines, and that the sparks of an induction coil, even with the aid of two Leyden jars, gave only insignificant results. After Andrews came Brodie, whose paper on this subject will never be forgotten in the history of ozone. The physicists and chemists of that day used minute apparatus, which yielded a very small quantity of ozone. (The tube made by De la Rive had a diameter of one line.) On the work done by these instruments however, were laid down the rules and principles which guide us to-day in producing, no longer milligrammes, but kilogs. of ozone, in powerful generators.

In round figures, 1 kilo. of ozone is the theoretical quantity formed by the expenditure of one electrical horse-power-hour, but there is a wide slip between theory and practice. We have never yet obtained more than 50 grms. per electrical horse-power-hour, and we know of no apparatus which will give in continuous work 35 grms. per horse-power-hour. This, of course, refers to large ozone generators; and not to the small laboratory apparatus in which a high percentage of ozone is produced, and through which pure oxygen is sent after having been dried and brought down to a low temperature. The qualities of a good ozoniser are durability, easy construction, and efficiency. I will now give a brief sketch of what had already been done. The Siemens ozoniser consisted of a glass tube lined with a metal foil and inserted in a larger glass tube coated with metal on the outside. The ozone was formed in the annular space. About 10 years later, Ladd constructed an ozone generator which consisted of parallel sheets of glass covered with tin and alternately connected to the two poles of high electric tension generators. Numerous imitations of these two types have been and are still made, but without much success.

The most formidable obstacle to be surmounted in producing large quantities of ozone is the heat of the discharges which destroys ozone. When the temperature rises, the glass which separates the metallic electrodes loses its dielectric proportion and gradually becomes a conductor. Current soon passes through the glass, which cracks; and the apparatus ceases to work. It is all very well to produce ozone between glass surfaces, which being inoxidisable absorb no ozone, but the interposition of two dielectrics between two electrodes tends to reduce the yield of ozone, and it is really more advantageous to use a metallic electrode between dielectrics separating it from other metallic electrodes placed on either side. In this manner only and subject to certain conditions which I will enumerate can the minimum amount of heat and the maximum amount of ozone be obtained. I was the first to construct, five or six years ago, an ozoniser, where between two dielectrics and apart from them was a metallic plate. This new departure constitutes what I call the open ozoniser. The oxidation of the metallic plates by the ozone is after all very slight, and by special means can easily be prevented; and, if, by neglecting the prescriptions of servile practice, a yield of twice the amount usually produced is obtained, who would complain of the little harm which possibly is done by this oxidation?

Dr. Houzeau, so far back as 1871, held that the quantity of ozone resulting from the free passage of air between two naked metallic electrodes is greater than when the air passes round similar electrodes enclosed or sealed within glass, *i.e.*, when the air does not pass freely, and is in direct contact with the metallic electrodes. In the construction of a good ozoniser it is therefore quite unnecessary to electrify the air between glass surfaces, and to prevent it from coming in contact with any metal. It is not by modifying haphazard the shape and dimensions of an ozoniser that its power is increased; on the contrary, everything should be calculated and have a *raison d'être*. No revival of what has been done up to now and was not good 20 or 30 years ago can be so in 1897.

The greatest obstacle to overcome, I already told it, is the heat, which renders impossible the continuous generation of ozone. The electric silent discharge, notwithstanding its inoffensive appearance, causes a considerable rise of temperature, and it is easily conceivable that its luminous waves being continually emitted from the electrodes on to a glass surface will eventually yield disastrous results and reduce the yield of ozone to a minimum. To get over this difficulty the air to be ozonised must be left as little as possible in contact with the electrified surfaces, or with the points of the electrodes. The larger the electrodes the less satisfactory the ozonisation will be, because the longer the passage between electrodes coated, so to speak, with a layer of flame, the higher will be the temperature of the gas. This means that to work under the most advantageous circumstances, the distance to be traversed by the gas to be electrified must be extremely small. If this condition is not realised, and the ozonised gas is not very rapidly drawn from the electrifying surfaces, no quantity of ozone greater than 12 to 15 grms. per electric horse-power-hour will be available. If the air is driven through the ozoniser at a convenient velocity between the ozonising surfaces, and its contact with these surfaces is extremely short, a comparatively large amount of ozone will be obtained, even after abstracting from it the nitrous compounds, which increase as the contact of the air with the electrodes is prolonged.

My ozonisers give 30, 40, and 48 grms. of ozone at the utmost per horse-power-hour. Is not this very little? It seems to render its use prohibitive; but when Dr. Frölich of Messrs. Siemens and Halske, was taxed with not producing more than 20 grms., his reply was that 20 grms. produced by one horse-power-hour "will bleach 110 lb. of linen as well as grass bleaching during three days. It will in presence of chlorine bleach and refine 88 lb. of potato starch to such a degree that the colour becomes a clear white, and the bad odour and tastes are removed; if this is roasted and ozonisation continued, a product resembling gum arabic is obtained, 20 grms. of ozone being sufficient for 66 lb. of this product; the same quantity will ozonise a room containing 8,000 cubic metres in such a way that healthy persons can just endure it."

Do not suppose that we exaggerate the value of our work in speaking of our improvements in the construction of ozonisers. No progress, as a matter of fact, would have been realised if we had not had at our disposal powerful dynamos and high tension transformers, which the first workers on ozone never knew. There is nothing extraordinary to-day in loading a 5 or 6 Kilowatt alternator with several 10,000 volt transformers. It is only fair to acknowledge the services rendered by those electricians who have facilitated the development of ozone-making.

Speaking of alternators and transformers, these essential factors of ozone production, is it not very remarkable that the first alternate current dynamo combined with an induction coil (a real step-up transformer) was used by Dr. W. Spottiswoode in 1875, before transformers were invented, and that he used them for the production of ozone?

Listen to this extract from the "Philosophical Magazine" (1875, p. 380): "I have lately tried," says he, "a mode of exciting an induction coil which I have not seen elsewhere described, and which appears to promise valuable results. It consists in connecting the primary circuit directly with a dynamo giving alternate currents. I have used one of M. de Meritens' excellent machines, driven by a $3\frac{1}{2}$ horse-power Otto silent gas engine. The speed of dynamo so driven is 1,300 revolutions per minute. The advantages of the method are:—

"1. That, as the machine effects its own make and break, both the contact breaker and the condenser of the induction coil can be dispensed with.

"2. That the breaking of the primary, and consequently the delivery of the secondary currents is perfectly regular.

"3. That the quantity of the currents in the secondary is very great.

"With a 20-in. coil by Apps, I have obtained a spark about 7 ins. in length of the full thickness of an ordinary cedar pencil. But, for a spark of thickness, comparable at least with this, and of 2 ins. length, an ordinary 4-in. coil is sufficient."

Du Moncel was the first to demonstrate that very curious property of the induction spark of passing through glass without breaking it, and also producing ozone. This phenomenon, he said, is the consequence of the fact that under the influence of condensation the molecules of the insulating substance interposed are polarized like liquid molecules in electrolysis: so that they all contribute to condense the discharge from surface to surface on the sheet of glass. This electrification is complicated by momentary absorption of part of the charge, absorption which varies with the electrostatic capacity of the dielectric.

Books, 30 years ago, tell us that the electric effluvium is a luminous discharge, which spreads in an infinite number of small discharges over a large surface, and produces neither heating nor disintegrating mechanical action. This definition is excellent and very accurate except as regards heat. The electric effluvium or silent discharge is characterised by a dissemination of sparks, feeble light, and small amount of heat produced. But when it acts on a gas between two electrodes, there is a strong evolution of heat, which hinders and finally stops the production of ozone. The difference between the silent and the disruptive discharge is that the latter is longer in duration and causes heating in the gaseous matter through which it passes, thus preventing formation of condensed products. The silent discharge on the contrary, which is, as a great chemist remarked, only the diffusion of the ordinary spark in thousands of discharges which occur from molecule to molecule in the electrified gas, crosses a much smaller interval. The duration of each isolated discharge is much shorter than that of the spark, and the matter acted on is less, so that condensed products are formed and are withdrawn by reason of the extreme brevity of the discharge from any destructive effect. It is the silent discharge and not the disruptive discharge which is utilised for the production of electrostatic ozone.

When the current from a high tension transformer passes between two parallel metallic plates, apart one from the other, there is an explosion, a violent discharge with evolution of nitrous emanations, but there is hardly any ozone produced unless the plates are at such a distance that owing to the resistance opposed by the dielectric properties of the ambient air, no disruptive discharge is possible. For reasons which it would be too long to explain, it is not desirable to try to produce ozone by reducing long sparks and making them smaller and smaller until they resemble the silent discharge. The dielectric properties of the air are variable, and I have preferably adopted the old and simple contrivance of a dielectric, such as glass or porcelain, which, after all, answers very well my purpose.

A short description of this apparatus will enable you to judge if I have constructed an ozoniser which can work in a continuous manner and produce ozone on a comparatively large scale. My main object has been to have a large electrifying surface, and at the same time to prevent the rise of temperature caused by the electric effluvia. My electrodes are boxes through which cold air or a cooling liquid may circulate. One set of electrodes is connected to one pole of a high tension transformer, the other is connected to the other pole, and there is between each electrode a glass separator or dielectric. The electrodes are narrow and long; they are kept at a proper temperature by cold air or water; they work on both sides, and their point-

bearing surface is very large and active. But it is not sufficient to have some means for cooling them, the longer the air is in contact with the electrodes and with the dielectric, the warmer it is, and therefore the smaller the quantity of ozone formed. To obviate this, the air is forced from beneath between the electrodes, through the slits or apertures of a false bottom. Its velocity is high; the gas traverses a very short electrifying space, and almost as soon as the gas is ozonised it is removed from contact with the glow of the silent discharge.

Although the oxidation of metallic electrodes is not so dangerous as some people say, and the loss of ozone in this respect is therefore slight, we none the less studied how to remove or at least diminish considerably the possibility of this occurrence. This we attained by covering our electrodes, that is to say, our point-bearing metal boxes, with a few coatings of a varnish, which resists a much higher temperature than that which might rise in ordinary ozonisers, and which is practically inoxidisable.

I can employ flat electrodes, but I prefer electrodes provided with numerous points, which on both sides of a dielectric separator, I set to work points against points, or points against flat surfaces. There is more to be said than one would think about the influence and rôle of points in the production of ozone. It is obvious that the electric effluvia will flow out better and more powerfully from a multitude of points than from a flat plate, on which the tension is uniform. More ozone is formed on angles or points than in condensers with flat surfaces, or in plain tubes, &c. It is at the top of a cone that electric density has its greatest value: and electricity accumulates there so abundantly when the point is very sharp, that, in spite of its insulating properties, the ambient air is electrified, repelled, and replaced by a fresh amount of air, which is, in its turn, electrified, and so on. Points have not, as Franklin thought, power to attract the electric fluid, but they have the property of liberating and discharging with the greatest rapidity the electricity with which they are charged. Each square foot of my electrodes bears 46 rows of 56 double points, and in my ozonisers of 12 boxes the number of points is 53,760.

While studying the question of commercial ozonisers I pursued the construction of small apparatus for household employment, and since 1891 I had made various models of ozonisers, which worked in a satisfactory manner, all of which were what I call open ozonisers, *i.e.*, where the air circulates freely and is partially transformed into ozone. But in 1893, having noticed the feeble luminosity of an incandescent lamp when placed in contact with only one of the terminals of the secondary circuit of an induction coil, it struck me that I could use a vacuum glass tube, traversed by a central wire, and surrounded by a spiral made of serrated wire. I put the central wire of the tube in contact with one of the terminals of an induction coil, and the outer spiral with the other. At once the tube filled with a beautiful whitish brightness, and simultaneously the violet glow of the silent discharge formed ozone in abundance between the surface of the glass tube and the points of the spiral. No glow, no ozone! no vacuum in the tube, no luminosity! and if the points of the aluminium discs which I substituted round the vacuum tube for the tinned steel spirals are too near the glass, the amount of ozone will be much less than when a space of about a millimetre separates the glass from the points. This last remark is applicable both to small and large ozone generators.

Eminent physicians, well known in England and abroad, have prescribed ozone in many cases. Ozonised oxygen insufflated on wounds produces a remarkably beneficial action. I am informed that an ulcerous wound, which had resisted every other treatment during two years, began to heal at once, and the wound is now filling with healthy tissues. In rooms, it purifies the air. Many consumptive people have been cured by inhalation of ozonised oxygen. The essential point is not to administer ozone *in extremis*. If you wish to see some examples of the efficacy of these ozone inhalers, I may refer you to the *Lancet* of 9th May 1896, and the *British Medical Journal* of the 10th October 1897, where two doctors tell how they treated patients, with complete success, in cases of anæmia and other diseases, simply by employment of the ozone of these luminous tubes.

When an ozoniser gets hot, it is not sufficient to stop it for a few moments to allow it to cool, in order to prevent the destruction of ozone. Hautefeuille and Chappuis have found that once started, the destruction of ozone continues. The only way is to wait until the electrodes are quite cool. It is to the unknown temperature of the electric effluvia that the instantaneous decomposition of ozone must be attributed, and if to this unknown temperature we add that of the exothermic decomposition of ozone back into oxygen, it is easy to realise the importance of designing ozone generators, which will not heat, or will not heat much.

It results from Clerk Maxwell's researches on the manner in which temperatures are distributed among gaseous molecules that ozonised oxygen, even at ordinary temperatures, returns to the state of oxygen, because there are always some molecules which are at a higher temperature than others, and these determine the commencement of dissociation. The lower the temperature, the greater the probability that ozone will not be destroyed. In the cold, more ozone is produced than at ordinary temperatures; at medium temperatures ozone is decomposed, and at 250° C. its existence is impossible; but what we are in ignorance of, although we might know the temperature of the gaseous mass in an ozoniser, is the temperature to which oxygen is raised by electrification when it is allotropically transformed.

The methods of titration of ozone are the same to-day as 30 years ago, and nowhere in any treatise of analytical chemistry will you be able to find a mode of quantitative determination of the ozone commercially produced by the electrification of atmospheric air. The only method I know is the one adopted by Marié Davy at the Montsouris Observatory for titrating the ozone of the atmospheric air, and I have been told that it is not absolutely reliable. Quite recently I invited a specialist, one of the men whose experiments on ozone are the best known in the scientific world, to come to London to make, by his own method, a quantitative determination of the ozone produced by my apparatus. But when he was informed that he would have to determine the ozone contained in electrified atmospheric air, he wrote that his method was devised for ozone produced from pure oxygen, but he could not use it for atmospheric air because of the nitrous compounds present.

Few persons at the present day can imagine what an important part nitrogen and its compounds have played in the history of ozone, and we may smile at the dogmatic and contradictory assertions, which, less than 50 years ago, chemists hurled at each other. After having spoken of ozone as a new substance, Schonbein found nitrous acid in it. Sterry Hunt considered there were nitrites of ammonia present. Rivière and De Fallenberg declared that the ozone of Schonbein was simply nitrous acid. In 1862, Sauvage asserted that ozone does not exist, and that what was called ozone is merely an oxygenated nitrogen compound, or rather the aggregate of all the oxygenated compounds of nitrogen. Under electric action, he said, the oxygen is charged with positive electricity and electrifies the nitrogen with which it is in contact: the two gases combine and form a nitrous substance which has been called ozone.

Williamson stated that electrolysis gives peroxide of hydrogen and not ozone, that the electric spark only formed nitrous acid; according to another, electric discharges in a mixture of oxygen and nitrogen produce hyponitric acid. Dubrunfaut denied the hypothesis of the existence of an allotropic oxygen, and his theory was that ozone is only a nitrous compound. It is quite beyond doubt that when atmospheric air is subjected to electric effluvia, nitrous products are formed; but with our small vacuum tube, which contains an electrode, and is provided with an outside armature where the glow of the silent discharge appears and ozone is generated, there are little or no nitrous products, because the tension is too weak and the air is too rapidly renewed to allow of their formation. Ozone forms in the air, and even somewhat rapidly, with comparatively low tensions, whereas Berthelot remarked, the proportion of nitrous products becomes nil, or, more accurately, unappreciable by the existing methods of research. The

formation of ozone and that of nitrous compounds are not correlative; and therefore to obtain a maximum quantity of ozone and a minimum of nitrous products in the electrification of atmospheric air, the gas must pass with the greatest possible velocity in an apparatus where the electrifying surfaces are long but not high.

If low electric tension were sufficient to form a large amount of ozone, said Berthelot, the oxygen of the atmosphere on which low electric tension constantly acts, would soon destroy all the organic and other oxidable substances spread over the surface of the earth. Does this not remarkably compare with this statement of an illustrious English chemist who, speaking of the combustibility of nitrogen, said, "the reason why, when once nitrogen is set on fire, the flame does not spread throughout the whole atmosphere, and deluge the whole world in a sea of nitric acid is that the igniting point of nitrogen is higher than the temperature produced by its combustion, and therefore the flame is not hot enough to set fire to the adjacent gas."

It requires only a very slight contact of the air with the glow of the silent discharge to produce ozone. The formation of ozone is instantaneous. Nitrous products take longer to form than ozone; the less the air is heated by the silent discharge the less nitrous compounds there will be, without any decrease in the amount of ozone.

Ozone may be regarded as an explosive since it cannot safely bear a pressure of more than 7 to 9 atmospheres, and then is assumed to be decomposed and to explode. According to Jouglet, nitro-glycerine, dynamite, nitrogen iodide, or chloride, and similar substances explode in contact with ozone and the properties of common powder which has been in contact with ozone are impaired after six weeks.

Jean, a French chemist, who was one of the first to devise an ozoniser, observed that when carbonic acid was passed between the glass plates of his condenser it was decomposed into carbonic monoxide and oxygen, which latter was partly converted into ozone. Sir Benjamin Brodie also was struck by the fact that in electrifying oxygen by the silent discharge, he only converted 20 per cent. of it into ozone. His description of the very little known experiments he made, is worth quoting:—"It occurred to me as possible to replace 80 per cent. of oxygen, unaffected by the action of electricity, by an indifferent gas, and thus to effect the complete conversion of oxygen into ozone.

"This idea was the starting point of the following investigation:—

When pure and dry carbonic acid gas is subjected in the induction tube to the electric action, a certain proportion of the gas is decomposed into carbonic oxide and oxygen a portion of which appears in the form of ozone. "Various experiments were instituted with a view of ascertaining the conditions most favourable to the conversion of a large proportion of this oxygen into ozone. The conclusion at which I arrived was that the greatest proportion of ozone in relation to the total oxygen was produced when a rapid current of dry carbonic acid gas was acted upon at low temperature. When those conditions were realised, it was found that 75 per cent. of the total oxygen eliminated in the induction tube by the decomposition of the carbonic acid gas could readily be converted into ozone, but that it was difficult to pass this limit."

(Proceedings, Royal Society XXI., 1872, pp. 483, 84, 85.)

Thus, so far back as 1872, Sir Benjamin Brodie recommended a rapid passage of the gas through the ozoniser to obtain high results, and it is because this was not, or could not be carried out, that most ozonisers could not and cannot be turned to industrial account.

Another remarkable fact in connection with the electrification of carbonic acid, is that according to Berthelot it leads us to suspect the existence of percarbonic acid. The substance formed attacks oxidisable bodies with great violence.

In regard to the employment of ozone to the manufacture of aerated waters, there are two points to be considered —the absorption of ozone by the water, and the formation of carbon monoxide by the silent discharge. The latter may not be found to be so noxious as would be surmised. Grüber has stated that below a certain point of dilution, say, 0·02 or 0·05, this gas ceases to be dangerous, and it appears that below this limit, carbon monoxide, when it finds its way into the human body, converts carboxyhaemoglobin into oxyhaemoglobin. On the other hand, Prof. Olczewski, in a letter dated 1896, said, that because of the low solubility of oxide of carbon in water, it is probable that the solution would be harmless, and he added that ozonised carbonic acid could be used safely if when leaving the ozonisers it was forced through a chromic acid solution, which would convert the oxide of carbon formed into carbonic acid. The real difficulty is that ozone does not readily dissolve in water, and that in contact with the metal of the cylinder through which it is forced, it decomposes. The sole result in this case is that there is a little more oxygen in the water than there was before.

The only industrial applications of ozone at the present day, which gives results, that is to say, real profits, and I may add, substantial profits, is the manufacture of certain artificial perfumes.

What importance should we give to the ozonisation of beer casks to cleanse and purify them, that is to say, destroy the moulds, micro-organisms, &c.? In breweries, it appears, they have excellent methods of destroying ferments, which cause diseases of beer.? I merely mention this, because of the sterilisation which ozone effects very rapidly.

Very few people know that ozone can be utilised for making "dégras," *i.e.*, spent oil, which is an indispensable substance in the leather industry. Oxidised fish-oil, mixed or not with animal oils, gives an artificial "article," which is cheaper than genuine "dégras" and possesses all its properties. For "dégras" as for most other applications of ozone, it is not enough to force ozone into the substance to be oxidised, there are conditions to be fulfilled which the manufacturers only know and there is a limit to oxidation which must not be overstepped.

It is preferable to ozonise wood for making musical instruments, instead of drying it by the traditional method, which consists in keeping it for a long time in sheds. The sounding-boards of pianos have then a great sonority, and resist variations of temperature better, which is important for exportation. Woods for cabinet and carriage making when seasoned by ozone have a more pleasing appearance; their tint is softer, and they take varnish much better.

There are in Germany two factories where Messrs. Siemens and Halske two or three years ago have introduced ozone. In one of them they bleach linen cloth and yarns, and in the other starch and other amylaceous products are bleached and refined. In Greifenberg (Silesia), fabrics and yarns are bleached; the starch works are at Kyritz, but ozone is not employed singly. Decolorisation by ozone alone is a myth; Messrs. Siemens, by alternate passages through hypochlorite of calcium and ozone, bleach well and cheaply. It is what we may call chlorozone bleaching, had not this term been already adopted.

We also wished to subject to an ozone treatment the starch manufactured in this country; but when we applied to manufacturers, they told us that they only make rice starch, which is white, and that bleaching is not required. I will add that the ozonised products of Messrs. Siemens are quoted much higher than ordinary starch, leiogum, and dextrin. In vinegar works there ought to be a great advantage in adopting the ozonisation of the wash.

We made numerous attempts to decolorise molasses, syrups, and moist sugar, but they were not crowned with any brilliant success. Firstly, the action is slow, and consequently the bleaching, which is never very marked, is expensive.

In the case of drying oils, it is very desirable to render them paler and transparent for the manufacture of varnish and for their other uses. There is no need to alter the plant for oil-drying or linoleum works, or for any other oil or fat works where oxidation has to be replaced by ozonisation. Wherever there are installations for forcing or aspirating air through fatty masses, you have simply to run this air through one or several ozone generators, whence in an ozonised condition it may pass into your receivers, where it does more work than blown air.

In Schönbein's time "ozonised turpentine" was the name given to oil of turpentine strongly agitated in the air and light, and which in this state possessed decolorising and strong oxidising properties. He was the first to form peroxide of hydrogen by agitating with air turpentine and water. I have no intention of discussing the existence or the absence of ozone in aerated turpentine. It is not air, but ozone, or rather ozonised air, which I pass through turpentine, either singly or mixed with water. I obtain, then, really ozonised turpentine and the water with which it has been bubbling is, according to the duration of the contact, a more or less rich solution of peroxide of hydrogen. Thus, by the aid of ozone, we can make peroxide of hydrogen, but you can pass as much ozone as you wish through water there will be no trace of peroxide of hydrogen in it. I will add that I look with the greatest confidence on ozone as a cheap producer of peroxide of hydrogen. This ozonisation of turpentine and water is much more rapid and energetic than mere oxidation by air, and if the products of this ozonisation were combined with coadjuvant chemicals, no doubt a sanitary compound would be obtained of exceptional value. The disinfection of rags by ozone is a topic which must not be neglected.

Ozone has shown its value as a bactericide and germicide. An installation is being constructed at present near Paris for sterilising water from the Seine, by means of the Tindal and Van der Sleen apparatus. The Municipal Council of Paris has granted a concession for the daily disinfection during three months of a certain quantity of water—a concession which will be made permanent if conditions of efficiency and economy are realised. It is estimated that the sterilisation of 5,000 litres of Seine water requires one electric horse-power-hour. The practicability of the sterilisation of water depends on the quantity of ozone available per horse-power-hour. With only 10 or 12 grms. of ozone it is evident that there is little chance to economically purify contaminated water. But here we have the starting point, which is the deadly action of ozone on bacilli and even spores.

As a bleaching agent ozone will not do the work which hydrogen peroxide or bleaching powder does. When alone it will not bleach tissues nor fibres. It only appears to bleach when in conjunction with other bleaching agents.

There is a note by a certain Wiedemann read before the Académie des Sciences, in which he stated that he had erected in Boston works in which he treated daily 300 barrels of raw whisky, the resinous matters of which which disappeared in 20 minutes under the mellowing action of ozone. It is evident that the improvement of alcoholic liquids being due to oxygen, the ozone treatment must hasten this effect. Ozonised air must not be used, however small the percentage of nitrogen compounds present, it would be detrimental to the quality of the final product. Electrified oxygen only is beneficial. It is difficult to say which is the best practical way to adopt for the treatment of alcoholic liquids. It is indispensable to take into account the nature of the wines to be treated and not to ozonise light wines in the same manner as richer varieties, sweet wines like dry, and so on. Port wine is that which shows the effect of ozonising most rapidly. At the end of two or three days a deposit is formed, and goes on increasing until a very clear wine is obtained, which has an appearance it would only have after remaining in bottle for years. The difficulty is not to overdo the treatment.

It is amusing to review the post of ingenious combinations which have been contrived in order to sell under the name of ozone, medicaments, beverages, disinfectants, and panaceas, in which ozone plays a very small part, or none whatever. If people understood how little ozone is retained by water and liquids, there would be fewer persons attracted by the grand alluring name of ozone spread out on labels. Pseudo-ozone seems to be more in demand and pay better than genuine ozone.

The production of an ozone generator constructed on a rational basis, such as was suggested by scientists like Sir Benjamin Brodie, and which would answer the purposes of manufacturers desirous of applying it on a large scale, has been my chief aim. The great problem is the economic production. I do not pretend to have solved it, but I think that I have constructed a good strong commercial apparatus, which can be adapted to the needs of manufacturers. Its average output is, as I said before, more than 30 grms. of ozone per horse-power-hour. Although this is very little, it is something, and I am sure much higher results are possible.

Calculating at the rate of 30 grms. per horse-power-hour, the cost of ozone would, at 1*d*. per horse-power-hour—

	£	*s*.	*d*.
300 grms. for 10 horse-power-hours	0	0	10
3 kilogs. for 100 " "	0	8	4
30 " 1,000 " "	4	3	6

I hope to be able to tell you ere long in a more complete paper, the progress which we realised, what we have done in various works, and I will give you facts and figures about the advantages arrived at by replacing air by ozone in several large branches of industry.

Discussion.

Mr. A. Gordon Salamon said that he had had some experience of the industrial working of the apparatus explained in the paper just read. That paper contained more information on the subject of ozone than was available in any other source, and he hoped the author would render it still more complete and valuable by adding to it a short appendix of the bibliography of the subject. He had been concerned in one of the first important industrial applications of ozone, and he was justified in saying that the results had been successful, both practically and economically. He would warn industrial chemists, however, not to indulge too much in theoretical speculations on the subject, or they would probably be disappointed. It was far better, in this case, to rely on the actual product as obtained than upon costs based upon theoretical yields. It must be recollected, too, that the stated production of the apparatus per horse-power-hour must, in the first place, be divided by three, because only one-third of the ozone produced was, in nearly all cases, actually available for oxidising purposes. Next, they must take into consideration the velocity of the air passing through the machine, as the production and any comparisons must obviously largely depend on that. With regard to any ozone machine, the great drawback was the tendency to heating, to which the author had called attention. Unless this could be satisfactorily obviated, he did not think the apparatus would be likely to succeed in industrial applications. Whether the author's means to that end were the best, time alone would show; at any rate, he had rendered service in pointing out that the heating of the ozone, and its consequent decomposition, was the important point to be avoided. Whether that heating could best be reduced to a minimum by a system of point discharge, was a question upon which he felt some doubt. He was satisfied, however, that the author had hit the true point in ozone production when he stated that the greater the frequency the better the yield and the less the heat produced. With regard to the source of production, he thought that at present, except in special cases, they were limited to atmospheric air. One could not at present deal with the industrial application of ozonised oxygen. To do so would be to court failure from many points of view—mainly because of the expense involved; and next, because of the heat developed. With respect to the industrial application of the apparatus, there were difficulties which the author had not touched upon, but which would become apparent to anyone dealing practically with the subject. For instance, he regarded the fittings of the apparatus as constituting just as important a feature as the apparatus itself. He had experienced much trouble, disappointment, and indeed loss in the rotting of the various pipes and fittings employed in the ozone apparatus. It was only right to mention this, and he was sure that Mr. Andreoli would admit its importance. The question of moisture also constituted an important difficulty, especially when using ordinary air as the source of production, seeing that to deal with dry air was practically impossible. He knew that it was possible to overcome these difficulties; at the same time it was only right that they should be pointed out to those who might be desirous of experimenting with the machine. As to the future of ozone, he would not like

to say much, except that from what he had already seen achieved, he believed it had a great future. Where one had, under ordinary circumstances, to employ large volumes of acid or other oxidising material in order ultimately to obtain a mere kernel as the oxidised product—anyone acquainted with the cost of manipulating large bodies of material would admit the industrial economy of employing a good ozone apparatus and good means of distributing its products. Again, ozone might have further applications, such as the acidification of alcohol, the sterilisation of water, the thickening of oils, for which purposes the ordinary oxidising agents were comparatively useless. He had seen Mr. Andreoli's machine applied to such purposes, and, so far as he was in a position to form an opinion, he regarded its future as most hopeful.

Dr. W. S. Squire asked whether the author had tried the effect of passing air, artificially cooled to a very low temperature, through the apparatus. That would, he thought, get rid of the difficulty caused by the heating of the air when used at the ordinary temperature. D'Arsonval had produced ozone by passing the electric discharge over liquid oxygen in a suitable apparatus, and he believed with fairly good results. If oxygen could be converted into ozone in larger quantities in consequence of using a very low temperature, it might be worth while even to employ liquid oxygen, for one would in that way get rid of the difficulty arising from moisture, because the gaseous oxygen derived from liquid oxygen was absolutely dry. As to the possible industrial applications of ozone, he feared they would be extremely limited. Mr. Salamon had suggested that it might be used for the acetification of alcohol. It appears that the utmost quantity of ozone that could at present be obtained was 48 grms. per horse-power. Of these, only 16 were really available for oxidising purposes. Now 16 grms. of oxygen—that is to say, one horse-power-hour—were required to convert 23 grms. of alcohol into 30 grms. of glacial acetic acid, a body worth at the present time about one shilling per kilogramme. These figures showed how utterly hopeless it was to use ozone for the purpose suggested. Ozone might perhaps be used to destroy certain colouring matters, for it frequently happened that these bodies, although affecting the tint of large quantities of material, were themselves present only in very minute proportions.

Col. Engledew thought that the difficulty arising from overheating in the production of ozone had been considerably overrated. He was now using a generator that could be run for six hours continuously without perceptible increase of temperature. The heat might rise after eight hours, when a new generator would take its place until the first had cooled down. They had thus no difficulty in continuously working. It was difficult to explain the precise cost of production, as they had not yet been able to measure it; but after two months' experimental working he might say that the cost for electrical energy did not exceed 12*s.* He felt sure that at that rate ozone could be adapted to commercial purposes. They had tried it for the deodorisation of sewage. A large beaker of very evil-smelling sewage was rendered quite inodorous in two minutes. They had also experimented with oils, and these had been perfectly sterilised. He need hardly remind members of the beneficient effects following the use of ozone in surgical cases, the prolongation of the life of the late Sir John Millais being a notable instance of its value in that direction. There could be no question, he thought, that ozone would come into general use for the ventilation of hospitals, factories, &c. Mr. Andreoli's work was the best existing paper on the subject of ozone, and the best thanks of those interested in the subject were therefore due to him for his valuable researches in it.

Mr. Otto Hehner agreed with Mr. Salamon and other speakers as to the immense amount of suggestions contained in the paper. Some of these might yield useful results in the future; at the same time there was much that was purely speculative in character. He would have liked to have seen something more tangible than a catalogue of possible applications of ozone, and to discuss these applications of it from the point of view of pounds, shillings, and pence. He feared there was nothing in the paper which would induce a manufacturer to spend a single farthing on plant for the production of ozone. He would like to know precisely what the author meant by a production of 48 grms. Did he mean 48 grms. of ozone or of active oxygen? In the former case only one-third of that quantity, namely 16 grms., would be active and available, and such a production per horse-power-hour was hardly satisfactory. It might, as Dr. Squire had said, be useful in dealing with certain colouring matters, and in bleaching generally there was a strong probability that ozone would be useful in future. But to talk of sewage purification by ozone was somewhat ridiculous, and likely to do harm to the commercial future of ozone.

Mr. Salamon asked if Mr. Hehner would state the amount of oxygen required to deodorise sewage.

Mr. Hehner, continuing, said, no doubt Mr. Salamon knew that as well as himself; but deodorisation was not the only question. There was, further, the removal of the organic matter, thus rendering sewage incapable of again becoming putrid. There were statements of fact in the paper which were open to doubt. If it were true that carbon monoxide could be produced from carbon dioxide by ozone, it was an interesting observation. In the last number of the *Berichte* had appeared an article discussing the action of ozone upon moist carbon monoxide, and showing that this was transformed into formic acid. The reduction of carbon dioxide mentioned in the paper was contrary to what one would expect. He did not say that ozone had not a future before it, but before it could be applied commercially a great deal remained to be done.

Dr. L. T. Thorne had had an opportunity of trying Mr. Andreoli's apparatus, and had also for many years past taken a deep interest in the question of ozone production. He could cordially agree with Mr. Salamon's remark that this question should be approached with an open mind, for the text-books were extremely unreliable. Mr. Hehner had referred to the uselessness of attempting to treat sewage by the small quantities of ozone now available. The force of that argument was obvious, and indeed Mr. Andreoli had disclaimed any such application of it on the basis of present production. On the other hand, they must be careful not to go to the opposite extreme, and imagine that ozone was not available for many other purposes, for in most cases they were hardly yet aware how much ozone was required for the work to be done. Mr. Hehner and Mr. Salamon had come into conflict over the sewage question; personally, he was inclined to side with Mr. Hehner, but would not like to be as positive as the latter was, or to say that ozone might not eventually be used in conjunction with other sewage purification processes. Mr. Hehner had spoken of one-third only of the ozone produced being available for work, but from his own experience he (the speaker) was not so sure of that being so in all cases. If one simply oxidised SO_2 into SO_3, one could only utilise a third of the oxygen in the form of ozone; but he had found in the case of mixtures of oxygen and chlorine, that in some cases (such as in the bleaching of some natural colouring matters) the whole of the oxygen was made available for oxidising purposes by the presence of the nascent oxygen liberated by the chlorine; and certain experiments seemed to point to similar effects from the active oxygen atom in the ozone molecule. That would make a vast difference to the industrial application of ozone. From his own experience of the apparatus he could confirm Mr. Andreoli's figures, and in the quantitative determinations he had used what he considered the most accurate method, namely, passing the ozonised air through acidified potassium iodide and measuring the iodine liberated. In that process they got the interference of the nitrogen compounds, and with the view of eliminating these, he had passed the ozone first over a solution of caustic potash or over pumice stone soaked in it. To his astonishment, the ozone was reduced to a much greater extent than he had expected. In one case the amount was over 50 grms. before washing, and was reduced to 28 grms. after. In order to verify the results he had modified the method by determining the apparent ozone, and under the same conditions had determined the nitrogen oxides by absorption in potash and subsequent estimation as ammonia. He then found that the quantity of ozone to be subtracted from the first determination was not 45 per

cent., but under 8 per cent. only; and, further, that in ozone produced in the Andreoli apparatus, the proportion of nitric oxide was very small. He had also found that ozone was a much less stable compound than was generally supposed, and was even partially decomposed by contact with aqueous potash. The instability of ozone was confirmed by Mr. Shenstone in his recent interesting paper. The question of moisture was of great importance in relation to the industrial application of ozone. Mr. Salamon had laid much stress on that point, though their information as to the extent to which moisture prevented ozone production were not very definite. In practice, only moderate dryness of the air seemed necessary.

Mr. Salamon remarked that he had not spoken so much as to the influence of moisture as affecting the yield of ozone, but rather of its action.

Dr. Thorne, resuming, was glad to have that point cleared up, because he thought that the need for dryness in the air had been over-estimated. That point was emphasised by Mr. Shenstone, who had found that in using absolutely dry oxygen he got practically no ozone at all, because of the rapid decomposition of absolutely dry ozone. With regard to Mr. Hehner's remarks as to the production of CO from CO_2 by ozone, he thought there must be some misunderstanding. He believed that if a mixture of carbonic acid and oxygen were subjected to the electric discharge, some of the acid would be reduced to carbonic oxide, but certainly ozone itself did not convert carbonic acid into carbonic oxide.

Mr. W. F. Reid regarded the paper as divisible into two parts—practical and speculative, the former being much outweighed by the latter. However interesting the apparatus before them, and the possible future applications of the ozone produced, he agreed with previous speakers that at present the industrial use of the process was very limited. He understood the author that even the claim that it might be used for bleaching was mythical. Then, again, various authorities differed as to its action upon alcoholic liquors. Further, the author had stated that ozone was not absorbable by water. In regard to one suggested application, namely, the forcing of ozonised air through oils, they had in atmospheric oxygen everything that was wanted; and if they employed a too active form of oxygen they might get a further stage of oxidation which would be injurious. Thus every suggested large field for the industrial application of ozone seemed to be cut off. He would like to ask, as to the current of air passing through the apparatus, whether the gain in the yield of ozone was not more than compensated for by the dilution of the current. It occurred to him that the oxides of nitrogen which seemed to be always produced at the same time as ozone might possibly at some future time themselves become a source for the chemical production of ozone.

Mr. Ling understood that an eminent scientific man had refused to estimate a sample of Mr. Andreoli's ozone on account of the presence of oxides of nitrogen. It seemed to him that most of the applications of the product proposed by the author would be vitiated for the same reason.

Mr. Bertram Blount regarded the paper as made up of extremely suggestive matter, but hardly useful from a practical point of view, and it seemed to him that discussion on a subject so highly speculative in its character was out of place in that Society. The results obtained by authorities such as Shenstone, to the effect that ozone can be obtained in quantity under particular conditions, had been to a great extent ignored. Further, he thought that chemical engineers would resent the implication that they were unable to cool and dry a gas economically; and the suggestion that because of this so-called difficulty, pure oxygen could not be used, he regarded as preposterous. For his own part he disbelieved in products which defied analysis, and as it had been suggested that the estimation of ozone was beyond the power of ordinary methods, he would like to know the method adopted to that end by Mr. Andreoli. Dr. Thorne had stated that in some cases there was reason to suppose that the whole of the oxygen in ozone was utilisable. That statement was interesting and important, and, if it was a fact, indicated a great advance towards the industrial application of this body. He was, however, anxious to learn the evidence on which Dr. Thorne had based his statement.

Dr. S. Rideal believed that there was a great field open to ozone in the future, but further experiments were required. He thought that Mr. Salamon had put the whole matter in a nutshell, and with his remarks he cordially agreed, excepting his sanguine opinion as to the applicability of ozone to vinegar-making. He thought it had a future as a disinfectant and germicide; but, apart from the reasons adduced by Dr. Squire with respect to its use in treating alcohol, it seemed to him that its application in that way was further prohibited by the fact that it would kill the specific organism whose action was necessary to the conversion of alcohol to acetic acid. With regard to the question of the solubility of ozone in water, some years ago a Mr. Fahrig had sent him a solution of ozone in salt water. The fact that it was soluble in the one liquid and not in the other possibly afforded an explanation of the action of electrolysed hypochlorites to which reference had been made. It would probably be too expensive to oxidise the whole of the organic matter in sewage, and he did not know that it was necessary. What they had to do was to destroy the pathogenic organisms in sewage, and it seemed to him quite possible to use ozone for that purpose after a cheaper oxidising agent had done its work. He was at present studying its behaviour on drying and other oils, and hoped to obtain useful results in that direction.

Mr. O. Guttmann observed that among the many uses of ozone contemplated by Mr. Andreoli, he had not included its use for the bleaching of beeswax and bones. Both of these were important industries to which he thought it might be applied, with the result of saving much of the time now taken up by the ordinary method.

Mr. A. Gordon Salamon wished to call attention to one other point to which Mr. Andreoli had not referred, namely, a most ingenious and valuable method of his for uniting glass piping and lead fittings, and which consisted in simply binding copper wire tightly round the glass tube, when it could be easily soldered to the lead in the ordinary way.

The Chairman understood that the results obtained by Mr. Andreoli's apparatus were a very marked improvement on the yield hitherto given by other apparatus.

Mr. Gabriel Andreoli, in replying on behalf of his father, said he would be most happy to add a complete bibliography of ozone to his paper if it would be desired by the Society. It had been objected that the paper was merely a list of possible applications. Quite true; but some of those applications had already been mentioned in Dr. Frölich's admirable paper; and if the learned German electrician was able to foresee a future for ozone and believe in the commercial applications, even with a lower yield of ozone than manufacturers were now able to obtain from the Andreoli apparatus, surely they were justified in mentioning them again, and also some other applications. They had been reproached also on the question of the cost of ozone; against that, he would point out that the ozone produced in their apparatus was cheaper than in all the other ones. It had been found that ozone would do what oxygen could not do; and if, as in the case of the oxidation of oils, for instance, they saved time by utilising ozone, that would more than compensate for the expense. It is certainly most regrettable not to be in a position to state what had been the results of commercial experiments, but it took time to induce manufacturers to try ozone, even when they had nothing to spend for the ozonisers, transformers, and even dynamos, which they might have at their disposal during one or two months for 1 to 5 or 10 h.p. installations. It has also been said that in the oxidation of wines and spirits, ozone would have a destructive as well as a beneficial effect, and that at a certain point one would get acetification; but there was no process which, unless carried out judiciously, would prove to be a success. No excess of ozone was required to mature wines, and it was out of question that ozone could be applied in that way. It must be understood that they never said a word about the purification of sewage by ozone, and that the sterilisation of water which had been mentioned in the present paper must be considered, not as a scheme, but simply as a highly

interesting fact which showed the germicidal action of ozone on the pathogenic and non-pathogenic bacteria which exist in water. Who would deny, for instance, the beneficial effects of sterilisation by ozone of the water for drinking purposes on board liners and also in the Colonies? The object of the paper really was to point out that ozone could be used industrially; and if the writer of this paper had only facilitated the getting together of detailed information on the production of ozone on a commercial scale, and thus drawing closer and more general attention to it, he would not have failed. As to the bleaching of beeswax and bones, he was sorry that he had not referred to it; he was quite aware that such use of ozone was practicable. As to the influence of points, it was a recognised fact that they facilitated the high tension discharge, and this did not require discussion. Some analyses referred to in the paper were made by Dr. L. T. Thorne, who was quite competent to defend them; the figures referring to 34·5 grms. and 40 grms. showed the results obtained by Prof. W. Ramsay.

Meeting held on Monday, February 15th, 1897.

DR. R. MESSEL IN THE CHAIR.

ELECTRO-DEPOSITION AND RECOVERY OF GOLD.

BY E. ANDREOLI.

The cyanide process cannot be a complete success without a satisfactory method of recovering gold from its solution. Zinc was the only medium used for separating the precious metal, until Messrs. Siemens and Halske set to work to apply the electrolytic method to the deposition of gold from its cyanide solution. The electro deposition of gold from its cyanide solution does not *prima facie* appear to be an operation which presents any great difficulty, and to the uninitiated, it comes to the same thing as electroplating. There is, however, a great difference between treating concentrated cyanide of potassium solutions carefully prepared and containing a large proportion of gold, the strength of which is kept up by a gold anode, at an electro-plating works, and electrolytically treating 100 to 150 tons of a very weak solution of cyanide containing a very small proportion of gold. In the former case, one has to coat metallic articles with a layer of gold of a certain thickness; in the latter, one has to practically exhaust the solution while it is passing through the tanks.

The treatment of 100 tons of solution per day of 24 hours represents a circulation of more than 4,166 litres per hour, or almost 70 litres per minute. A mode of circulation must be adopted which allows a constant and perfect contact of the electrolyte with the plates. Then the arrangement of the plates in the tanks must be such that they are not too far apart, and that the electro-motive force must be as low as possible, and therefore ensure economy of working.

Now, assuming that one has an electrolyte which is a good electrical conductor, that the electro-motive force is not too high, and that the best system for arranging the electrodes in the vats and for circulating the liquid between them at a rapid rate has been adopted, what material is to be used for the construction of the anodes? All the metals, without exception, dissolve at the positive pole in a cyanide of potassium solution; the best carbon will soon be attacked and disintegrated. Iron is, of all the metals, the only one which resists somewhat when acting as an anode in a cyanide of potassium solution; still, it dissolves. Complicated reactions take place, and the electrolyte is contaminated with all sorts of impurities.

It has been my good fortune to find an anode which is neither carbon nor metal, and which cannot be destroyed under the corrosive action taking place at the positive pole in a cyanide electrolyte, and which does not decompose or spoil the solution. I must say, however, that it is not a discovery resulting from my researches and studies, it is simply the result of chance. I was making experiments on what I call secondary electrolysis, when I noticed that, having put in the central compartment of a tank containing a gold and potassium cyanide solution, a piece of perforated lead close to the negative partition of the compartment, the lead, which in this case acted as an anode, had become brown. I soon ascertained that the plate was covered with a strongly adherent coating of peroxide of lead. But for this observation, I am sure I should never have thought of using this substance, which I had already unsuccessfully tried as an anode in a chlorine solution. There is therefore, no other merit attached to this discovery except my having noticed the formation of peroxide of lead, on a lead plate in the course of experiments on the deposition of gold, which were very satisfactory on a laboratory scale, but could not practically be carried out in the gold districts where the use of porous partitions is an impossibility.

I commenced then a long series of tests which all proved the excellence, the solidity, and the insolubility of these anodes when properly made, and I started electrolytic vats, each containing 30 large anodes. These plates were formed according to the Planté method, which means that they require a very long treatment; but at present I prefer to use a solution of plumbate of soda, in which they are rapidly coated with peroxide of lead, then withdrawn, washed, and placed in a strong cyanide of potassium solution, where, under a heavy current, they become hard and have a good crystalline appearance. The first thing I did when I had these anodes at my disposal was to use them to deposit gold on zinc plates. It was better and more rapid than the usual deposition on zinc shavings, but I did not consider it at all good; besides, in my mind, there was a great drawback to this electro-deposition, where each anode had to be placed between two cathodes. It can be realised how long it must take to disconnect, say, only 1,000 cathodes, and then to replace them by another 1,000 plates, and this is still worse if one has to withdraw the anodes and to put in fresh ones. What then if the plates have to be framed? I need not insist on this waste of time and labour, and to this must be added that, if the anodes are soluble, the liquid is spoilt and the tanks have to be cleaned. I may here point out that with my insoluble peroxide anodes the gold cyanide solution is as clear after as it was before treatment.

In my mind, the real problem of the electrolysis of a large volume of gold cyanide solution is to have as few plates as possible and a large surface on which to deposit the gold. I constructed a tank with two grooves cut in the bottom, which were filled with mercury. My cathodes were amalgamated perforated copper plates, standing in the mercury in the grooves, doing away with terminals and connecting bars. These cathodes were thus all in contact with the mercury, which was itself, by means of a copper strip, connected to the negative pole of the dynamo. Each plate could thus be withdrawn and replaced without the slightest trouble. There were in the tank about 50 perforated cathodes, forming an enormous active surface, and, instead of 51 anodes, I used only 5 to 6 peroxide of lead plates. The mercurial surface with which the gold solution had to be in constant, intimate, and perfect contact, under the action of the electric current, was enormous, but the quantity of mercury in each tank was very small, and the absorption of the gold by the amalgamated plate, which formed a sort of electric filter, was practically complete. Four of these tanks were capable of doing the work of eight ordinary tanks. This was a great advance, but I was soon disappointed when I was told that nobody in mining districts would care much for adopting such a process, which involved too much trouble in distilling the mercury and cleaning the plates in order to recover the gold amalgamated with it.

I firmly believe, however, in this method, which allowed me to rapidly and very efficiently exhaust the solution in a comparatively very small installation. I had, however, to find something else and something better which would suit the wants of mine managers. I commenced to deposit the gold on iron plates, which take gold much more readily than lead. Gold adheres better to iron and steel than to any other metal, and I was rather puzzled as to how to recover the gold from these cathodes, when it struck me that molten lead, which alloys so easily with gold in crucibles and

cupels, ought equally to absorb it from my gilded cathodes. I dipped a few pieces of iron coated with gold in a crucible containing molten lead, and after a very short time I had the satisfaction to withdraw them deprived of every particle of gold. This is how I recover the gold now. I deposit it on iron cathodes, from which I strip the gold by dipping them for about one minute in a bath of molten lead or bismuth kept at a proper temperature. An alloy is instantaneously formed which becomes richer and richer in gold, and which can easily and economically be cupelled, with the result that the bullion is absolutely pure.

To give you an idea of the extent of the application of electrolysis to the recovery of gold, I will quote the following lines, written in October 1896 by Mr. Charles Butters, Manager of the Central Rand Ore Reduction Company, who has started the Siemens and Halske process in the Transvaal:—

"We have been treating for the past two years 3,000 tons a month at the Worcester Mine. We have treated at this property about 70,000 tons of material, and the works are a complete success. This success was followed up by the introduction of the process into several other properties. We have been treating about 25,000 tons a month for several months past with most satisfactory results. There will be under treatment on the Witwatersrand by this process, about the beginning of next year, 100,000 tons a month of tailings and slimes. We have been treating at the Robinson Mine 6,000 tons of slimes per month, handling 800 tons of cyanide liquor per day, containing in the liquor to be precipitated from 4 dwts. a ton down to 15 and 18 grains, and this liquor is precipitated from 4 dwts. down to 8 or 9 grains, and from 18 grains down to 3 or 4 grains."

Liverpool Section.

UNIVERSITY COLLEGE, BROWNLOW STREET.

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Vice-Chairman: G. Schack-Sommer.

Committee:

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C. L. Higgins.	

Hon. Treasurer: W. P. Thompson.

Hon. Local Secretary:
T. Lewis Bailey, University College, Liverpool.

SESSION 1896–97.

Wednesday, March 3rd, 1897.—Dr. G. Schack-Sommer. "The Part played by Organisms in certain Chemical Industries; with special reference to Tanning, Brewing, and Wine Manufacture."

April 7th, 1897.—Election of Committee and Officers.

Meeting held on Wednesday, January 13th, 1897.

DR. C. A. KOHN IN THE CHAIR.

EFFICIENCY OF AN ELECTROLYTIC CELL.

BY F. HURTER, PH.D., AND B. ZAHORSKI, PH.D.

In a previous paper read before this Section by one of us, a formula was discussed which expressed the efficiency of an electrolytic cell, *i.e.*, that fraction of the energy consumed by the cell which was converted into the desired potential chemical energy.

That formula was—

$$\frac{a\,e}{V}$$

wherein e represents the so-called back electro-motive force corresponding to the products which are desired, whilst V represents the actual electro-motive force measured at the terminals of the cell. The factor a was not discussed in that paper at any length, and it is the purpose of the present paper to communicate the results of a number of investigations which have been made in the central laboratory of the United Alkali Co., part of which were communicated to the Liverpool Physical Society, but have not been published. Most of the investigations we are about to communicate have, however, been made since that paper was read.

The factor a in the above formula represents the fraction of the electro-chemical equivalent of the desired substance actually obtained per ampère.

Theoretically, we ought to obtain for every ampère of current one electro-chemical equivalent of the substance. In many carefully arranged electrolytic experiments the theoretical yield is actually obtained. In many practical processes of electrolysis the yield approaches closely to the theoretical, but in many other processes the yield per ampère is very much less than that calculated by Faraday's law. The theoretical yield is easily approached in all processes where the separated ions are deposited as solids or removed as gases; such is the case in the process of copper-refining, in a well-conducted electrolysis of zinc salts, &c., &c. But in such processes, where the products of the electrolysis remain in solution, where so-called secondary reactions take place, the newly-formed products take part in the conduction of the current; and, from the manufacturer's point of view, the current which produces other than the desired reaction is wasted.

The aim of our investigations is to trace quantitatively this "wasted" current, and to connect it with previously well-known phenomena in such a manner that the whole quantitative course of a given electrolytic process might be precalculated with a similar degree of accuracy as we now can precalculate the necessary terminal voltage for a given process.

We desire at once to state that it would be presumption on our part if we asserted that we have solved the problem, but we have at any rate succeeded in making a fair beginning in the science or precalculating, so to say, the crude outline of the quantitative course of the electrolytic changes, *i.e.*, the current efficiencies.

In order to prevent misunderstanding, we wish to present in the most elementary manner our methods of calculation, and to recall as concisely as possible the essential features of the phenomenon known as the migration of ions. There are no elementary English treatises on practical electrolysis which deal with the subject. Even a modern German treatise on the subject—*viz.*, "Handbuch der Electrochemie," by Ahrens (1896)—does not contain a chapter on the application of the phenomena of migration, and that word cannot be found in the index. There is not, to our knowledge, any scientific or technical treatise which indicates how the current efficiency might be estimated by means of the known velocities of ions, and this must be our excuse for troubling you with a few elementary remarks on the subject.

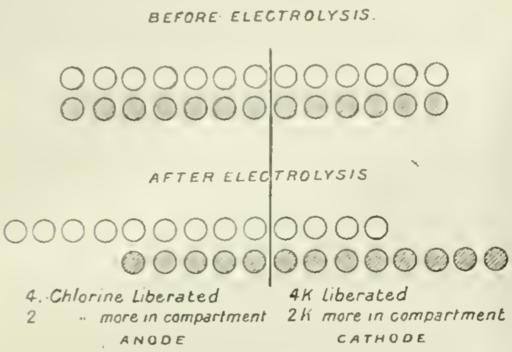
In order to fix our ideas, let us assume an electrolytic cell, in which we decompose potassium chloride. The instant we close the electric circuit, chlorine is evolved at the anode, hydrogen at the cathode; and we know by Faraday's law that after 100 ampères have been passed through the cell, there will be 100 (electro-chemical) equivalents* of chlorine liberated at the anode, and 100 equivalents of hydrogen at the cathode, corresponding to the decomposition of 100 equivalents of potassium chloride.

We are all acquainted with the explanation of the process as given by Grothus, *viz.*, the exchange and passage in opposite directions of the ions from one molecule to its neighbour. This explanation demands that after the passage of 100 ampère seconds through the cell there should be 50 equivalents of chlorine more on the anode side of the cell than there were before, and 50 equivalents more potassium

* Throughout this paper the term "equivalent" means an electro-chemical equivalent.

on the cathode side than were there before, including in the counting the free chlorine as well as the combined on the one side, and the potassium present as hydrate as well as in the form of chloride on the other side. This is usually represented by the following diagram:—

Diagram No. 1.



If the experiment be made with potassium chloride, it is found that the increase of chlorine and potassium in the respective compartments is very nearly 0·5 of the amount of the substance liberated, but if the experiment be made with sodium chloride, then the results are different, *viz.*, in a saturated solution we find an increase of 0·648 equivalents of chlorine in the anode compartment and 0·352 of sodium in the cathode compartment per equivalent liberated. It would appear as if, for 100 ampère seconds passed through the cell, 35 equivalents of salt had been decomposed at the anode, liberating there 35 of chlorine and causing 35 sodium to migrate towards the cathode, whilst 65 equivalents of salt had been decomposed at the cathode, liberating 65 of sodium and causing 65 of chlorine to migrate into the anode compartment.

Hittorf, to whom we are indebted for all the leading thoughts on which our work is based, has established, by an exhaustive series of experiments, that for each salt there is such a transport ratio, which is slightly affected by the concentration of the solution, but is not affected by the material or distance of the electrodes, nor by the material or number of diaphragms employed, nor by variation of temperature within certain narrow limits. To Hittorf is also due the explanation of the phenomenon by ascribing to the various ions different velocities. Thus, from Hittorf's numbers we find that if sodium travels with a velocity 1, then chlorine travels with a velocity $\frac{0·65}{0·35} = 1·85$.

In this respect the electrolysis of sodium chloride differs very materially from that of potassium chloride, owing to the fact that potassium and chlorine travel nearly with equal velocities, whilst sodium and chlorine travel at very different rates. The technical importance of this fact we shall refer to later.

Hittorf also foresaw that a connection must exist between the relative conductivities of the solutions and their transport ratios. But an accurate determination of the conductivity of a solution was almost an impossibility in Hittorf's time, and it was reserved for Kohlrausch to devise the elegant method for the determination of the conductivities of saline solutions which enabled this relation to be established. Kohlrausch's discoveries in this direction resulted in showing that the conductivity of a salt was the sum of two constituent parts, *viz.*, the conductivity of the anion and the conductivity of the cation, and that the same anion or cation had the same conductivity in a great number of its salts.

These relative conductivities were found to bear the same relation to each other as the transport ratios of Hittorf; they were, in fact, a different numerical expression of the same fundamental phenomenon—the different velocities of the ions.

Thanks to the efforts of Hittorf, Kohlrausch, Wiedemann, and many other observers, we are now in possession of a table of the relative conductivities of the various ions, by means of which we can replace lengthy tables of transport ratios for dilute solutions.

The following is such a table (Wiedemann):—

Table I.

Cations.		Anions.	
K	60	Cl	62
Na	40	J	63
Li	33	NO_3	58
NH_4	60	ClO_3	52
H	290	ClO_4	54
Ag	52	OH	165

The numbers given in the table may thus be used for calculations (1) of the molecular conductivities of dilute solutions.

Thus the molecular conductivity of NaHO is the sum of the numbers (Na = 40) + (OH = 165), or 205, whilst for a solution of sodium chloride of similar concentration the molecular conductivity is only 40 + 62 = 102.

Thus a dilute salt solution conducts only half as well as a solution of caustic soda.

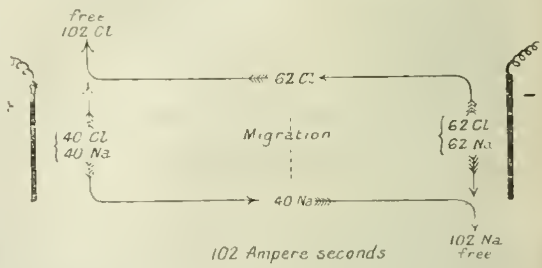
(2.) We can calculate from these numbers the transport ratios. Thus the transport ratios for sodium chloride are for—

$$\text{cation } \frac{40}{40 + 62} = 0·392; \text{ anion } \frac{62}{40 + 62} = 0·608.$$

It is thus very easy to represent for dilute solutions the quantitative results, not only with respect to Faraday's law, but also with respect to alterations in the concentrations and constitution of the solutions in the immediate neighbourhood of the electrodes after the passage of a given current, such that the constitution of the solution is not materially altered.

In order to show how this is best done, let us represent the electrolysis of a salt solution diagramatically by means of these numbers. We have, after the passage of a certain current (102 ampère seconds),—

Diagram No. 2.



40 NaCl were decomposed at the anode; the sodium of these migrates towards the cathode.

62 NaCl were decomposed at the cathode, the chlorine of which migrates towards the anode.

There are liberated 62 + 40 Cl at the anode; there are liberated 62 + 40 Na at the cathode; and since the current is measured by the ions liberated, the current passed through the cell is the sum of all the migrated ions, *viz.*, 62 + 40 (ampère seconds).

It is important to remember that the current can always be measured by the sum of the number of ions transported towards both electrodes.

When, however, the passage of the current produces a new electrolyte, as it does in the case of the electrolysis of potassium chloride or sodium chloride, then the question arises, How can we foretell approximately the further course of the electrolysis when the constitution of the electrolyte has been materially altered?

Let us again suppose a cell, divided by a diaphragm and containing a solution of common salt. Let a certain amount of current be passed through the solution, and, in consequence, let the cathode compartment contain a mixture of salt and

sodium hydrate. Since sodium hydrate is much the better conductor of the two, a great amount of the current will electrolyse sodium hydrate with reproduction of the same amount of sodium hydrate, and with a corresponding migration of hydroxyl ions into the anode compartment. Only a part of the current will electrolyse sodium chloride, with production of additional sodium hydrate in the cathode compartment, and with corresponding migration of chlorine ions from the cathode compartment to the anode. At the same time some sodium ions will migrate from the anode compartment into the cathode compartment and increase the amount of sodium hydrate there formed.

The increase in the number of equivalents of sodium hydrate in the cathode compartment is due to, first, the emigration of chlorine ions from the cathode compartment towards the anode, and secondly, to the immigration of sodium ions into the cathode compartment from the anode compartment. If we add these two numbers of newly formed equivalents of sodium hydrate and divide by the ampères which have caused this change, then we have calculated the current efficiency of that particular cell. We have thus reduced the problem of calculating the current efficiency to a problem in calculation of migration of ions.

In order that the principle of the calculation may be more clearly grasped, let us assume that, by reason of the given, but at present unknown, mixture of sodium hydrate and sodium chloride in the solution in the cathode compartment, there are 20 equivalents of hydroxyl emigrating together with 80 equivalents of chlorine from the cathode compartment. No new formation of sodium hydrate accompanies the emigration of the 20 equivalents of hydroxyl, but to the emigration of 80 equivalents of chlorine corresponds the formation of 80 equivalents of sodium hydrate. In addition to these 80, there will be a certain number of sodium ions migrating from the anode to the cathode. That number is easily calculated by a simple proportion, thanks to the principle of constant velocities of the ions.

We know that for 62 chlorine which migrate in one direction, 40 sodium migrate in the other; hence, for the 80 which we assumed to migrate in the above case, there will be immigration of—

$$\frac{80 \times 40}{62} = 51 \cdot 5 \text{ sodium.}$$

Similarly we know that for a migration of 165 hydroxyl ions there will be a contrary migration of 40 sodium ions, and consequently the migration of 20 hydroxyls involves a migration of—

$$\frac{20 \times 40}{165} = 4 \cdot 85 \text{ sodium ions.}$$

The total number of sodium ions newly formed, and immigrated into the cathode compartment, is therefore—

$$80 + 51 \cdot 5 + 4 \cdot 85 = 136 \cdot 35.$$

But we also know that the sum of all the migrations measures the current. Hence the value of the current is—

$$\underset{\text{Anion migration.}}{20 + 80} + \underset{\text{Cathion migration.}}{51 \cdot 5 + 4 \cdot 85} = 156 \cdot 35.$$

and the current efficiency in that case would be—

$$a = \frac{136 \cdot 35}{156 \cdot 35} = 0 \cdot 875.$$

We hope to have thus clearly explained the calculation of a current efficiency by means of the relative velocities of migration.

But in doing so we have assumed a relative migration of 20 hydroxyl ions and 80 of chlorine ions, and such an assumption is of no use except as an illustration.

To render the principle useful, we must be able to state how the current will divide itself in the cathode compartment between the sodium hydrate and the sodium chloride contained therein in known proportions.

This problem is readily solved for dilute solutions. In all such solutions the conductivity is simply the sum of the conductivities of the several ions they contain.

Thus, if a solution contain M molecules of sodium salts, of which x are sodium hydrate and $(M - x)$ are sodium chloride, then the conductivity would be—

$$102\,(M - x) + 205\,x,$$

and the current would divide itself between salt and caustic soda (if we disregard the influence of the electro-motive force of polarisation) in the ratio—

Salt.	Caustic Soda.
$102\,(M - x)$	$205\,x$

To the former current corresponds a migration of $62\,(M - x)$ chlorine and $40\,(M - x)$ sodium; to the latter current corresponds a migration of $165\,.\,x$ hydroxyls and $40\,.\,x$ sodium; hence the current efficiency is—

$$a = \frac{102\,M - 62\,x}{102\,M + (205 - 102)\,x}$$

This formula for the current efficiency is, however, only valid for a short moment, since on continuing to pass the current, x continually increases, and M also undergoes alterations. It is easy to change the above formula into a differential equation. If the current be C ampères, the amount of sodium hydrate produced will be in the time $d\,t$—

$$d\,x = a . C\,d\,t.$$

Substituting for a its value, and integrating, we obtain the efficiency for the whole current needed to produce x mols. of sodium hydrate in the cathode compartment, which at the beginning contained only salt.

Thus, for dilute solutions, the problem of calculating the current efficiency is reduced to a simple problem of calculating migration or conductivity phenomena.

But to the technologist the knowledge of the behaviour of very dilute solutions is of but small value. He is concerned as a rule with concentrated solutions, for which the simple laws above stated do no longer hold good, and the methods of calculation just explained can only furnish a hint towards an investigation of the problems of precalculating the current efficiency for any given cell filled with concentrated solutions.

The first question is, naturally, How do the transport ratios change with the concentration of the solutions? This has been ascertained by various investigations. We give in Table II. a few of the results for such salts as have a particular interest in connection with our experiments.

TABLE II.

Salt.	Volume Litres per Grm.-Molecule.	Transport Cation.	Observer.
KCl	0·36	0·484	Hittorf.
	0·49	0·484	"
	0·80	0·486	"
	2·93	0·485	"
	0·61	0·483	Weiske.
	1·73	0·486	"
	3·93	0·482	"
	0·36	0·484	Kuschel.
	2·94	0·485	"
KOH	1·20	0·261	"
	5·30	0·270	"
	9·70	0·258	"
NaCl	0·20	0·352	Hittorf.
	0·32	0·355	"
	1·21	0·366	"
NaOH	0·92	0·173	Kuschel.
	3·50	0·200	"
	9·20	0·157	"
Na_2SO_4	1·67	0·359	Hittorf.
	7·20	0·366	"
Na_2CO_3	0·35	0·470	"
	1·02	0·450	"
	5·30	0·480	"
	10·70	0·520	"

As regards the alteration of the transport ratios with temperature, but few observations are yet at our disposal. At ordinary temperatures there is but little change.

It has also been proved that the transport ratios are independent of the material of which diaphragms are made, porcelain giving the same result as tissues or parchment. They are also independent of the nature of the electrodes.

The net result of the investigations represented by the table is that, as far as transport ratios in concentrated solutions are concerned, they are sufficiently constant to permit their use for technical calculations.

The next point to consider is, How can we calculate the ratio in which a given current divides itself among different electrolytes in one concentrated solution?

For this purpose, let us imagine a trough divided lengthways by a partition, so that one half of the trough is filled with, say, sodium chloride solution, and the other half with, say, sodium hydrate solution. Let the ends of the trough be formed by two platinum plates, which serve as electrodes. Let a current be now sent through the trough, such that the plates show a terminal potential difference of V volts.

Let the electro-motive force needed for the decomposition of the salt be E_1 and the conductivity of the solution C_1; and, similarly, let the electro-motive force for decomposing sodium hydrate be E_2 and its conductivity C_2; then we have the currents A_1 and A_2 passing through the salt and caustic compartment respectively—

$$A_1 = C_1(V - E_1)$$
$$A_2 = C_2(V - E_2).$$

The total current is thus represented by—

$$A_1 + A_2 = C_1(V - E_1) + C_2(V - E_2);$$

he fraction travelling over salt is:—

$$\frac{C_1(V - E_1)}{C_1(V - E_1) + C_2(V - E_2)}$$

and if we consider this fraction of the current as the only useful one, then it also represents the current efficiency of the given arrangement. If the two solutions are such that they contain an equal number of molecules per litre, and the sectional areas of the two parts of the trough are M_1 and M_2, the number of molecules of the constituents will be in proportion M_1 and M_2, and the conductivities grow with the areas; hence we have the current efficiency—

$$\alpha = \frac{M_1C_1(V - E_1)}{M_1C_1(V - E_1) + M_2C_2(V - E_2)}$$

or—

$$\alpha = \frac{M_1}{M_1 + M_2\frac{C_2}{C_1}\left(\frac{V - E_2}{V - E_1}\right)}$$

If we now remove the partition and allow the solutions to mix, the formula will still hold good if the conductivity of the mixture is equal to the sum of the conductivities of its parts.

In that case the above formula represents the current efficiency with respect to the one constituent in an open cell containing a mixture of electrolytes.

It is readily seen that this efficiency becomes perfect, *i.e.*, equal to 1, only when the term—

$$M_2 \cdot \frac{C_2}{C_1} \cdot \frac{(V - E_2)}{(V - E_1)} = 0$$

vanishes. And this can only happen in two ways either if M_2 itself is 0, *i.e.*, if only one electrolyte is present; or if the difference $(V - E_2)$ becomes $= 0$, *i.e.*, if the terminal voltage can no longer decompose the substance M_2.

The current efficiency is zero, if the difference $(V - E_1)$ becomes zero, *i.e.*, if the terminal voltage is not great enough to decompose the substance M_1.

If the terminal voltage is increased, the fraction $\left(\frac{V - E_2}{V - E_1}\right)$ tends towards unity, and in that case the current efficiency tends towards a limiting value—

$$\alpha = \frac{M_1}{M_1 + M_2\frac{C_2}{C_1}}$$

The increase of the terminal voltage means a high current density, and the formula shows that if $E_1 > E_2$, a higher current density will increase the current efficiency. This explains why, for certain processes, a better energy efficiency is obtained with a higher voltage than a lower one, and confirms what has often been observed—that of two cells, the one using the lower terminal voltage is not necessarily the best.

This limiting value of the current efficiency is in most cases the only value that can at all be calculated, since in complicated processes we know but little of the real values of the electro-motive forces of polarisation. Consequently, in what follows we give for comparison with actual experiments only the limiting values of the calculated current efficiencies.

In order to test the applicability of these principles we made two solutions, one of potassium chloride and one of potassium hydrate, containing exactly 3 grm.-mols. of the substance per litre.

We estimated carefully the conductivity (by Kohlrausch's method) of the pure solutions of potassium chloride and potassium hydrate, and of mixtures of these at various temperatures. We give in Table III. the details.

TABLE III.

No.	Composition of Solution. Grms. per Litre. KCl.	KOH.	On 100 mols. of Electrolyte. KCl.	KOH.	Conductivity at Temperature (Centigrade).* 20°	30°	40°	50°	60°	70°	80°	90°
1	223·34	0	100	0	2·566	2·987	3·430	3·839	4·249	4·687	5·115	5·550
2	200·90	16·8	90	10	2·708	3·193	3·641	4·111	4·573	5·050	5·516	6·036
3	178·60	33·6	80	20	2·881	3·379	3·839	4·373	4·851	5·352	5·811	6·283
4	156·21	50·4	70	30	3·045	3·550	4·086	4·630	5·112	5·605	6·205	6·645
5	133·98	67·2	60	40	3·193	3·751	4·320	4·890	5·429	6·000	6·571	7·058
6	111·65	84·0	50	50	3·372	3·965	4·630	5·170	5·820	6·330	6·910	7·500
7	99·32	100·8	40	60	3·543	4·142	4·753	5·378	6·025	6·657	7·272	7·844
8	66·99	117·6	30	70	3·768	4·302	4·995	5·684	6·320	7·011	7·623	8·272
9	44·66	134·4	20	80	3·827	4·536	5·233	5·952	6·593	7·290	8·021	8·693
10	22·33	151·2	10	90	4·038	4·706	5·461	6·233	6·959	7·700	8·375	9·062
11	0	168·0	0	100	4·165	4·952	5·618	6·394	7·231	8·053	8·767	..

* Amperes per volt per litre.

The net result of the investigation is, that the conductivity (ampères passing per volt through one litre) for a solution of KCl containing 223·34 grms. KCl per litre (3 mols.) is, at the absolute temperature T,—

$$C_1 = 0{\cdot}04263\ T - 9{\cdot}924,$$

and that a KHO solution, 168 grms. per litre (3 mols.), is $C_2 = 0{\cdot}0767\ T - 18{\cdot}308$, and for mixtures containing x volumes of such a potassium chloride solution, and $1 - x$ volumes of a potassium hydrate solution, the conductivity at any absolute temperature T is—

$$C = (0{\cdot}0767 - 0{\cdot}0341\,x)\ T + 8{\cdot}384\,x - 18{\cdot}308;$$

that is, at any temperature T the conductivity of the mixture is the exact arithmetic mean between the conductivities of the two solutions at the same temperature.

If the conductivity of the 3-molecule potassium chloride solution is chosen as unit, that of a corresponding potassium hydrate solution at the same temperature is 1·623. The conductivity of a mixture of $(100 - x)$ volumes of the one with x volumes of the second is very nearly—

$$\frac{(100 - x) + x \times 1{\cdot}623}{100}$$

or, otherwise expressed, the conductivity of the mixture is very nearly the sum of the conductivities of its constituents; so that, on the principles we have just explained, the current efficiency for the decomposition of the potassium chloride in such a mixture in an open cell would be—

$$a = \frac{100 - x}{(100 - x) + x \times 1{\cdot}63} \frac{(V - E_1)}{(V - E_2)}$$

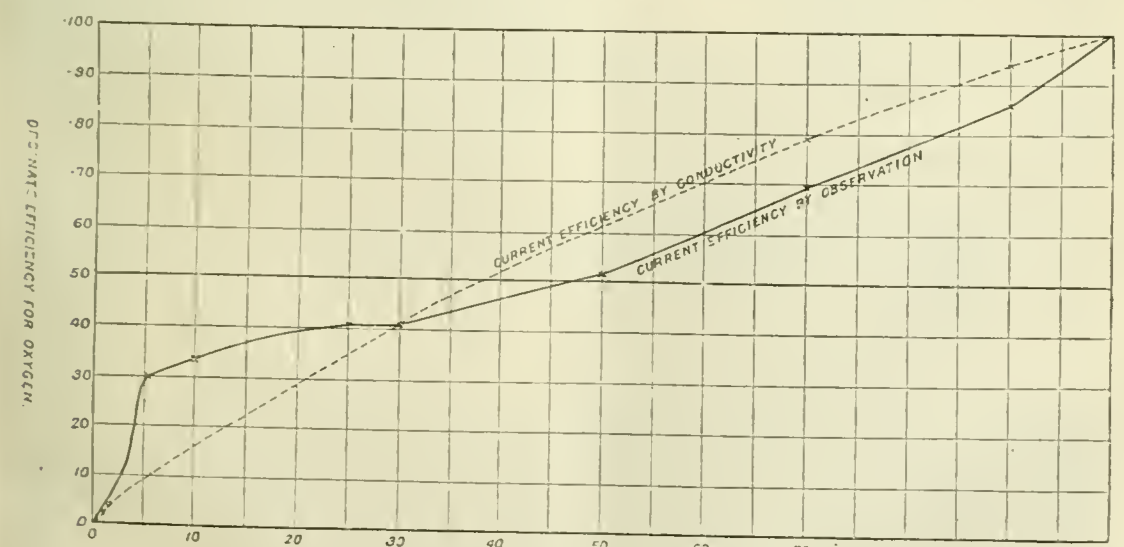
A number of various mixtures of these solutions were next subjected to electrolysis in a cell without diaphragm between platinum electrodes. The gaseous products of the electrolysis were measured and analysed. The volume of detonating gas evolved in a voltameter by the same current in the same time was also noted. From the given data we calculate the amount of oxygen which the current evolved in the cell and in the voltameter; this gives the current efficiency for oxygen for the given mixture of KCl and KOH solution; the difference between that and 100 is the chlorine efficiency.

Thus, on subjecting a mixture of 90 c.c. of the KCl solution and 10 c.c. of KOH solution to electrolysis, we found 11·4 c.c. of oxygen evolved, whilst in the voltameter 99 c.c. of detonating gas were found, equivalent to 33 c.c. of oxygen. Thus the current efficiency for oxygen is $\frac{11{\cdot}4}{33} = 34{\cdot}5$ per cent., and, by difference, the efficiency for chlorine is 65·5 per cent.

The Table IV. exhibits the details of these experiments and the efficiencies actually found, compared with the results of a calculation of the limit of current efficiency based upon the principle explained, *viz.*, that the current divides between the two salts in proportion to conductivity, disregarding the difference in electro-motive force.

Table IV.

Experiment.		Electrolyte.				Gaseous Products of Electrolysis.				Current Efficiency for		Calculated Current Efficiency for		Reduction at Cathode.
		Grms. per Litre.		C.c. of Solutions mixed.		In the Cell.			Voltameter.					
Series.	No.	KCl.	KOH.	KCl.	KOH.	Total.	H.	O.	H_2+O.	Cl.	O.	Cl.	O.	
c	1	220·70	1·68	99	1	66·6	65·6	1·0	99·9	97·00	3·00	98·3	1·6	1·50
b	2	219·50	3·36	98	2	67·8	66·6	1·4	105·2	96·10	3·90	96·8	3·2	5·10
c	3	211·80	8·40	95	5	75·8	65·6	10·2	100·0	69·40	30·60	92·1	7·9	1·50
b	1	211·80	8·40	95	5	75·8	66·0	9·8	100·8	70·90	29·10	92·1	7·9	1·70
c	4	200·70	16·80	90	10	77·2	65·8	11·4	99·0	65·50	34·50	84·7	14·8	0·30
a	1	200·70	16·80	90	10	77·4	66·2	11·2	99·8	66·30	33·70	84·7	14·8	0·50
b	2	200·70	16·80	90	10	81·0	69·2	11·8	104·8	69·19	33·81	84·7	14·8	0·85
a	2	156·21	50·40	70	30	80·0	66·2	13·8	100·0	58·10	41·60	58·9	41·1	0·60
b	3	156·21	50·40	70	30	80·0	66·5	13·5	100·4	59·67	40·33	58·9	41·1	0·62
a	3	111·65	84·00	50	50	86·0	67·2	18·8	100·6	44·10	55·90	38·1	61·9	0
b	4	111·65	84·00	50	50	83·4	67·0	16·4	100·8	51·20	48·80	38·1	61·9	0·23
a	4	66·99	117·60	30	70	91·8	67·2	24·6	100·0	26·90	73·10	20·8	79·2	0
b	5	66·99	117·60	30	70	95·0	71·4	23·6	107·2	33·96	66·04	20·8	79·2	0
a	5	22·33	151·20	10	90	97·2	67·2	30·0	100·6	10·70	89·30	6·4	93·6	0
b	6	22·33	151·20	10	90	94·0	65·6	27·4	100·0	17·80	82·20	6·4	93·6	0



ABSCISSAE cc of KHO solution in 100cc mixture
KHO solution 168 Grms per liter in
K Cl. 223·3 " " "

C

It will be seen that the limiting current efficiencies calculated from the conductivities do not agree with those actually obtained by experiment. This is strikingly exhibited by the diagram. The experiments were carefully made, but they are of course influenced by the formation of secondary products, particularly hypochlorite, the presence of which makes itself felt at the cathode, to which it only moves by diffusion and agitation.

This is shown in the column "Reduction at Cathode," which indicates the percentage of hydrogen which was not evolved in the cell owing to reduction of hypochlorite at the cathode.

The liquid surrounding the anode rapidly becomes richer in oxygen compounds than the given composition of the electrolyte indicates, since from 70 to 90 per cent. of the current produces chlorine, which at once is absorbed as hypochlorite. Thus we must expect the oxygen evolution to be greater than would be the case if a uniform composition of the solution could be maintained.

As Hittorf already pointed out, the way in which the current distributes itself between two electrolytes can only really be found by migration experiments, and such are very difficult to make and very tedious.

The result of these experiments teaches us that in an open cell the calculation of the current efficiency from the assumed conductivities is a higher limit for the production of chlorine or hypochlorites; that the current seems to electrolyse more readily the potassium hydrate than the calculation from conductivities would indicate.

In a similar manner the conductivities of mixtures of sodium chloride and sodium hydrate were determined by Mr. Auer. The net result of his measurements is given in Table V.

From those results it appears that very approximately the conductivity of the mixtures is again the arithmetical mean of the constituent parts. If the conductivity of the sodium chloride solution is taken as unit, that of the corresponding NaHO solution is 1·58.

The following experiments to ascertain the current efficiency for sodium hydrate with various mixtures of NaCl and NaHO solution were made:—

1. In a porous porcelain cell filled with a solution of sodium chloride containing 5 grm.-mols. per litre, a platinum anode was placed. A beaker was filled with the same solution, and a platinum cathode surrounding the porous pot was employed. The current was passed until the outside compartment contained 5 grms. NaHO per litre. The current efficiency varied from 0·91 to 0·93.

Table V.

Conductivities, 16°.

Molecules per Litre.		Specific		Temper.	C = Conductivity at 16°.	
NaCl.	NaHO.	Resistance.	Conductivity.		Observed.	Calculated.*
5	0	0·5226	1·913	15·5	1·940	1·940
4	1	0·4712	2·122	16·1	2·115	2·164
3	2	0·4376	2·285	15·6	2·313	2·388
2	3	0·3974	2·517	15·6	2·547	2·612
1	4	0·3536	2·828	15·6	2·862	2·820
0	5	0·3284	3·045	15·7	3·063	3·063

* $C = 0·388 (M - x) + 0·612 x$.

2. The same arrangement was used, but the cathode was surrounded with a solution containing 3·15 grm.-mols. of NaCl and 1·85 grm.-mols. of NaHO. The current efficiency was found to be from 0·369 to 0·416.

3. The same arrangement was used, but the solution in the cathode compartment contained 4·75 grm.-mols. of NaHO and only 0·25 grm.-mol. of NaCl per litre. The current efficiency was found to vary from 0·10 to 0·16.

In all these experiments the current was measured by a copper voltameter, and the increase in alkalinity of the solution ascertained, from which two data the current efficiency was calculated.

These experiments cannot be compared with the formula for the limit of current efficiency given for the open cell, where questions of migration do not play any part. In these experiments a diaphragm was used, and the electrolyte in the anode compartment differed from that in the cathode compartment. Hence the phenomena of migration must be taken notice of. This is done as follows:—

If the transport ratio for a concentrated solution (5 grm.-mols.) of sodium chloride is taken as Hittorf's figure = 0·352, then the velocity of the sodium ion is 0·35, that of chlorine 0·65. Since the relative conductivity of a 5-molecule sodium hydrate solution is by our measurements 1·58 times as great, the relative velocity of the hydroxyl ion must be—

$$1·58 - 0·35 = 1·23.$$

Hence the relative velocities of the ions in this concentrated solution may be taken as—

Na	0·35	1·00
Cl	0·65	1·86
HO	1·23	3·50

and the transport ratio for NaHO are 0·222 and 0·788.

In the cathode compartment the current divides for the limiting condition as—

$$M_1 : M_2 \frac{C_2}{C_1}, \textit{ i.e., } \text{as } M_1 : 1·58\ M_2$$

if M_1 and M_2 are the number of molecules of sodium chloride and sodium hydrate respectively.

The current M_1 produces M_1 ions of sodium, which count as new sodium hydrate. The decomposition of the sodium hydrate in the cathode compartment produces no new sodium hydrate, but to the emigration of hydroxyls corresponds an immigration of sodium. The amount of sodium hydrate thus brought into the cathode compartment is $0·222\ M_2 \frac{C_2}{C_1} = 1·58 \times 0·222 \times M_2$. Hence the current efficiency is, for sodium hydrate,—

$$a = \frac{M_1 + 0·350\ M_2}{M_1 + 1·58\ M_2}$$

Comparing this with the efficiency for the open cell *viz.*,—

$$a = \frac{M_1}{M_1 + 1·58\ M_2}$$

we see at once the beneficial influence of the diaphragm.

The following Table VI. compares the results of actual experiments with the efficiencies so calculated, and again indicates, what the previous experiments with potassium solutions have shown, that the calculated results are, higher limits for the production of soda and chlorine; that more current travels over NaHO than the calculation from conductivity would lead us to suppose.

Table VI.

Electrolyte Molecules per Litre in Cathode Compartment.		Efficiency.	
NaCl(M_1).	NaOH(M_2).	Actual.	Calculated.
5·00	0	0·91—0·93	1·000
3·15	1·85	0·37—0·42	0·640
0·25	4·75	0·10—0·16	0·209

Electrolyte in anode compartment: saturated solution of NaCl.

These results show also that if the cathode compartment contains only sodium hydrate, the current efficiency falls very low. It is well known that many inventors have constructed cells which should contain in the cathode compartment pure caustic soda solutions, and the investigation of the current efficiency of such cells has consequently commanded our attention at various times.

Some of our older experiments (made by Mr. Auer) may find a place here.

Into the anode compartment was placed a solution of common salt containing 312 grms. NaCl per litre. Into the cathode compartment were placed caustic soda solutions of various concentrations, the current being measured by a copper voltameter. The following Table VII. exhibits the results:—

Table VII.

Solution near Anode.	Solution near Cathode.	Observed Efficiency.
312·6 grms. NaCl per litre	80·0 grms. NaOH per litre	0·150
312·6 ,, ,,	4·0 ,, ,,	0·241
312·6 ,, ,,	1·0 ,, ,,	0·247
312·6 ,, ,,	4·0 ,, ,,	0·334
312·6 ,, ,,	2·0 ,, ,,	0·283
312·6 ,, ,,	0·8 ,, ,,	0·250
Mean		0·250

In this case the extreme efficiency would be theoretically simply the transport ratio for sodium in caustic soda, which for dilute solutions is, according to Kuschel, about 0·20.

It will be seen that the results are variable—some higher, some lower. Mr. Auer at that time thought the variability was due to the mode of filling the cell. If the diaphragm was moistened with salt the efficiency was good; if the caustic was first put in and the diaphragm soaked therewith the efficiency would be bad.

Two experiments which he made to test this point gave the following results:—

Electrolyte used.		Current Efficiency.
Anode Compartment.	Cathode Compartment.	
Concentrated NaCl solution, filled first.	NaHO solution, 80 grms. per litre, filled last.	0·43
Concentrated NaCl solution, filled last.	NaHO solution, 80 grms. per litre, filled first.	0·22

We therefore repeated these experiments, but under such conditions that the diaphragm was used for some time and got into a condition of stability as to the composition of the solution filling its pores, and then rapidly replaced the solutions one after the other, each operation being a quantitative experiment. Thus we found the following results:—

Table VIII.

Time.	Cu deposited in Voltameter.	NaOH formed.	Current Efficiency for NaOH.	Chlorine as NaClO and $NaClO_3$	Current Efficiency for Cl combined.
	Grms.	Grms.	Per Cent.	Grms.	Per Cent.
60	1·187	0·240	16·0	..	..
60	0·943	0·228	19·1	0·614	58·1
60	1·102	0·284	20·4	0·701	56·7
120	2·161	0·552	20·1	1·508	62·3
300	5·455	1·528	22·1	3·969	60·6

Porcelain diaphragm.

Electrolytes:—
Anode .... NaCl solution (saturated).
Cathode............. NaHO solution (normal).

It will be seen that after the stationary condition of the diaphragm had been reached the experiments gave fairly constant current efficiencies, which are nearly equal to the transport ratio = 0·20 (Kuschel).

This table also gives the amount of chlorine which was found in the anode compartment as hypochlorite and chlorate.

If the current efficiency in such a cell really depends upon the migration or transport ratio only, then a totally different result must be obtained if the cathode compartment be filled with another solution, the transport ratio of which is different. In order to test this, the cathode compartment was filled with Na_2CO_3 solution, the transport ratio for which is given by Kuschel as 0·47.

The following results were obtained, *viz.*:—

Table IX.

Time.	Cu deposited in Voltameter.	NaOH formed.	Current Efficiency.
	Grms.	Grms.	Per Cent.
60	1·189	1·080	72·3
60	1·212	0·744	48·5
60	1·147	0·720	50·0
60	0·580	0·368	50·1

Porcelain diaphragm.

Electrolyte:—
Anode .... NaCl solution (saturated).
Cathode............. Na_2CO_3 solution (normal).

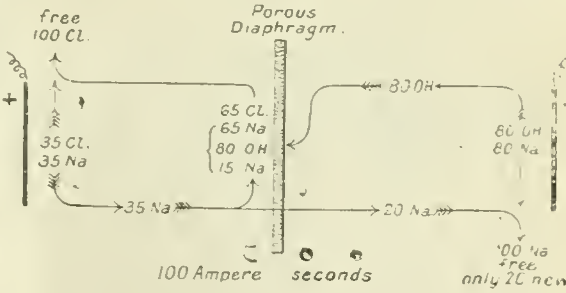
These show that after the diaphragm had assumed a stationary condition, the results obtained were about 0·5, clearly pointing to the transport ratio of carbonate of soda.

These experiments show that the same cell will give a vastly improved yield, if working for carbonate of soda than if working for caustic—a result which Mr. Hargreaves avails himself of, and which, he held, could not be explained by the migration theory.

So far, we have dealt only with the cathode compartment and the current efficiency referring thereto. We must now cast a glance at the anode compartment, and try to obtain an insight as to what happens at the other side of the diaphragm.

Let us again take the diagrammatic representation of the cell—the anode compartment assumed to be filled with salt solution, the cathode compartment with caustic soda solution. We will assume the cation transport ratios for salt 0·35, for caustic 0·20. We shall then have the following pictorial representation of what happens:—

Diagram No. 3.



At the cathode 80 NaOH are decomposed, sending 80 OH towards the anode compartment. In passing through the diaphragm they meet there 65 sodium ions liberated in the neighbourhood of the diaphragm by reason of the 65 chlorine migrating towards the anode, and another 35 sodium ions migrating from the anode towards the diaphragm. Of these 100 sodium ions liberated in the anode compartment, 20 pass through the diaphragm; the other 80 simply form NaHO with the 80 hydroxyls, and that in close proximity to the diaphragm. This caustic is not found in the anode compartment as such, but at once combines with some of the liberated chlorine to form hypochlorite and chlorate, as shown in Table VIII. This again participates in the conveyance of current, and thus matters become more complicated.

Mr. Auer made one experiment in which he attempted to measure all the products of the electrolysis. Into the anode compartment was first placed the concentrated salt solution, allowed to stand therein for 30 minutes, so that the salt solution might penetrate into the diaphragm; then the cathode compartment was filled with caustic soda solution. The results obtained are as follows.

Copper deposited in voltameter	3·320 grms.	
Soda obtained as Na_2O—		Per Cent.
in cathode compartment, Na_2O = 1·395 grms., or		43·0
in anode compartment as NaClO = 1·540 „		46·9
		89·9
Chlorine obtained—		
as gas ....1·7128 grms., or		46·2
as hypochlorite.................1·7395 „		46·9
Total Cl efficiency		93·1
Oxygen gas obtained ....0·0804 grms., or		4·8

To obtain the soda formed on the anode side of the diaphragm as alkali, and not as hypochlorite, we made a number of experiments with a cell containing three compartments separated by two diaphragms. The anode A was filled with a saturated solution of salt, the centre compartment B with a 4 per cent. solution of sulphate of soda, and the cathode compartment C with a normal caustic soda solution. We employed diaphragms of porous porcelain, and also of nitro-parchment and ordinary parchment. These latter are not good for experiments of this kind; they may contain pinholes, and they easily break. Thus no constant results can be obtained.

The following Table X. gives the results: —

TABLE X.

No.	Time.	Cu deposited in Voltameter.	NaOH formed. B.	NaOH formed. C.	Current Efficiency in Cathode.	Total Current Efficiency.
		Parchment Paper Diaphragm.				
		Grms.	Grms.	Grms.	Per Cent.	Per Cent.
1	75	1·1516	0·740	0·170	11·6	62·5
2	60	0·9280	0·700	0·096	8·1	67·9
3	90	1·9110	0·516	0·476	19·6	41·0
4	60	0·9750	0·488	0·392	31·7	71·3
5	60	0·9600	0·672	0·224	18·4	73·6
6	70	1·6240	0·658	0·280	21·6	72·2
7	64	1·0250	0·604	0·368	28·4	74·9
8	63	0·9810	0·524	0·464	37·3	79·6
		Porous Porcelain Diaphragm.				
9	60	1·0295	0·514	0·200	15·3	54·8
10	60	0·9010	0·556	0·204	17·8	66·6
11	60	0·9340	0·572	0·208	17·6	65·6
12	75	0·8600	0·512	0·184	16·9	63·9
13	60	0·8481	0·504	0·176	16·4	63·3
		Porous Porcelain Diaphragm, Current reversed.				
14	60	0·8970	0·072	1·076	94·8	101·1
15	60	0·9950	0·062	1·184	94·1	98·8
		Porous Porcelain Diaphragm, Partition B; NaCl Solution, Current reversed.				
16	60	1·0300	0·088	1·228	94·3	101·0
17	60	1·0653	0·100	1·264	93·8	101·2

These experiments do not show the whole of the soda made to be contained in compartments B and C, because some of it is contained in the anode compartment A and in the diaphragms.

We have recorded in the same table some experiments made in the same apparatus with the current reversed. In that case, if a diagram for the migration be made, it will be seen that all the NaHO must be found in the cathode compartments and *none* in any other compartment. The experiments fully confirm this, and thus furnish another proof that the current efficiency depends chiefly upon the phenomena of migration.

Our experiments show that in a cell which contains pure caustic soda in the cathode compartment a current efficiency amounting to 0·20 only can be obtained, and that the other 80 per cent. of soda are produced in the anode compartment, near the surface of contact of the two solutions.

If that surface of contact can by any means be brought into the cathode compartment, or nearer to the cathode side of the diaphragm, then some of the 80 per cent. of alkali made in the anode compartment may, by process of diffusion, pass into the cathode compartment, and the current efficiency will increase.

This seems to occur with very low current densities. In the cell of a French inventor, in which only 1 ampère passes per square decimetre of diaphragm surface, the current efficiency varied from 37 to 50 per cent., but the soda solution obtained was very dilute. In such a case ordinary diffusion will bring salt enough to the cathode side of the diaphragm to account for an improved current efficiency.

The cell patented by Hargreaves and Bird effects a material improvement in the efficiency by bringing the cathode in immediate contact with the diaphragm and washing the soda away as fast as it is formed.

We have added a few experiments with an apparatus embodying Hargreaves and Bird's principle of placing the cathode in contact with the diaphragm.

A platinum plate as anode was placed within a porous cylinder, which was filled with concentrated salt solution. The cylinder was closely wrapped with fine wire gauze, serving as cathode, and this was first submerged in a beaker full of cold water, by which means the following results were obtained :—

TABLE XI.

No.	Time.	Cu deposited in Voltameter.	NaOH formed.	Current efficiency.
		Grms.	Grms.	Per Cent.
1	60	1·1575	0·456	31·10
2	60	1·1870	0·608	40·05
3	60	1·2585	0·620	38·85
4	60	1·2375	0·608	38·80

Porous cell, wrapped with wire gauze, placed in cold water.

A very material improvement upon these efficiencies resulted from the employment of a beaker full of hot water (90° C.), as shown in Table XII.

When the cathode was surrounded by steam and washed from time to time with hot water, the results obtained were as shown in Experiment 1 to 3 of Table XIII. In Experiment 4 the cathode was only washed once at the end of the experiment.

TABLE XII.

No.	Time.	Cu deposited in Voltameter.	NaOH formed.	Current Efficiency.
		Grms.	Grms.	Per Cent.
1	60	1·180	1·488	99·7
2	60	1·212	1·404	91·3
3	60	1·147	1·216	83·7
4	30	0·580	0·620	84·6

Porous cell, wrapped with wire gauze, placed in hot water.

TABLE XIII.

No.	Time.	Cu deposited in Voltameter.	NaOH formed.	Current Efficiency.
		Grms.	Grms.	Per Cent.
1	60	1·3850	1·216	69·4
2	60	1·3720	1·318	75·9
3	60	1·3570	1·180	68·7
4*	60	1·2571	0·676	42·4

Porous cell, wrapped with wire gauze, surrounded by steam.
* Washed only once—at the end of the experiment.

In all cases the soda solution contains salt, and is very dilute. It is comparatively easy even for this case to show what strength of caustic solution can be obtained if the current efficiency is to be kept at a given limit. The result of such a calculation has already been published in our Journal.

The results we have now communicated show that, before any satisfactory calculation of the exact value of the current efficiency can be made, much more work must be done in the direction of ascertaining how a current divides between several electrolytes in one solution.

Our results leave no room for doubting that the current efficiency depends chiefly upon the phenomena of migration, or, what is practically the same thing, the conductivity of the solutions; and that, though no accurate calculations can be made at present, a higher limit can always be assigned which in practice is never reached.

Our results show that the phenomena of migration are capable of explaining why the electrolysis of potassium chloride is technically easier than that of sodium chloride; why potassium chlorate is easier to produce than sodium chlorate; why a cell with common salt on both sides of the diaphragms gives better efficiencies than one having pure salt on one side and caustic on the other; and why the production of carbonate of soda is accompanied by a higher current efficiency than the production of caustic soda.

We wish, finally, to thank the Directors of the United Alkali Company, Ltd., for their kind permission to publish our results.

Discussion.

Mr. Harry Baker said there were several points he was unable to understand, and which Dr. Hurter would doubtless be able to explain at once. In the first place, as to the literature dealing with electro-chemical matters, Dr. Hurter's remark as to some of the most recent works, particularly that of Ahren's, published only last year, was correct, but there was a book which he thought might be brought to the notice of the meeting, "Solutions and Electrolysis," by Mr. W. C. Whetham. That book, only published at the end of 1895, contained a summary of the theories of solution and of electrolysis, and gave many tables of data. At the end of the book there was a long list, some 60 pages of printed matter, of tables of conductivities and migration constants, and references.

In the paper brought before them was a table of the conductivities of ions (Table I.), and he would ask Dr. Hurter if, instead of designating that table "relative conductivities of the various ions," he should not have said that it was a table of the "absolute conductivities." Comparing that with the table of the absolute velocities of the ions which was given in Whetham's book, the significant figures were almost identical, but in the latter they are not given as whole numbers, but are multiplied by 10^{-5}, and then expressed in centimetres per second the velocities of the ions in infinitely dilute solutions under a potential gradient of 1 volt per centimetre at 18° C.

Thus they read in Dr. Hurter's table for the potassium ion a conductivity of 60, in Whetham's, a velocity of 0·00066 cm. per second. The conductivities of those dilute solutions varied considerably with the degree of dilution, and he presumed that Dr. Hurter had taken some convenient average strength of dilution and calculated a table for that. He would like to know what that strength was.

With reference to Table III., dealing with the conductivities, he presumed that the expression "ampères per volt per litre" meant the ampères that would pass from opposite faces of a cube, 1 decimetre in each edge, with a terminal pressure of 1 volt.

Dr. Hurter spoke of an "open cell," and he (Mr. Baker) interpreted that to mean a cell not provided with a diaphragm. Hittorf made use of closed cells, for the reason that if the cells were open, and not closed perfectly tight, and made with inflexible walls, there is generally a bodily transfer of liquid through the porous partition by reason of the electric endosmose.

Referring to the experiments which were described in Table IV., there was one point especially he wished to call attention to, and that was—the various estimations did not agree among themselves with any extraordinary degree of accuracy. Thus, for instance, taking the current efficiency for oxygen, they had under the same conditions 89·30 and 82·20; that was to say the error was nearly 10 per cent. of what they were estimating. It seemed to him that much of the discrepancy arose from the fact that at the electrodes they would have formed layers of liquids of differing composition and specific gravity; and any very trifling matter, as whether an electrode or a partition was exactly upright or horizontal, or slightly tilted over, would make a great deal of difference in the rate at which these layers would mix with the bulk of the electrolyte, and, as these layers may exert a profound influence on the progress of the electrolysis, no two experiments would give exactly the same result. As a remedy, he suggested that it would be well to keep the contents of the cells in a state of constant and violent agitation, either by rapidly moving the electrodes, as by small turbines, or by blowing in a current of air, or by circulating the electrolyte. Another method of getting over the difficulty would be to flow quantities of solutions through the electrolytic apparatus towards electrodes or partitions, as the case might be, at such rates that the products were swept away as fast as formed almost, and certainly at a rate greater than the opposite velocities of any of the ions. This might be simply done, and there would be but one disadvantage, *viz.*, large quantities of solution would have to be examined.

In the experiments described in Table VII. a higher efficiency was obtained than was calculable from the transport ratios of the ions, *viz.*, 0·20 to 0·22. It was very easy to obtain an efficiency which was less than theory, but remarkably difficult to obtain an efficiency greater than theory, and there must be some explanation to hand to explain why they should have those large efficiencies. The key to that he thought would be found in an analysis of the caustic soda, for there they would find a noticeable percentage of sodium chloride, the remnant of a still large quantity that had diffused through the diaphragm and by its electrolysis, given a higher efficiency. That remark would apply to a number of the subsequent observations. Thus in experiments made on the principle of the Hargreaves and Bird cell very high efficiencies were obtained, and also in Table X., they had efficiency in the cathode department with a parchment paper diaphragm very variable, but as high as 37 per cent., instead of the theoretical 20 to 22 per cent. In the last case it was specifically mentioned that parchment was very apt to contain pin holes and when Dr. Hurter used porous porcelain the efficiency only varied from 17 to 19 per cent., *i.e.*, just below the theoretical efficiency. It would have been very interesting if tests of the purity of the soda obtained had been given.

There was a point with regard to such experiments as the first few in Table IV. where the cathode is immersed in a limited volume of a solution containing a very small but measured quantity of caustic potash; during the process of the experiment on that weak solution of caustic potash, further amounts of potash are formed, that may be very considerable in comparison with the original amount, and therefore have a considerable disturbing effect on the result of the experiment. Apparently such influences could be traced in the experiments referred to. In many electrical measurements, and in such electrolytic experiments as those named very particular care had to be taken that the test should not alter the thing to be tested, and the remedy here would be to have a constant flow of fresh electrolyte through the apparatus and to aid the removal of the product of electrolysis by agitation of the electrode on the electrolyte.

Attention was drawn to the important remark that "if that surface of contact can by any means be brought into the cathode department, or nearer to the cathode side of the diaphragm, then some of the 80 per cent. of alkali made in the anode compartment may, by process of diffusion, pass into the cathode compartment, and the current efficiency will increase." Dr. Hurter had explained that those pro-

cesses had an efficiency of only 20 per cent. and now here apparently was the loophole through which they could drag the remaining 80 per cent. But if they took into consideration the absolute velocity of the ions, they would be able to calculate readily the speed at which the liquor from the anode compartment must travel through the porous diaphragm into the cathode compartment, and would find that for caustic soda they would be able to get an efficiency of 100 per cent. and pure caustic into the bargain, but only if the cell is fed with lime containing about 1·5 per cent. of salt, and the resultant soda solution is proportionately weak, and that it was impossible to get a strong solution of even approximately pure caustic soda by any means of driving the liquid from the anode compartment into the cathode compartment through the porous partition.

Mr. A. Hay said that with regard to the two curves given on page 101, one of which was theoretical, and the other experimental, it was pretty plain he thought that the great divergence between the two was mainly due to the fact of the counter E.M.F.'s not having been taken into account. That fact would be rather difficult to consider satisfactorily; the counter E.M.F. would depend not merely on the concentration of the solution, but would also depend on the area of the electrodes, so altogether the effect would be a very complicated one. There appeared to be one point common to the two curves; at that point, therefore the results agreed. Higher up, however, there was another point which appeared to be common to the two. Had it been actually obtained by experiment?

Dr. Hurter replied that it was simply put as the beginning and termination of the curves.

Mr. Hay drew attention to another point—he thought a trifling one—in several passages Dr. Hurter spoke of the electrical equivalents due to certain currents passing through the solution. He (Mr. Hay) would like to suggest the term "coulombs" for "ampères" as it would render the terminology somewhat more precise. With regard to the formation of layers of different density, it may be remarked that so long as the layers were arranged in a direction which was parallel to the current, it did not matter, so far as calculation went, whether the liquid was differentiated into such layers or not. But when they had two layers arranged in a direction which was normal to the current as would be the case with a cell having horizontal electrodes, then the conductivity could not be calculated in the simple manner suggested by Dr. Hurter, from the conductivities of the constituents.

Mr. J. B. C. Kershaw said, with regard to the theoretical aspect of Dr. Hurter's paper, I think it is to be regretted that he has not in any way alluded to the recent correspondence in the columns of "Nature," upon the vexed question of "Dissociation" and the presence of "free ions" in an electrolyte. Dr. Hurter's paper is based upon the theory of "ionic migrations," and it is questionable whether this latter theory is so wholly independent of the former as to justify Dr. Hurter in completely ignoring the controversy. If the theory of "free ions" is found to be out of harmony with many facts of chemical change it will have to be relegated to the lumber room for "false theories"; and it is questionable whether the theory of "ionic migration" could then be maintained. Dr. Hurter's experiments, recorded in the present paper, may be urged by himself and others, as supporting the theory, since his calculated and observed current efficiencies agree fairly well.

But in the diagramatic representation of the experiments with mixed potassium chloride and potassium hydrate solutions recorded in Table IV., there are considerable deviations from the calculated efficiencies; and as Mr. Baker has already pointed out, there are very marked discrepancies in the six last experiments entered in Table IV., each two of which were performed under precisely similar conditions. I do not think, therefore, that Dr. Hurter would care to base his support of the "ionic migration;" theory upon experiments, which not only show a marked deviation from the calculated results, but also differ widely, in some cases, amongst themselves.

Turning to the practical aspect of the paper, I wish first to draw Dr. Hurter's attention to the statement that a higher E.M.F. sometimes increases the current efficiency, and that consequently a better *energy* efficiency is obtained. Dr. Hurter, in a paper read in 1895, gave the expression $\frac{a\,e}{V}$ as representing the energy efficiency for any electrolytic cell. It is evident then, that any increase in V, occasions loss of energy, and that a very considerable gain in current efficiency is requisite to balance it, if the energy efficiency is even to remain the same, not to speak of a gain.

To take a practical illustration. Say a cell working at 2 volts pressure, shows a 30 per cent. current efficiency. If the voltage is increased only to 6 the current efficiency must spring up to 90 per cent. to maintain merely the same *energy* efficiency.

It is evident, therefore, that there is quickly a limit to the possibility of a balance in this way, and that the case in which an absolute gain in energy efficiency occurs is almost excluded.

This present paper contains experiments that show a current efficiency of 20 to 40 per cent. is the maximum that may be expected, when electrolysing solutions of sodium chloride, under conditions which may be surmised to obtain in actual manufacturing operations. The inference of course is, that a similar current efficiency, or a lower one, will be obtained in operation on a larger scale, and that the commercial success of such a manufacture will be impossible. This inference is, I believe, wrong.

It is a very common fallacy to suppose that a 90 per cent. current efficiency must be obtained in order to give an electrolytic process the slightest chances of success. Mr. Swinburne, in his recent Cantor Lectures on Commercial Electrolysis, pointed out that this was a mistake, and some recent calculations I have made relating to these processes prove that his views are correct.

I find that power, in most of these estimates of the cost of electrolytic alkali and chlorine compounds, only represents from 30 to 35 per cent. of the total cost, and that even supposing the current efficiency be only half that usually calculated upon, or even as bad as Dr. Hurter's experiments would seem to show, this increase would only bring up the proportion of power cost to between 50 and 60 per cent. of the whole, and there is still a fair margin left, in spite of the increased cost of power, for manufacture at a profit.

Dr. Hurter in replying, referred, first of all, to Mr. Baker's remarks as to how far migration was treated of in text books. The reason for the sentence in his paper was an adverse criticism of a paper he had read before the Liverpool Physical Society. He there referred to the fact that migration was not very well known, and that was his excuse for treating it in an elementary manner. He was perfectly well aware that scientific treatises and scientific men knew all about migration, but he was equally well aware that the ordinary inventor did not know, and that no treatise on practical electrolysis dealt with the subject otherwise than superficially. As regards the application which he had made of the facts of migration, he did not think that they would find any indication of it elsewhere than in the paper he had the honour to bring before them.

With respect to Table I., the numbers there given were not "velocities" but were conductivities, which, when multiplied by 10^{-8}, referred to the conductivity of mercury as unit. It so happened that they appeared to be multiples of the absolute velocities in centimetres per second. These conductivities were referred to infinite dilution of the electrolyte.

With respect to Table III., Mr. Baker had properly interpreted the meaning of conductivity. It gave the conductivity as the number of ampères which would pass, per 1 volt difference of potential, between opposite sides of a cube of 1 decimeter. Similarly he had been properly understood that an open cell meant one in which the current could go from electrode to electrode through the liquid, without having to pass a diaphragm. With reference to the experiments in Table IV., they had always found great difficulty in obtaining absolutely concordant results on repetition. No doubt agitation or circulation of the liquid would assist, but it was difficult to employ these devices, and at the same time insure a correct gas analysis. The experiment was arranged that the electrolysis should

not affect seriously the constitution of the electrolyte by having a large cell with much more than 100 c.c. of liquid in it. Moreover, Mr. Baker would see that it was not the liquid with the smallest amount of KOH which deviated most, but one with 8·41 grms. per litre. Also the three series of experiments *a*, *b*, and *c* were made at very different periods, and after they had learned that a potassium hydrate must be purified by electrolysis. As no effort made so far had brought the results much nearer the theoretical curve, they had given all the results they had in their possession.

The variations in Table VII. to which Mr. Baker referred were no doubt due to salt getting into the cathode compartment. Dr. Hurter had given that as the explanation in his paper to the Physical Society, but since Mr. Auer's experiments bore out Mr. Auer's suggested reason for the discrepancies they had given his agruments due weight.

In reply to Mr. Hay, he quite agreed with his remarks that it was difficult to calculate the electro-motive force of polarisation in such cells, and that was the reason he had omitted that correction in all his calculations as explained in the paper. He had used in a former paper the word "coulombs," but he found that that term was not understood by many readers. As to the calculation of the composition density and velocity of exchange of the layers of solution in contact with the anode and cathode he did not see his way out of that difficulty.

In reply to Mr. Kershaw, any difficulties with respect to the theory of migration or dissociation did not in the least affect his results or the technical value of them. They often used the words "theoretical" and "theory" when they ought to use the words "experimental" and "experience." When they spoke of the theoretical composition of a chemical compound, they really meant the composition as found by such skilled men as Berzelius, Rose, Liebig, Stas, &c. The transport ratios were facts well established, and so were the conductivities. Upon those facts the calculations and experiments of the paper before them were based, and whatever might happen to the theory of migration or dissociation these facts would remain.

As regards energy efficiency, the formulæ he had given in the present paper gave some sort of a key to what was difficult to explain, that in some cells a considerable increase in the terminal voltage resulted in a better current efficiency, so that more produce was obtained per horse-power day.

The subject was by no means clear, and he hoped the paper would be a stimulus for more work and thought in the same direction; much more light was still wanted.

Manchester Section.

Rooms of the Chemical Club, Victoria Hotel.

Chairman: George E. Davis.

Vice-Chairman: Peter Hart.

Committee:

F. H. Bowman.	J. M. Irving.
R. Forbes Carpenter.	M. J. Langdon.
R. S. Dale.	J. Lewkowitsch.
R. Le Neve Foster.	E. Schunck.
B. W. Gerland.	W. Thomson.
H. Grimshaw.	D. Watson.
J. Grossmann.	

Hon. Local Secretary:

J. Carter Bell,

Bank House, The Cliff, Higher Broughton, Manchester.

SESSION 1896–97.

March 5th, 1897:—

Dr. A. Liebmann.—"Paranitraniline Red."

Mr. Wilfrid Irwin.—"The Acetylene Theory of Luminosity."

Meeting held on Friday, February 5th, 1897.

DR. F. H. BOWMAN IN THE CHAIR.

LABORATORY NOTES.

BY W. F. KEATING STOCK.

Extraction of Soluble Phosphates from Artificial Fertilisers in the Process of Analysis.

It is a debateable point whether the treatment of a weighed quantity of any phosphate, which has been acted upon by and still contains free sulphuric acid, with successive small quantities of water, is quite a fair way of extracting the soluble phosphates for analysis. When the phosphate was finely pulverised like superphosphates I have never noticed any appreciable difference in the results whether this was done, or whether the whole quantity of water was added at once, and the extraction made by the method about to be described. But when the phosphates to be examined consisted of vitriolised bones or bone manures containing coarse fragments of even ¾ in. in diameter then the treatment of even 5 grms. by any of the usual methods has failed to give me sufficiently satisfactory results, and I prefer the following method which I have used now for some years:—

From 20 to 30 grms. of the fertilizer, carefully mixed and free from adherent lumps, are put into a strong bottle, and from 2 to 3 litres of water, carefully measured, are added at once, and the mixture thoroughly shaken immediately. 20 or 30 ordinary glass marbles are now dropped into the bottle, which is then closed with an indiarubber stopper, and the disintegration is effected by repeated swinging of the bottle to and fro in a horizontal position during two or three hours. The liquid is then allowed to clear by subsidence. An aliquot portion is drawn off, when clear, and the soluble phosphoric acid is determined. If the liquid clears too slowly it may be filtered through a double dry filter, rejecting the first 50 c.c. or so.

Improved Copper Zinc Couple for the Determination of Nitric Nitrogen in Water Analysis.

A 7-oz. wide-mouthed stoppered bottle is filled nearly to the neck with granulated zinc. Water is added, then a few drops of 1 to 3 of sulphuric acid and 10 c.c. of 3 per cent. solution of copper sulphate. The stopper is inserted, and the bottle is vigorously shaken for one minute, during which time the stopper is held by a finger, and the operation is performed over the sink. The stopper is now removed, and the mouth of the bottle is covered with a piece of soft copper gauze. The couple is then thoroughly washed at the tap and drained. 100 c.c. of the water to be analysed are placed in the bottle; the stopper is securely inserted and the arrangement is allowed to stand at rest at a temperature of from 20 to 25° C. for 48 hours. The test is completed by thoroughly shaking the bottle, drawing off 50 c.c. of the water, adding this to 200 c.c. of ammonia free water in a retort or flask, running in 5 c.c. saturated sodium carbonate, distilling and nesslerising as usual. This process has been found correct between the limits of 0·0863 and 4·181 grains of nitric nitrogen per gallon, when pure potassium nitrate was used in solution in ammonia-free distilled water. The couple when washed and recoppered is again ready for use. These couples will last for many months, and their convenience will be obvious to anyone who has had to clean and prepare 20 or 30 slips of zinc foil at one operation. It will be well to add that *all* new stoppered bottles intended for this purpose should have their stoppers carefully reground into the necks with a little fine emery and dilute sulphuric acid. And I may also say that dilute sulphuric acid is an excellent agent to employ with emery for grinding glass, being far superior to turpentine or anything else I have tried (see Chem. Soc. Jour. 1881, Trans. 100).

A New Extraction Apparatus in which the Solvent acts at Boiling Point.

The apparatus exhibited has been in use in my laboratory for three or four years. It consists of (1) a boiling flask

of 150 c.c. capacity, (2) a glass tube which acts simultaneously as a bye-pass for the vapours of the solvent and a support for (3), another tube which contains the substance to be extracted. The bye-pass tube is 12 cm. long and 2·5 cm. internal diameter. It is shaped like a test-tube. At the bottom it has a circular aperture of 5 mm. diameter, and at one side, 4 cm. from the bottom, another circular aperture of 7 mm. diameter. It is fitted to the neck of the flask by means of a sound cork. The substance to be exhausted is contained in another tube, which slides easily into the bye-pass tube, and it is provided with a short stem which protrudes through the aperture at the bottom of the bye-pass tube. The sample rests upon a filter made by putting a plug of cotton-wool into the short stem of the containing tube, and then pouring a little filter paper pulp on to this, after which alcohol and ether are passed through to free the filter from water and extractive matter. The top of the bye-pass tube is connected to an upright condenser by means of a good cork, and the boiling-flask is heated either by water-bath or sand-bath. I prefer the latter even with ether as the solvent. The apparatus before you will exhaust 10 grms. of cotton cake and uses only 50 c.c. of solvent.

Apparatus for ascertaining the Softening Point of Coal Tar Pitch.

This apparatus consists of a copper vessel A, of cylindrical form, 13 cm. high and 12 cm. wide. It is fitted with three little copper brackets fixed inside the vessel 1 cm. from the top, and three similar brackets 7 cm. from the top. These brackets are arranged at equidistant points around the circumference of the vessel, the lower three being fixed so as to lie in the intervals between the upper three. The object of these brackets is to carry a circular perforated zinc platform, B, fitting easily into the copper vessel. This has three semi-circular gaps cut out of its circumference corresponding in position to the brackets above mentioned. This zinc platform has an aperture in the centre 1 cm. in diameter, and round this is soldered a perforated zinc tube 5 cm. high. At opposite sides of this tube are soldered two stoutish brass wires 14 cm. long, and these carry a little copper disc having a 5 mm. hole bored through it centrally. This fitting acts as support for the necessary thermometer, which is fixed in the perforated tube by means of an ordinary cork. The thermometer is set so that the zinc platform is just midway of the bulb. The use of the apparatus is to obviate the necessity for strings or wires to support the test pieces of pitch. The copper vessel is filled with water at the required temperature. It is set on an iron plate over a suitable burner. The zinc platform is lowered on to the bottom set of brackets, and left there till the temperature is steady. It is then raised, and a half revolution allows it to rest on the upper brackets. The test pieces are then laid upon it, four at a time. It is reversed a half turn, and lowered again. It now rests upon the lower brackets, and the test commences. At the end of the time agreed upon by the contract note, the platform is raised, a half turn is given, and it rests upon the upper brackets. The test pieces are then twisted one after the other without loss of time. The thermometer acts as a handle by which to raise, lower, and turn the platform. The gain in time and uniformity of results is very great when this apparatus is used as compared with tying or wiring of the test strips.

INDIGOTIN AND NITROBENZOL.

BY DR. B. W. GERLAND.

An examination of the behaviour of indigotin with nitrobenzol has become necessary in the interests of the method for the determination of indigotin described by me in this Journal, 1896, 16. The preparation of pure nitrobenzol causes great difficulties, while the commercial article is of a high degree of purity but still contains a small amount of brown tarry matter, which is only partially left behind by repeated distillations and blowing-over with steam. Sulphuric acid acts more energetically upon the impurity than upon the nitrobenzol, but even an acid reduced to 30 per cent. gives unpleasant results: the nitrobenzol separates badly, and when distilled or blown over by steam, has a deep yellow colour after washing with caustic soda. Trials with mild reducing agents or with permanganate or chromic acid in acid or alkaline solutions proved equally unsatisfactory. The best results I obtained by repeated crystallisation. The nitrobenzol thus purified has a very pale colour and freezing point of 7° (thermometer certified by the Berlin Reichsanstalt). These impurities may be products of decomposition of the nitrobenzol; they do not affect the results of indigo tests unless the quantity of the indigotin fall as low as a few decimilligrammes.

Nitrobenzol dissolves indigotin very slowly in the cold. On warming the action takes place readily, the solution is pure blue and soon becomes of great depth. With the temperature rising to about 65° red becomes mixed with blue, and at about 95° the solution is pure blood red, and this increases in brilliancy with the further rise of temperature. The cooling solution does not change back to blue at once; the red remains for hours and if cooled to about 2° pale blue crystals appear in the red liquid, which at from 1 to 2° turns blue again. Contact with a fresh glass surface immediately effects the change from red to blue; a pipette for instance dipped in the cooled red solution can only be charged with a blue solution. Weak solutions on the other hand do not alter their blue colour on heating.

It is difficult to ascertain the solubility of indigotin in nitrobenzol at the boiling point. The figures obtained vary from 0·5 to 1 grm. in 100 c.c. With the experience recently gained I hope to succeed better. The solution has a remarkable tendency to supersaturation, which is the more persistent the less the excess of indigotin it contains. It may require days, weeks, and even months before the normal condition is reached, and weeks even with a small amount of crystals present. This peculiarity has to be taken into consideration in accurate estimations of indigotin, the more so, the smaller its proportion to nitrobenzol, and should be counteracted by long standing with frequent shaking. In ordinary indigo testing abundance of indigotin crystals are present which expedite the separation, so that after a few hours' standing nearly all the indigotin has separated. Still the quantity remaining in solution may be three or four times that which would be left after some days.

Attempts to estimate the indigotin left in solution in nitrobenzol in the cold by chemical methods were not successful From 25 c.c. of such a solution (the quantity I usually employ), evaporated on the water-bath, a residue weighing 0·0021 grm. was obtained; another solution gave the same result. These residues were of a dirty colour and with careful manipulation only would give the indigo reaction. In several trials the nitrobenzol was blown over by steam, care being taken to remove the last trace, but the residue was more contaminated, and I despaired of arriving at approximate results even by the sulphonic acid described (*loc. cit.*). Presence of hydrochloric acid whilst steaming did not improve matters. But colorimetrically the estimation is easily done withe every appearance of reliability, if the tendency to form supersaturated solutions be taken advantage of.

0·0090 grm. indigotin were heated with 50 c.c. nitrobenzol; directly after cooling, before the red had changed to blue and crystals had separated, measured quantities were added to nitrobenzol until the shades were matched.

1 c.c. of the solution in 45 c.c. nitrobenzol was equal to the normal solution.

1 c.c. in 20 c.c. nitrobenzol was equal to the solution of indigotin crystals two hours after cooling.

Therefore—

	Grm. Indigotin.
25 c.c. of the normal solution contain	0·0001
25 c.c. of the lead supersaturated solution contain..	0·000225

The total neglect of the amount of indigotin left in the solvent would accordingly have no appreciable effect upon the results of an indigo test.

I have taken all the more interest in these estimations as my colleagues, who had adopted this method, arrived indirectly at figures of incredible height, neglect of which would vitiate the results.

My former recommendation of a saturated solution of indigotin in nitrobenzol for indigo testing may, therefore, stand, and the loss of solvent during the operation, amounting to from 2 to 5 c.c., need not be made good; it will compensate the larger amount of colour left in the supersaturated solution.

The nitrobenzol solution is rapidly bleached when exposed to light but it keeps indefinitely in the dark.

Testing Indigos.

The apparatus I described has rendered good service and has not been altered, except by the glass tube, which acts as condenser and connects the test tube of boiling nitrobenzol with the aspirator, being replaced by a brass one, which has the advantage of not breaking off and thereby endangering the trial when drops of water fall inside.

The operation should be started with well dried apparatus and materials, the crystals of indigotin collected on a small filter, extracted with acid and alcohol, dried and weighed, washed with benzol, digested with strong hydrochloric acid, and boiled with addition of water, then boiled with solution of sodium carbonate, washed, dried, and weighed. Subsequent washing with cold alcohol decreases the weight by about 0·1 for 100 indigotin. The latter still contains ash to about 0·1 per cent., which I suspect to be derived from the glass vessels. With careful working concordant results are obtained, and even with spurious indigos the true amount of indigo is found. Thus "refined indigo of 98 per cent." yielded: moisture 1·30, nitrobenzol extract 74 per cent., but in the same only 24·31 per cent. indigotin.

My expectation that the results by this method would be lower than those by the permanganate one, especially for poor indigos, has not been borne out by experience. The results are sometimes higher and sometimes lower without any regularity.

Estimation of Indigotin on Dyed Cotton and Wool.

The method has served excellently for the determination of indigotin on dyed goods, but it has to be adapted to the purpose. Indigo-dyed cotton in the vapours of boiling nitrobenzol rapidly loses the greater part of its indigotin and becomes dirty white, but no amount of boiling will make it quite white. Exposure to air again turns the cotton blue and nitrobenzol will again extract some indigo, but with numerous repetitions I succeeded in removing only about $\frac{3}{4}$ of the total amount. Then it takes weeks for the colour to develop. But if the cotton be treated with boiling acetic acid, complete extraction rapidly takes place and the cotton becomes pure white. It is, however, preferable for the estimation of indigotin to destroy the fibre (both wool and cotton) by treatment with acid, and extract the residue after filtering, washing, and drying, with nitrobenzol. The results of parallel tests show an agreement, not surpassed by inorganic analysis.

Respecting the second method of estimating indigotin, by titration of the sulphonic acid with hydrosulphite according to Muller-Bernthsen, I have collected some experience. I have also investigated the indigotin-sulphonic acid, and shall have much pleasure in communicating the results on a future occasion.

Discussion.

Dr. Grossmann said that the fact of a number of tests agreeing to a second decimal was not, in his opinion, a proof that the method must be, under all circumstances, correct. In all cases of indigo, samples of different strengths should be prepared by mixing chemically pure indigotin with red, brown, glutens, and ash substances in different proportions, and these different mixtures should be used for checking the accuracy of the method.

Dr. Gerland perfectly agreed with Dr. Grossmann respecting the proof of the correctness of a new analytical method. In this case he had measured the indigo against ammoniacal copper solution, by means of hydrosulphite, according to Miller's method. He had, further, in order to test the fact of impurities, worked with weighed quantities of indigotin and natural indigos, and found the results agree with the calculated quantities, and he thought this was sufficient proof for any chemist. In reply to Dr. Knecht, he thought the method of nitro-benzol adapted itself well to the exact estimation of indigotin on cotton, wool, or any other fibre; but he preferred to destroy the fibre by treatment with weak sulphuric acid; then, after washing and drying the residue, to treat it with nitrobenzol, and extract pure indigotin, as mentioned in the paper. He had found that in three parallel analyses the difference in results amounted to 1 in 300—a result quite as good as they might expect in any mineral analysis.

THE FIXATION OF PIGMENTS WITH ALBUMIN.

BY R. J. FLINTOFF.

Last session I read a paper before this Society, entitled "The Functions of Albumin as a Fixing Agent for Pigments on Cotton," and drew attention to the fact that the addition of albumin to a printing colour increases its intensity of shade. The explanation of this phenomenon as stated in the paper was that the albumin increased the opacity of the layer of colour, and also, while in solution, distributed the printed pigment over the fibre with a more even and solid surface. The result is that the actual quantity of light rays reflected is increased by both these conditions; because in the former case less rays are transmitted, and, in the latter, less are scattered.

I have now done further work on this subject with a view to discover which of the above causes plays the most important part in bringing about "intensification."

The method of investigation consisted in producing on the fibre two layers of colour, each presenting a perfectly solid and smooth surface, that is, with one of the above conditions common to both. To the one an addition of albumin had been made, and in every other particular they were similar. It is, therefore, obvious that any difference in shade between these two colours, due to the albumin, must be attributed to an increase in the opacity of the colour on the fibre.

To produce a pigment on the fibre possessing these required properties, it is necessary to print the constituting parts on the cloth in a state of solution or else so finely divided that it almost approximates to solution, and to bring about their combination afterwards. Then a pigment colour will have been produced on the cotton fabric having the desired reflecting surface. Accordingly the following mixtures were made:—

1.—150 grms. alizarine, 20 per cent. (blue shade).
96 c.c. acetate of lime, 16° Tw.
90 c.c. sulphocyanide of alumina, 32° Tw.
84 c.c. oxalate of tin, 25° Tw.
580 grms. starch thickening.

2.—100 grms. alizarine, 20 per cent. (blue shade).
40 c.c. acetate of lime, 16° Tw.
50 c.c. acetic acid, 9° Tw., 30 per cent.
90 c.c. acetate of chrome, 32° Tw.
720 grms. starch thickening.

3.—50 grms. alizarine, 20 per cent. (blue shade).
250 c.c. acetic acid, 9° Tw., 30 per cent.
75 c.c. acetate of lime, 16° Tw.
25 acetate of iron, 23° Tw.
600 grms. gum thickening.

Each of these mixtures was then divided into two equal parts. To the one an aqueous solution of albumin was added, and to the other a corresponding volume of thin starch paste. The two printing pastes made from each of the above mixtures were then printed on the same fent from the same roller and dried. After drying, the colour containing the albumin was darker in each case. For instance, in No. 1 mixture, the albumin was an orange colour, and the other was lighter in shade. The difference in shade between the comparable portions of each mixture was judged to be about the same.

The fents were now steamed 1 hour and 20 minutes without pressure, and during this process the pigments were precipitated on the fibre, being respectively coloured red, chocolate, and lilac. In my previous experiments the pigments were precipitated before printing, whilst now they have been precipitated on the fibre for the purpose previously explained. On comparing the two parts of each

fent, there appeared very little difference in shade between them; but the one containing the albumin was slightly darker. Now, since the only difference between these two series of shades is that those containing the albumin have increased opacities, and as their intensities exhibit only slight differences, the deduction must be made in these cases that the decreased transparencies of the layers of colour have not produced corresponding increased intensities in the shades of the colour-lakes. Further trials were made similar to the above, only the colouring matters and mordants were varied and gave in every case corroborative results.

One interesting fact noticed was the addition of albumin to the printing colour increased the fastness to light of the resulting shade. This, I presume, may be attributed to the coagulated albumin surrounding the particles of pigment and protecting them from the direct action of the light. Similar experiments could not be made with basic colouring matters, in so far as albumin precipitates the tannic acid and prevents its complete combination with the colouring matter.

I may here mention, incidentally, that the precipitate formed when aqueous solutions of albumin and tannic acid are mixed together, does possess to some extent the power of combining with certain colouring matters. If, for instance, albumin solution be added to the printing paste of a basic colouring matter, a slight thickening of the mixture is noticed, owing to precipitation, and the colour is very materially altered in shade. This may then be printed upon cotton, and after steaming it is found of an entirely different shade from the one produced with tannic acid alone. Moreover, it is apparently quite as fast to soap as the antimony-tannate lake of the same colouring matter. It may be shown that this substance possesses dyeing properties in a totally different and more direct manner. Aqueous solutions of tannic acid and albumin are mixed together in the cold and the precipated substance washed by decantation with cold water, and finally filtered. If this compound is then suspended in the aqueous solution of a basic colouring matter, it becomes dyed and may be washed without losing its colour. After drying and grinding, it has the appearance of a bright highly coloured powder. This method may possibly be useful for the preparation of pigments for special purposes.

I fear these remarks are a little irrelevant to the general tenour of this paper; but I have inserted them because they have an indirect relationship to the subject, since they were suggested from the results of experiments performed during the investigation.

From the facts so far obtained one would naturally conclude that the cause of the phenomenon of intensification is the reflection of an increased number of rays of light, owing to an improved reflecting surface, which results almost entirely from the more regular distribution of the pigment over the surface of the cotton fabric. But there is one important possibility which has not yet been taken into consideration in this argument, and supposing it to exist one might very easily prove the conclusions arrived at not altogether reliable. All the pigments precipitated on the fibre were very dark shades and hence the opacity of a film of such colour is very much greater than a film of so small a quantity of coagulated albumin as exists in the colour.

The point I wish to emphasise must be fairly obvious. That so long as the opacity of the actual layer of colour is great as compared with the opaqueness of the coagulated albumin contained in the colour, so long will the latter be a very small factor in the phenomenon of intensification. But should the former become less and less, whilst the ratio of the latter to the whole opacity increases—although the actual quantity of albumin remains a constant—then the opacity due to the albumin will become a proportionately greater cause in the phenomenon. And it is possible to conceive a certain adjustment between pigment and albumin, when the opacity of the former would be infinitesimal as compared with the latter; and should "intensification" be still observed, the former generalisations could hardly be true.

Now, since in the experiments previously described very little light is transmitted through the layer of colour itself, a still further slight decrease in the quantity of transmitted light would not materially affect the intensity of the colour; but if any alteration could be perceived, it would be a very slight increase in the depth of shade. This has actually been observed, and, therefore, in order to note the effect of precipitating on the cotton fabric more transparent films of colour, a further series of experiments were made. With this object in view the mixtures previously prepared were considerably reduced with starch paste, and these were then printed exactly under the same conditions as the dark shades. These shades being very much lighter were consequently more transparent; but the differences in intensities between the albumin pigments and the other ones were practically just the same as with the darker colours.

From the results of these experiments I have arrived at the conclusion, that the increase in the intensity of the colour of pigments fixed with albumin is almost entirely due to the property possessed by albumin solution of spreading the pigment over the surface of the cotton fabric with a more even face, and, hence, with an improved reflecting surface. With regard to the coagulated albumin increasing the opacity of the layer of colour and, consequently, resulting in an increase in the intensity of shade, I have found this to be always a constant quantity, regardless of the strength of the colour, and, further, only a very small causal factor in the phenomenon.

Nottingham Section.

University College, Nottingham.

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SESSION 1896–97.

Dates of Meetings.—March 24th (Nottingham), April 28th (Derby), May 26th (Nottingham).

Meeting held at Burton-on-Trent, Wednesday, January 27th, 1897.

MR. F. J. R. CARULLA IN THE CHAIR.

THE COMMERCIAL POSITION OF SULPHATE OF AMMONIA.

BY F. J. R. CARULLA.

(This Journal, 1896, 863.)

Discussion.

The Chairman congratulated the section on the fact that they were holding their first meeting at Burton-on-Trent. A desire had been expressed, after the reading of his address, that on some convenient occasion a discussion should follow. That such a course might do good was obvious from the discussion that took place at a meeting of the Manchester Institution of Gas Engineers, following a paper by Mr. T. N. Ritson, three days after the delivery of the address, and which by a curious coincidence, but quite independently, went over much of the same ground as the address, a proof, if one were needed, of the acute character of the situation as it was in November last, but which had since improved, although the anomaly that they desired to remove still remained. It would seem useless, however, to look for relief to suggestions for the better employment of ammoniacal liquor in the manufacture of

some other product than sulphate, for, if followed, those interested might be placed in the position of the fruit-growers of the south of England, who, following Mr. Gladstone's advice, turned their harvest into jam, spending large sums in plant for the purpose. So many went into the new industry as to cause a plethora, thus placing them in a worse plight than they were before. And yet no sensible man would say that Mr. Gladstone's advice was not good. The obvious requirement in the case of sulphate of ammonia was to bring the farmer to see where his true interest lay, and in this sense Mr. Ritson's paper and the discussion which appeared in the Journal of Gas Lighting for December 1 and 8, 1896, would repay perusal. A noteworthy point in the discussion by the gas engineers was the fact how some of the largest buyers of sulphate of ammonia, namely, the manufacturers of artificial manures, were overlooked. Almost every speaker who referred to the farmer, including the author of the paper, recommended that he should be taught how to compound artificial manures for himself, thus turning every farm into a small manufactory. That this might be the right thing to do in particular instances one could quite believe, but it was not easy to see how the abolition of the large customer, and his replacement by a number of small ones, was going to mend matters.

Mr. Ritson said, with great discernment, "There is no doubt that sulphate of ammonia is the nitrogen *par excellence* for compounded artificial manures. It can be mixed and stored. . . . Mixed manures prepared with nitrate of soda cannot, however, be stored for any length of time, from the fact that it deteriorates and deliquesces." This property of sulphate, which ought to be impressed on the farmer, would evidently recommend it with still greater force to the artificial manure manufacturer, who might be left with heavy stocks at the end of a season.

What seemed of importance was that the farmer should understand the value of nitrogen as such in any manure, no matter the form in which it might be present. The Fertilisers and Feeding Stuffs Act, passed recently for the protection of the farmers, rightly took no cognisance of the form in which the nitrogen appeared, the unit, no matter whence derived, being given the same value. Such legislation would appear to be a powerful tool, which, properly handled, should help to bring about the proper appreciation of the nitrogen in sulphate of ammonia.

It was not likely that there was any intention on the part of the gas engineers to injure the artificial manure makers, and as probably none of the latter were present, the discussion became rather one-sided. For this reason it was all the more desirable that the matter should be ventilated before a meeting in which all the various interests were represented.

Mr. J. O'Sullivan said that the authorities he had consulted concurred on the equal value of the unit of nitrogen, whether derived from nitrate of soda or from sulphate of ammonia, and he thought, consequently, that farmer was the wisest who availed himself of the variations in prices, and always bought the manure which, nitrogen for nitrogen, was the cheapest. He would ask whether any experiments had been made, such as those of Lawes and Gilbert, to prove the equal value of the unit of nitrogen in nitrate of soda and in sulphate of ammonia for the purposes of the agriculturist.

Dr. F. Clowes referred to the enormous expenditure incurred by the Scottish Ironmasters in adding ammonia absorbing plant to their blast furnaces. He knew that the owners of some English blast furnaces had followed suit and that there were others who were also about to put down plant for this purpose. If the use of sulphate of ammonia was decreasing he failed to see what benefit could be expected to result from this large expenditure.

Mr. John White said that what he felt to be a grievance was that wherever nitrate of soda supplanted sulphate of ammonia they were losing the benefit of a home manufacture. In regard to the Fertilisers and Feeding Stuffs Act, to which reference had been made by the chairman, it was quite true that the unit of nitrogen was given the same value in it no matter whence derived. Although going away a little from the subject he would add that the Act was practically inoperative owing to the manner in which it prescribed that the sample should be taken. What was wanted is the appointment of inspectors as had been done under the Food and Drugs Act.

Mr. J. O'Sullivan said it struck him that the great obstacle to the spread of the use of sulphate of ammonia was its name. The name "nitrate" carried at once the idea of nitrogen to the ordinary man's mind, whereas "sulphate" did not. If sulphate of ammonia could be re-christened so that the name should convey this idea it might be a good thing.

The Chairman, in reply, said that experiments had actually been conducted over and over again in the sense indicated by Mr. O'Sullivan, with the result that the unit of nitrogen was found to be of equal value in the two manures, or practically so. Of course failures might occur with sulphate of ammonia if ignorantly used and brought into contact with lime, when loss of ammonia must of necessity occur. He would have a caution printed on every page of all sulphate literature to prevent the occurrence of this. In regard to the question of English ironmasters having followed the Scottish ones, it should be remembered that in Scotland almost all the furnaces were worked with raw coal, whereas the English practice was to use coke. In this case the ammonia could only be recovered at the coke ovens. The English firms who were putting down absorbing plant, doubtless would also use raw coal and they might have made their arrangements before the commencement of the present depression. As tar, however, was also recovered and this product was valuable, the action of the ironmasters might turn out profitable after all.

Dr. Clowes thought it was desirable to point out that the reference to lime made by the chairman was to caustic lime, as most soils contained lime in combination.

The Chairman said this was so, it was of course only in the case of bringing lime in an uncombined form or only hydrated into contact with sulphate of ammonia that loss occurred.

ON KJELDAHL'S PROCESS.

BY JAMES O'SULLIVAN, F.I.C., F.C.S.

At the time this process for the estimation of organic nitrogen was published I had considerable experience (this Journal, 1892, 327) with Dumas', and knowing the time and trouble entailed by the latter I wished to adopt the former, which would take less time. I inquired if it had been employed to determine the nitrogen in bodies similar to those I had to deal with, and I found it had, yet no one had proved its results in this direction, by checking them with those obtained from the same bodies with Dumas' method. A few determinations were made with this object in view, and I came to the conclusion, as the results will show, that Kjeldahl's process gives the same results as Dumas'.

Re-agents used. A 40 per cent. solution of sodium hydrate to which 2 to 3 grms. of potassium sulphide per 100 c.c. were added, this solution was boiled for some time.

A mixture of 1 part of strong sulphuric acid and 2 parts of Nordhausen sulphuric acid.

A solution of sulphuric acid containing 0·008 grm. of sulphuric anhydride per c.c.

Powdered potassium permanganate.

A blank experiment with these re-agents gave no ammonia.

It is not necessary for me to go into details as to the preparation of a solution for a determination of its nitrogen, or to describe the manipulation of either of the processes as these are already well known; I wish, however, to mention that at the suggestion of Dr. Harrow, about 0·5 grm. of mercuric oxide was added with the mixture of sulphuric acids in the Kjeldahl's oxidation process. This oxide hastens the oxidation and the potassium sulphide in the sodium hydrate when added in excess, before distillation, dissolves the sulphide of mercury first formed.

The following are the results obtained by these processes, and are expressed in percentage of albuminoids on total solids in solution (nitrogen × 6·25 = albuminoids) :—

(1.) Malt extract (mash extract), Kjeldahl's 3·8, Dumas' 3·78.

(2.) Ale, Kjeldahl's 3·5, Dumas' 3·22.

(3.) Ale, Kjeldahl's 4·15, Dumas' 4·31.

By employing the sulphuric acid of the strength mentioned for receiving the ammonia in the distillation the number of c.c. neutralised multiplied by 0·0028 give nitrogen, and by 0·0175 give albuminoids.

Discussion.

Dr. Clowes remarked that the Kjeldahl process was now in very general use for estimating nitrogen in organic substances. In his own laboratory the substance was simply heated with the strong acid and potassium sulphate until the liquid became clear. The cool liquid was then transferred to a larger flask, and a solution of sodium hydrate, containing more than enough alkali to neutralise the acid, was poured into the flask in such a way as to form a separate layer. Steam from a separate vessel was then blown through these layers of liquid. Heat was produced by the mixture of the acid with the alkali, and the current of steam carried over the ammonia through a condenser into the standard acid. This procedure was convenient, and was rapidly carried out.

A NOTE ON MALTOSE.

BY JAMES O'SULLIVAN, F.I.C., F.C.S.

The numerous papers on the hydrolysis of starch which have appeared since C. O'Sullivan's first paper on this subject (Chem. Soc. Journ., 1872) leave little to be desired. I must, however, agree with Dr. Armstrong (Proc. Chem. Soc., p. 244), "that we should no longer be content merely to determine certain analytical factors; we ought rather to seek for chemical methods which would render it possible to separate and isolate the products." Maltose was separated by C. O'Sullivan from the transformation products of starch, obtained by the action of the cold extract of malt, and its properties described. He gave the $[\alpha]_j$ 154—155, and its cupric reducing power between 62—63, and Brown and Heron 61·0 (Chem. Soc. Journ., 1879).

It occurred to me to determine the properties of maltose prepared by transforming starch with precipitated diastase —diastase precipitated from malt extract by alcohol—thus insuring the absence of the crystallisable sugars contained in malt extract from the maltose. Maltose prepared in this manner and crystallised from water gave an $[\alpha]_j$ 154, and this preparation crystallised from alcohol gave $[\alpha]_j$ 154·5 K = 62·7. The latter was again crystallised from alcohol and gave the following results :—

(I.) C = 4·164 D = 3·96 $[\alpha]_j$ 155·0

(II.) C = 1·558 D = 4·00 $[\alpha]_j$ 155·3.

Determinations of the cupric oxide reducing power were made by employing weighed quantities of the maltose, and the following are the results in a tabulated form :—

Experiment.	Grms. of Maltose taken.	Cupric Oxide weighed.	Grms. of Maltose to reduce 1 grm. of CuO.
I.	0·1070	0·1490	0·7181
II.	0·1180	0·1645	0·7173
III.	0·1065	0·1465	0·7269
IV.	0·1170	0·1640	0·7134
V.	0·1175	0·1630	0·7208
VI.	0·1200	0·1660	0·7228

The average quantity of maltose, from these results, required to reduce 1 grm. of CuO is 0·7198 grm. or a K of 63 taking dextrose at 100. If this number be employed in calculating from the CuO obtained in the above determinations, the quantities of maltose found come out as near as one could wish with the amounts of maltose taken as the following columns show :—

Experiment.	Grms. Maltose taken.	Maltose found.
I.	0·1070	0·1072
II.	0·1180	0·1184
III.	0·1065	0·1050
IV.	0·1170	0·1180
V.	0·1175	0 1173
VI.	0·1200	0·1195

The maltose was first dried in vacuum over sulphuric acid, then in a current of dry air at 100°, and finally in a current of dry air at 100° under a pressure of 100—150 mm.

In determining the cupric oxide reducing power I have in no way deviated from C. O. Sullivan's method (Chem. Soc. Journ., 1876, 2, 130), I find, however, that the weight of the ash of a filter is doubled by filtering the copper solution through it, as in the determination of the reducing power, and that this is not due to any precipitation of the solution but is due to a combination of the copper with the cellulose of the filter.

25 c.c. of Fehling's solution diluted with 25 c.c. of water were boiled, as in the determination of a reducing power, for 12 minutes, it was then passed through a filter, (1) the filtrate from this was immediately passed through another filter, (2) and the filtrate from this through another filter, (3) about 400 c.c. of boiling water were used in washing the solution out of each of these filters. The ashes of the three filters weighed 0·0025, 0·0025, and 0·0023 grms. respectively, whilst the ash of the filter weighed only 0 001 grm. The ash of each of the filters was dissolved in a drop of dilute hydrochloric acid, and upon testing with potassium ferrocyanide showed the presence of copper.

The numbers given in the above determinations of cupric oxide reducing powers were obtained by employing different copper solutions so far as "age" was concerned; some were determined with Fehling solution which had been made up 13 months, and others with a solution which was only made three days, and I fail to see any practical difference in the results obtained. Two determinations of the K of a sample of large crystallised dextrose prepared from cane sugar by invertase—not yeast or yeast extract—and dried as this maltose was, gave with one of the copper solutions a K 101·4 and 102·4, and the last number is the highest I have ever obtained for a sample of dextrose from cane sugar. I must say, from my experience, that the K of dextrose almost invariably comes out 1—2 per cent. above 100, whilst that of invert sugar comes out as much below.

Discussion.

Dr. A. L. Stern pointed out that Mr. J. O'Sullivan, in calculating a mean value of the K of maltose from his experiments on one sample of maltose, and employing the mean value to calculate (in each of the same experiments), the amount of matter equivalent to the copper oxide weighed, and comparing this with the amount of maltose taken, had adopted a method which was bound to give apparently good results whether his original specimen of maltose was pure or not.

He also expressed surprise that Mr. J. O'Sullivan still found such low numbers of the K of dextrose, as Mr. C. O'Sullivan, F.R.S. and himself had recently published a paper in the Journal of the Chemical Society describing a large number of experiments, which had led them to the conclusion that the old value was materially low, and which was confirmed by other workers.

Yorkshire Section.

Chairman: Thos. Fairley.
Vice-Chairman: Christopher Rawson.

Committee:

H. E. Aykroyd.	W. Leach.
J. Cohen.	A. G. Perkin.
J. R. Denison.	F. W. Richardson.
N. Farrant.	A. Smithells.
T. Glendinning.	Geo. Ward.
A. Hess.	Thorp Whitaker.

Hon. Local Secretary and Treasurer:
H. R. Procter, Yorkshire College, Leeds.

SESSION 1896-97.

Meeting held on Monday January 25th, 1897.

MR. THOS. FAIRLEY IN THE CHAIR.

THE QUALITATIVE SEPARATION OF ARSENIC, ANTIMONY, AND TIN.

BY S. G. RAWSON, D.SC., F.I.C.

Lecturer in Chemistry, Technical College, Huddersfield.

To any originality in the process which I propose describing I can lay no claim, the principles involved depending upon a combination of certain well-known methods. The suggested separation of these metals is based primarily upon the use of oxalic acid as originally published by Clarke in his paper, Chem. News, 21, 124, upon the quantitative estimation of these metals, a process which to me never seems to have come into the prominence which it so thoroughly deserves. I have but little faith in the methods now employed, especially in those for the separation of antimony and tin. The former metal is frequently evolved either as the hydride or deposited as a black stain upon platinum; the tin being thrown down upon zinc, this deposit is then scraped off, dissolved in acid, and the solution treated with mercuric chloride. That other people share in my disbelief in this method is shown by the fact that in so far as my knowledge goes, I have never heard in any examination of more than two of those bodies being given for qualitative determination in one and the same substance. Even under these favourable conditions, it is seldom that the mixture contains only a small percentage of one of the constituents; the amounts more usually approximate to equality, the difficulties of separation being thereby much reduced. The method I would suggest is as follows:—The sulphides of the metals are washed upon the filter paper, and the whole or part of the residue is placed in a test tube and boiled with 2 or 3 c.c. of concentrated hydrochloric acid, to which a drop or two of nitric acid is added, and again boiled. A yellow residue after the treatment with hydrochloric acid gives a preliminary clue as to the presence of arsenic, this sulphide being slightly, if at all, attacked by the acid. A saturated solution of oxalic acid is added in quantity sufficient to fill two-thirds of the test tube, the whole is boiled and crystals of oxalic acid are added until a hot concentrated solution of oxalic acid is obtained. A stream of sulphuretted hydrogen is passed, the whole of the arsenic and antimony being precipitated as sulphides, which are filtered off, the tin remaining in solution. To the filtrate ammonia is added until distinctly alkaline. If a precipitate should then appear, which will not be the case unless a large amount of tin be present, add ammonium sulphide drop by drop until the precipitate redissolves, this it will do very readily. Acidulate with acetic acid, a heavy white precipitate, turning brown, indicates tin as a mixture of oxide and sulphide. It may be here noted that the treatment of ordinary ammonium sulphide with acetic acid produces a precipitate of sulphur, but the appearance of the precipitates formed in the two cases is quite distinct and cannot be mistaken. Turning again to the residual sulphides of arsenic and antimony, these may be treated either with ammonium carbonate in the well-known way or, and preferably I think, as follows:—Dissolve in hydrochloric acid with two or three drops of nitric acid, boil and place the solution in a Marsh apparatus. The evolved hydrides are then passed through a solution of silver nitrate, and the antimonide of silver formed filtered off. To the filtrate add a few drops of silver nitrate, and then very cautiously ammonium hydrate until the yellow precipitate of silver arsenite appears. The silver antimonide precipitate is washed, boiled with tartaric acid and filtered, a little hydrochloric acid is added and sulphuretted hydrogen passed through the filtrate, orange red antimony sulphide being thrown down. To both of these precipitates the ordinary reduction and sublimation tests can be applied. The above method gives thoroughly good results and with amounts of the respective sulphides varying within wide limits, and does not require that tinkering with bits of platinum foil and of zinc which is both unreliable and unpleasant.

Discussion.

Mr. F. W. Richardson had at one time used Clarke's method quantitatively, but had given it up qualitatively in favour of one where the sulphides were oxidised with hydrogen peroxide and ammonia, and the arsenic precipitated as magnesium ammonium arseniate, the antimony being then converted into hydride.

Dr. Rawson said that this still left the exact difficulty he especially sought to avoid, *viz.*, the separation of the antimony and the tin after removal of the arsenic. Moreover, the complete precipitation of the arsenic as the magnesium salt required much time.

In reply to Mr. C. Rawson, the author thought his method would work for the estimation of tin in the ash of small pieces of cloth where chromium and aluminium might be present.

Mr. F. W. Richardson considered, in connection with the precipitation of magnesium ammonium arseniate, that only a short time was necessary to bring down 99 per cent. of the arsenic provided a considerable quantity of ammonia were present.

Mr. Farrant said he had found that when very accurate results were required, 18 to 24 hours must be allowed for the magnesium ammonium arseniate to settle out (Fresenius gives 24 to 48 hours). In the case of an estimation where arsenic was present in large quantities, a great amount of the precipitate came down immediately on adding the magnesia mixture, but nothing like the whole amount; and in the case of an estimation where the amount was only small, say, in refined copper containing one-tenth per cent. arsenic, any precipitate appeared only after long standing and occasional stirring.

THE QUANTITATIVE AND QUALITATIVE SEPARATION OF BARIUM, STRONTIUM, AND CALCIUM.

BY S. G. RAWSON, D.SC., F.I.C.

Lecturer in Chemistry, Technical College, Huddersfield.

In the quantitative or qualitative separation of barium, strontium, and calcium there has been always a considerable amount of difficulty due to the remarkable properties of strontium which in so many respects lie almost midway between those of barium and of calcium. Hence though the estimation of both barium and calcium when present together, or of strontium when alone, presents but little trouble, yet if the whole three members of the group be taken the task becomes one of greatly-increased difficulty, it being no easy matter to ensure either the complete precipitation or the entire retention in solution, according to the process to be employed, of the strontium. To attain one or other of these ends many methods have been proposed, amongst these I may mention the following:—Fresenius recommends in his work the precipitation of the barium as silicofluoride and the subsequent separation of the strontium and calcium by means of prolonged boiling

with an enormous excess of ammonium sulphate. The particulars of the remaining processes are taken from the Journal of this Society. Fresenius and Ruppert (1892, p. 776) have investigated the relative solubilities of strontium and calcium chromates in dilute alcohol: though of value qualitatively they consider that quantitatively the method is lacking in accuracy. McElroy and Biglow (1893, p. 181) suggest the use of aqueous acetone upon these same salts, but seemingly only for qualitative purposes. In this year also (1893, p. 627) Fresenius recommends the separation of calcium as nitrate from the other nitrates by its solubility in ether-alcohol, the results he gives being very accurate. Browning (1894, p. 282) carefully examined the solubility of strontium nitrate in boiling amyl alcohol in which the salt is almost insoluble the corresponding calcium nitrate dissolves readily, but to effect a complete separation a second treatment is necessary. Dupasquier (1895, p. 822) points out that calcium is converted into a tartrate by boiling in a liquid containing a soluble tartrate and sulphate, while barium and strontium remain as sulphates. These are estimated indirectly for quantitative purposes. Banbigny (1895, p. 1065) separates the strontium as sulphate in the presence of potassium sulphate, but the precipitation is not complete.

This bibliography though not complete includes the most important of the plans suggested. It will be noticed that in almost all of them there arises the difficulty of the slight solubility of the strontium salts in the solvents necessarily employed. Further, it rarely happens that the same method can be used both quantitatively and qualitatively. But the special objections to these methods are due to the fact that either out of the way reagents are employed, or that much and prolonged boiling is necessary to convert the given salts into the required modifications. Both these are hindrances in the way of quantitative analysis and are most serious detriments to qualitative work.

The principle of the separation upon which I have worked, depends entirely upon the behaviour of the nitrates of these three metals towards concentrated nitric acid. The method is also readily applicable to qualitative analysis; the nitrates of barium and of strontium are quite insoluble in this acid, whilst calcium nitrates dissolves very rapidly. That barium nitrate is insoluble appears to be accepted, but there is considerable divergence of opinion as regards the strontium salt, thus, Wurtz (Am. J. Sci. (2) **25**, 377) states that it is sparingly soluble in concentrated nitric acid, whilst Schultz (Zeit. Ch. (2) **5**, 537) considers it to be insoluble. The experiments by which I sought to settle this initial contradiction were as follows:—The nitric acid used and which was bought as pure was redistilled, the water also being similarly treated. In the case of both the nitric acid and of the water, portions upon evaporation either left no residue or one which it was not possible to weigh. The carbonate was obtained, at different times, from Germany and as being perfectly pure. I may here mention that it is most difficult to procure pure strontium carbonate. All the material which I obtained invariably contained traces of calcium in amounts varying from 0·25 per cent. to even 2 per cent. The presence of this impurity caused me great inconvenience and at first much loss of time. In order to purify the carbonate a considerable quantity was converted into nitrate and thoroughly stirred with nitric acid, filtered, and the residue dissolved in water and precipitated as carbonate, washed and dried. In the spectroscope no trace of the calcium lines were now visible. So satisfactory and rapid is this method that I would recommend it strongly as being the most easy and the most safe for the purification of strontium from calcium salts. I may here mention that throughout my experiments I always used the carbonates of the metals partly because of the much greater convenience and certainty in weighing and also because the carbonates are far more likely to be required for estimation under usual conditions than the nitrates, and therefore it seemed advisable to work upon the more commonly occurring body.

The pure carbonate was treated with dilute nitric acid and evaporated to dryness upon the water-bath. Pure concentrated nitric acid (sp. gr. 1.445) was then added and the mixture kept well stirred for many hours, in some cases three days. The solution was then filtered through filter paper or glass wool and the filtrate evaporated to dryness taken up with a little hydrochloric acid and again evaporated. The residue gave, in the spectroscope, no trace of the strontium lines, but consisted of small traces of sodium and iron sulphates derived from the acids employed, and of celluloid matter arising from the filter paper, when used. Similar experiments were repeated many times varying the amounts of carbonate and of nitric acid, but only upon one occasion, in one of my earlier experiments, did I distinguish the strontium lines. I had treated some 3 grms. of the carbonate with nitric acid, and upon filtering and evaporating to dryness the residue clearly contained strontium. This was due to the solution of traces of the nitrate, the nitric acid having become diluted by the considerable amount of water formed in the conversion of the carbonate into nitrate. On all subsequent occasions I invariably evaporated the nitrate to dryness upon the water-bath and then took up with fresh nitric acid. Under these conditions the reaction was only as between the nitrate and nitric acid and the strontium lines did not again become visible in the filtrate. Hence strontium nitrate is insoluble in concentrated nitric acid. Direct experiments upon barium nitrate showed this salt to be also insoluble, whilst calcium nitrate readily dissolved. As regards the nitric acid, I prefer the specific gravity to be about 1·46 (corresponding to 80 per cent. of the acid) or even higher, but for most purposes the ordinary acid of specific gravity 1·42 (containing 70 per cent. HNO_3) will suffice.

The actual quantitative estimation of these elements carry out in the following manner:—The carbonates are evaporated with nitric acid, either in a beaker or in a porcelain dish, preferably the latter, upon the water-bath until the mass is quite dry. Concentrated nitric acid is added in excess, and the mixture kept well stirred. The crystals settle out after each stirring very rapidly, and the clear supernatant liquid is poured through a double filter paper which has been moistened previously with concentrated nitric acid. The residue may be washed either with the concentrated acid by decantation, a method to which the mass from its crystalline nature readily lends itself, or in the customary way upon the paper.

The filtrate is evaporated either to dryness, taken up with hydrochloric acid, and the calcium precipitated as oxalate, or evaporated down with sulphuric acid, and the residue after ignition weighed as sulphate.

The remaining nitrates are dissolved in water, and the solution made alkaline with ammonium hydrate, acidified with acetic acid, and the barium precipitated as chromate. The filtrate is warmed with hydrochloric acid and alcohol until the chromate is reduced, the chromium precipitated as hydrate and filtered off. The filtrate is evaporated to dryness upon the water-bath with a little sulphuric acid. The residue is treated with dilute alcohol and washed, and the residual strontium sulphate weighed as such. Two out of the different test analyses, which I have made, give the following results:—

—	1.		2.	
	Taken.	Found.	Taken.	Found.
Barium carbonate	0·5393	0·5379	0·2127	0·2130
Strontium carbonate ...	0·2578	0·2584	0·4583	0·4576
Calcium carbonate......	0·5473	0·5470	0·2773	0·2780

For the qualitative separation the above process may be much simplified. The carbonates, after treating with the minimum amount of nitric acid, are boiled down just to dryness; then treated with concentrated nitric acid, the paper having been first moistened with this acid, filtered, and the residue washed by decantation. The filtrate is diluted considerably with water made alkaline with ammonia, and the lime thrown down as oxalate. The residue is dissolved in water (the filter paper will not disintegrate unless boiling cold water be used), made alkaline with

ammonia, acidulated with acetic acid, and the barium precipitated as chromate. To the filtrate is added hydrochloric acid, and it is boiled with a little alcohol, and the chromium precipitated as hydrate, which is filtered off. To the filtrate sulphuric acid is added, and the solution heated: if necessary alcohol is added, the strontium coming down as sulphate. The separation of the chromium as hydrate is not in all cases necessary.

The process can be carried out very rapidly, and, further, is a complete separation of these three metals, and does not rest upon grounds such as the length of time allowed for boiling or standing, or the greater or lesser concentration of solutions.

Discussion.

The Chairman (Mr. Fairley) in opening the discussion, referred to the necessity for a good separation of these metals, he himself having experienced the want in the analysis of waters such as those of Harrogate.

Considering the fact that chalk dissolved only with difficulty in concentrated nitric acid, he should have imagined that calcium nitrate would hardly have been sufficiently soluble in concentrated nitric acid for purposes of separation.

Dr. S. G. Rawson had observed the difficulty of dissolving chalk in strong nitric acid, but pointed out that in his method dilute nitric acid was used to convert the carbonates, a procedure which eliminated the difficulty, and although chalk might be only soluble with difficulty in strong nitric acid, calcium nitrate was easily soluble in that medium. He also drew particular attention to the impurity of the strontium carbonate supplied as pure, stating that he had invariably found some calcium present, and in some cases even as much as 2 per cent.

Prof. Procter raised the question as to whether, in the spectroscope examination which the author had used to determine the completeness of the separation, a trace of one metal might not easily be obscured by an excess of another.

The Chairman, replying, stated that masking might easily take place, and cited a case where a considerable proportion of lithium was introduced into a water, and was unrecognisable spectroscopically until the other salts (of sodium and potassium) had been removed.

Prof. Smithells considered that masking might be due to the continuous light generally present in the predominating spectrum, and, referring to electric spectra, cited the recently observed case of Helium and Argon. He also referred to the masking of the carbon spectrum of a Bunsen burner, which took place immediately the flame was coloured by a foreign substance. He considered that examining the spark direct from the liquid was less likely to lead to masking of traces than evaporation and heating of the solid residue on a wire.

He also remarked that the lines at the blue end of a spectrum are less easily masked than those at the red, as, *e.g.*, in the case of lithium.

Prof. H. R. Procter was of opinion that masking might be a chemical rather than an optical effect.

Dr. Rawson thought that as regards the detection of traces of strontium and of calcium for the present purpose, the delicacy of the flame reaction in the spectroscope was sufficient.

Scottish Section.

SESSION 1896-97.

Meeting held at Glasgow on Thursday, Jan. 28th, 1897.

PROF. G. G. HENDERSON IN THE CHAIR.

NOTES ON RESIDUAL CYANIDE SOLUTIONS, AND A METHOD OF EXAMINING THEM.

BY CHAS. J. ELLIS, F.I.C.

The extraction of gold and silver from ores, tailings, &c., by means of weak solutions of the alkaline cyanides, is one of the youngest of the chemical industries, but is fast taking rank among those of the first importance. As far as I am aware, less than ten years ago, hardly any, if any at all, gold or silver was being extracted by this means on a practical scale, and already to the value of several millions stirling per annum is being produced, a large part of which is recovered from tailings which had, to a great extent, been cast aside as valueless, and in many cases considered an unavoidable nuisance by the mine owners, until this process had been devised and proved competent to deal with them profitably.

For the intelligent carrying out of the cyanide process, a more considerable knowledge of the chemistry involved in the details is necessary than might be at first supposed; of course, certain ores, &c. are very much more easily dealt with satisfactorily than others, and in some cases more or less satisfactory results may be obtained by means of the process in the hands of one who knows little or nothing of the chemistry of the more minute details; this, however, may be taken to be the exception rather than the rule, and hardly ever will the treatment be effected in the most economical way possible under superintendence of this class.

Of recent years, valuable contributions to the literature on the subject have from time to time been made by James, Bettel, Feldtmann, Butters and Clennell, and others; but the field is such a wide one, that I think it is unnecessary for me to make any apology for recording the following notes, even though they are partly a recapitulation of some facts now more or less well known, in addition to a statement of others which are perhaps less generally known.

When ores, tailings, &c., are treated with weak cyanide solution, a certain amount of free cyanide is consumed or destroyed, and the cause of this consumption may be tabulated under three general headings, supposing zinc is employed as the precipitant of the precious metals, *viz.*:—

(*a*.) Its combination with certain constituents of the material under treatment.

(*b*.) The action of the zinc in the precipitating boxes on it.

(*c*.) Its decomposition by exposure to atmospheric influences, prolonged contact with water, &c., &c.

(*a*.) Under this heading of course comes the small amount of cyanide actually consumed in forming double cyanides of gold and of silver with alkaline cyanide, during the solution of these metals; this item of the consumption is generally so comparatively minute as to be practically of little or no importance.

There are few, if any, gold-bearing materials which do not contain at least a small quantity of some compound of iron in the ferrous state, a portion of which is in a condition in which cyanide can readily attack it, and form ferrocyanide. It has been stated also that, in some cases, cyanide attacks certain ferric compounds and forms ferricyanide. As far as my experience goes, this action is, however, at most, trifling, compared to its action on ferrous compounds, and it is partly for this reason that the addition of lime or soda to ores, &c., which have become acid through weathering, and the subsequent oxidation by the air or otherwise, before actual treatment by cyanide, is so beneficial in preventing excessive consumption of cyanide in the treatment of such material.

Zinc is another metal which is occasionally dissolved out of ores, &c., probably by the cyanide forming double cyanide of zinc and potassium ($ZnCy_2 2KCy$) (this point

will be referred to again below); however, many of the compounds of zinc existing naturally in minerals resist the action of cyanide much more completely than would be expected from studying the action of cyanide on similar compounds produced artificially by precipitation from solutions.

When copper is present in ores, &c., at least a portion of it is almost invariably attacked by the cyanide, cuproso-cyanide of potassium ($Cu_2Cy_2 2KCy$) being mainly formed, accompanied, if the copper happens to exist in the cupric condition, with liberation of cyanogen, which, however, will form cyanide and cyanate of potassium if free alkali is present (which is generally the case), or react with soluble sulphides to form sulphocyanide, and possibly also go to form a little oxalate, oxamide, &c., &c. Arsenic is also often found in cyanide solutions after use, but has probably been dissolved by the alkali in the solution rather than by the cyanide *per se*. In somewhat exceptional cases, maganese, nickel, and even a little cobalt have been found in residual solutions, probably existing as manganicyanide, double cyanide of nickel, and potassium and cobalticyanide.

Much depends on the physical condition in which compounds of the bases exist in ores, &c., as well as on their chemical composition. One sometimes comes across ores, &c. containing a high percentage or copper in the form of sulphide, physically in a very hard condition, and finds little or no copper dissolved from them by cyanide.

While the bases are being attacked by the cyanide and forming double or compound cyanides, other compounds are being of necessity simultaneously produced. For instance, while the metal is being removed as cyanide from, say, a sulphide, alkaline sulphide is being simultaneously produced in the solution, which may further go partly to form sulphocyanide, especially if cyanogen is present also; thus, $2Cy + K_2S = KCy + KCyS$.

This formation of sulphide in solution is a matter of considerable practical importance, and if it takes place to any extent, means should at once be adopted for its removal from solution, as, even in comparatively minute quantity, its presence greatly retards, or even partly prevents the solution of gold and silver by cyanide. I first noticed this fact about eight years ago, while investigating the effect of the addition of various salts to cyanide solution in relation to its action on the constituents of ores, &c. I found that chlorides, sulphates, nitrates, ferro-cyanides, and even hyposulphites and sulphocyanides, &c., when present, even in considerable quantities, have, as a rule, little or no bad effect, but that even as little as one part of alkaline sulphide in 10,000 of solution has often quite a marked effect in retarding the action of cyanide in dissolving gold.

It having been thus found of importance to avoid, as far as possible, sulphide remaining in solution, in this connection MacArthur and myself some years ago took out a patent for the addition of certain salts of lead, &c. to the solutions where found necessary for its removal.

It is the practice to employ the same cyanide solution continuously for a more or less prolonged period, simple alkaline cyanide being added from time to time. It was at first feared that by doing so, the impurities, such as zinc salts, sulphides, &c., &c., would accumulate in the solutions to such an extent as to make them in time quite unfit for use; this fortunately appears seldom to be the case, however, in practice, as in addition to the formation of such compounds, certain reactions are also taking place in the course of employing these residual solutions, tending to remove or decompose at least some of the impurities. For instance, a portion of the zinc tends to be removed as sulphide during the action of the solution on pyritic material, or by reaction with any sulphide in the solution, in the latter case a corresponding regeneration of simple cyanide simultaneously taking place; a portion of the sulphide may also be decomposed or removed during its passage through the zinc box, or by reaction with zinc in solution, to form the insoluble sulphide, and another portion by slow transformation into sulphocyanide, &c.

(*b*.) The consumption of cyanide in the zinc extractors (if this really does take place in presence of free alkali) is probably mainly due to the formation of double cyanide of zinc and potassium in accordance with the equations—

$$4KCy + Zn + H_2O + O = ZnCy_2 2KCy + 2KHO,$$

$$\text{and } 4KCy + Zn + 2H_2O = ZnCy_2.2KCy + 2KHO + H_2.$$

The whole question of the reactions taking place in the zinc extractors has beeen ably and somewhat fully discussed by James, in his paper on "Cyanide Practice," read before the Institute of Mining and Metallurgy in May 1895.

(*c*.) The decompositions which come under this head, namely, prolonged exposure of cyanides to the influence of of the atmosphere, water, &c., involve reactions of an even more complex and deep-seated nature than those under the former two headings. I do not propose here to enter into a discussion of these reactions, but simply to state that the products of decomposition may include any or all of the following, *viz*., cyanates, cyanurates, urea, carbonate of ammonia, formates, and possibly oxalates, oxamide, &c., &c. The direct effect of the accumulation of these various compounds in residual solutions on the efficiency of such solutions for extracting gold, does not appear to have yet been very fully studied; it is, however, known that the presence of any considerable quantity of such a compound as ammonium carbonate tends to accelerate the formation of such bodies as paracyanogen, azulmic acid, &c., in the solutions, and generally the destruction of cyanide.

In the examination of ores, tailings, &c., for treatment by the cyanide process, one of the first things to be determined, after making preliminary assays, is the amount of cyanide consumed by the material, and if this is found to be in any way considerable, the cause of such consumption should be determined approximately. It is usually found that when the consumption is at all high, a large part of the total consumption takes place almost immediately on mixing the ore and solution, this being generally at least more than half the total consumption which will take place during prolonged contact. The determination of this portion is made as follows:—If the material has been found to be acid, it is mixed with a slight excess of caustic soda solution and agitated with it for half an hour or so, with free access of air; the requisite cyanide solution is then added and agitation continued for ten minutes, after which the liquor is filtered off and the cyanide remaining determined; the difference between this amount and the quantity employed giving the consumption. Subsequently a similar experiment is carried out. agitating with the cyanide for a number of hours.

To determine approximately the cause of consumption, an aliquot portion of the liquor from one or other of the above experiments is taken, evaporated nearly to dryness, and then drenched with strong sulphuric acid. and warmed to thoroughly decompose any compound cyanide, and the excess acid driven off; the metals will now be in the form of sulphates, and in a suitable condition for separation and determination by the ordinary analytical methods. The zinc is generally taken to have existed as double cyanide of zinc and potassium, the copper as cuproso-cyanide of potassium, and the iron as ferro-cyanide of potassium.

The consumption of cyanide-accounted for by calculation from the bases found in solution generally falls somewhat short of that determined by testing the cyanide remaining undecomposed after use; the difference between the two is taken as being decomposition, due to causes other than the direct action on bases. It is almost invariably found that the total consumption of cyanide in practical working is very considerably less than that found in small-scale experiments; this smaller consumption is considered to be mainly due to the proportionately smaller surface of solution exposed in large than in small operations; and to reactions, some of which are referred to above, tending to the regeneration of simple cyanide in the solution. The portion of the consumption due to direct action of the cyanide on bases, as determined in the above manner, is, however, probably more or less the same on whatever scale the treatment may be carried out.

The determination of free alkaline cyanide in the solutions to be employed, or remaining in solution after use, is almost invariably made by Liebig's method, *viz*., titration with silver

nitrate solution until a permanent cloud is obtained. In the case of pure solutions of cyanide, or even of those containing considerable quantities of most other salts commonly met with, this method gives results of great accuracy, *viz.*, within one part of cyanide in one million parts of water being able to be determined. The presence of ammonia interferes with the test, from the fact that the cloud, which consists of silver cyanide, is dissolved by the ammonia; this, however, is easily corrected for by the addition of a little iodide of potassium. As soon as all the cyanide is in the form of double cyanide of silver and potassium, the succeeding drop of silver solution forms silver iodide, which is practically insoluble in ammonia. If sulphide is present it must be removed by means of lead carbonate before applying the test. The presence of double cyanide of zinc and potassium along with alkaline hydrate, or even carbonate, however, entirely does away with any accuracy in this test for the determination of free alkaline cyanide in distinction to the cyanide existing in the double zinc cyanide, if this latter really does exist in solution in presence of the free alkali. In a paper, "On the Technical Analysis of Cyanide Working Solutions," read before "The Johannesburg Chemical and Metallurgical Society" in August 1895, Bettel draws particular attention to this, and discusses it somewhat fully; and also to the fact that if sufficient caustic alkali is present in, or is added to a solution containing double zinc cyanide, on adding some iodide and titrating with silver nitrate, the point at which opalescence begins indicates sharply the total cyanogen present in the double cyanide—of course, in addition to any also existing as simple alkaline cyanide; and even that the presence of alkaline carbonate interferes with the correct testing of simple cyanide in presence of double zinc cyanide. He suggests the following equations to explain these facts:—

$$K_2ZnCy_4 + 4KHO = ZnK_2O_2 + 4KCy + H_2O,$$

and in the case of the presence of excess of akaline carbonate

$$K_2ZnCy_4 + 4Na_2CO_3 + 2H_2O =$$
$$2KCy + 2NaCy + ZnNa_2O_2 + 4NaHCO_3$$

If we can accept these equations as correctly describing what takes place, it follows that no double cyanide of zinc and potassium can form, or exist, in solution so long as excess of free caustic alkali, or even carbonate, is present, and consequently that no actual consumption of simple cyanide, to form double cyanide of zinc and potassium, will take place during the passage of the solutions through the zinc extractors, or during their contact with zinc compounds, contained in ores, &c., so long as sufficient free alkali is present in the solution. The equations which I believe are generally accepted as representing the main reactions which take place in the zinc box, and which have been adopted by James in his paper on "Cyanide Practice," are as follows:—

(*a*.) $2KAuCy_2 + Zn = K_2ZnCy_4 + Au_2$,
(*b*.) $4KCy + Zn + H_2O + O = K_2ZnCy_4 + 2KHO$,
(*c*.) $4KCy + Zn + 2H_2O = K_2ZnCy_4 + 2KHO + H_2$,
(*d*.) $Zn + 2KHO = K_2ZnO_2 + H_2$,

and further, as suggested by Feldtmann,—

(*e*.) $AuKCy_2 + H = HCy + KCy + Au$,

in addition to others which I need not recapitulate. The last of these is interesting as showing how the hydrogen evolved may of itself play an important part in the precipitation of the gold in the zinc box.

In the same paper he expresses his opinion, and gives his reasons for so doing, that the reaction in the zinc box is rather between the caustic alkali and the zinc, than between the cyanide and the zinc.

The question as to whether a mixture of dilute solutions of zinc double cyanide and caustic alkali remains as such, or that the compounds react to form free alkaline cyanide and zinc-potassic oxide, is one which is worth discussing, and the subject seems to be deserving of even further investigation than has yet been made of it. It is, however, one of those cases concerning which it is difficult to prove the truth of one or other view by direct tests, as generally the mixed solution would behave in the same way towards reagents in whichever of the two ways the various elements were combined.

In connection with this whole subject, I may mention that the effect of the presence of free alkali on the titration by silver of cyanide solutions containing zinc came under my notice many years ago, and during 1890–91, in conjunction with Adam Dickie, I carried out an investigation of the matter, and before it had proceeded far we considered the reason to be that a mixture of zinc double cyanide and caustic alkali or alkaline earth did react to form simple cyanide and the double oxide of zinc and the alkaline metal, and we then lodged a provisional specification to patent the addition of caustic alkali or lime to residual solutions, supposed to contain double zinc cyanide, in sufficient quantity to effect this reaction; or preferably to cause such solution to flow through or over beds of lime. For certain reasons we did not complete the patent, but I am still strongly of opinion that especially the second of these methods, *viz.*, from time to time flowing the residual solutions over or through beds of lime, where it could be applied cheaply enough, would, in many cases, be found most beneficial to such solutions, by revivifying them, removing certain impurities from them, and generally bringing them into a more fit and active condition for continued re-use than if not so treated.

During the course of our investigation we carried out numerous trials on various samples of ores, using solutions of double zinc cyanide, or of residual solutions practically free from simple cyanide, but containing double zinc cyanide alone, and after addition of caustic alkali or passage through a lime filter; also, in some cases, after treatment with a soluble sulphide and removal of excess of sulphide by lead carbonate; and arrived at the following conclusions, *viz.*, that in almost all cases some of the gold and silver is extracted from ores, &c. by the use of double zinc cyanide alone, and that in most cases the extraction is better with the solution treated with alkali, or by sulphide, than not so treated. We also proved that part of the zinc is removed from zinc double cyanide by contact with certain pyritic ores, by mixing a pyritic ore with a solution of double zinc cyanide and agitating for some hours, and determining the zinc in solution before and after use.

Any very large excess of caustic alkali in the solutions should, however, be avoided, as its presence tends to cause reactions to take place, or at least to intensify them with some of the constituents of the ores, &c., such as sulphides, arsenic compounds, &c., &c., and to thus introduce objectionable impurities into the solution.

The fact that all the cyanogen in zinc double cyanide, on addition of excess of free alkali, can be determined by silver solution, as stated above, may be explained in another way than by assuming the mixture to have formed free cyanide and zinc-potassic oxide; *viz.*, that the reaction does not take place except in presence of another metal, such as silver (and probably gold), which has a superior affinity to zinc for cyanogen—at least, to the extent of the formation of the double cyanide.

I have proved the following reactions to take place on adding silver nitrate solution to solution of double zinc cyanide in absence of free alkali:—

$$ZnCy_2 2KCy + AgNO_3 = AgCyKCy + KNO_3 + ZnCy_2$$

and

$$ZnCy_2 2KCy + 2AgNO_3 = 2AgCy + 2KNO_3 + ZnCy_2.$$

This substitution of silver for zinc might be expected to take place even more readily in presence of free alkali, which, by its affinity for zinc to form the double oxide, might lend its aid in inducing the substitution. I have not tried the use of a solution of a gold salt in place of a silver, but a similar reaction might be expected to take place. These facts may possibly at least partly explain how double zinc cyanide alone, and especially in presence of free alkali, effects extraction of gold and silver from ores.

We may suppose the following reactions to take place simultaneously in presence of caustic alkali:—

$$(a.)\ 2ZnCy_2 2KCy + 2AgNO_3 =$$
$$2AgCyKCy + 2KNO_3 + 2ZnCy_2$$
$$(b.)\ 2ZnCy_2 + 4KHO = ZnCy_2 2KCy + ZnOK_2O + 2H_2O,$$

the potassic cyanide portion of the double zinc cyanide, re-formed as in (*b*), being then acted upon further by the

silver salt, as in (a), this going on until all the zinc has been brought into the form of zinc-potassic oxide. If the caustic alkali is able thus to act as a carrier of cyanogen between zinc cyanide and silver, the fact may perhaps partly explain why the presence of free alkali in residual solutions makes them more efficient for extracting gold and silver than if it were absent.

From thermal data, zinc cyanide appears to be a much more stable compound than silver cyanide. Thomsen gives as thermal values—

$$[Ag_2, Cy_2] = 2{,}790, \text{ and } [Zn, Cy_2] = 53{,}400\,;$$

and thus silver would not be expected to be able to displace zinc from zinc cyanide. Thomsen's thermal values for the double cyanides are—

$$[Ag_2, Cy_2, 2KCy, Aq] = 15{,}780, \text{ and } [Zn, Cy_2, 2KCy, Aq] = 62{,}230\,;$$

from the above the following can be deduced, *viz.*:—

$$[Ag_2Cy_2, 2KCy, Aq] = 12{,}990, \text{ and } [ZnCy_2, 2KCy, Aq] = 8{,}830,$$

the difference, 4,160, being thus in favour of the probability of the reaction—

$$ZnCy_2 2KCy + 2AgCy = 2AgCyKCy + ZnCy_2$$

taking place, and further experiments I have made prove that it does.

I have been unable to find a statement of the thermal value of $[ZnO, K_2O, Aq]$, so cannot get direct evidence regarding the probability, from thermo-chemical reasoning, as to whether or not the reaction—

$$ZnCy_2 2KCy + 2K_2O = 4KCy + ZnOK_2O$$

will take place; however, thermally it would be represented so. Again taking Thomsen's figures—

$$\left\{\begin{array}{lllll} + [Zn, O, H_2O] & + & [K_2, Cy_2, Aq] & + & [ZnO, K_2O, Aq] \\ + \quad 82{,}680 & + & 124{,}680 & + & ? \\ - [Zn, Cy_2] & - & [K_2, O, Aq] & - & [ZnCy_2, 2KCy, Aq] \\ - \quad 53{,}400 & - & 164{,}560 & - & 8{,}830 \end{array}\right\}$$

which would necessitate the thermal value of—

$$[ZnO, K_2O, Aq]$$

being at least 19,430 for the reaction to take place without absorption of heat—a somewhat large quantity for a combination of this sort.

Bettel states that, using phenolphthalein as indicator, and titrating solutions of the following salts with acid; to KCy, the full equivalent of acid requires to be added before the red colour is discharged; to $ZnOK_2O$, acid equivalent to the contained K_2O; and to $ZnCy_2 2KCy$ only 7·9 per cent. of the equivalent of acid requisite for its complete decomposition. Taking these figures, it will be seen that if the reaction $ZnCy_2 2KCy + 2K_2O = ZnOK_2O + 4KCy$ does take place, more acid would be required to be added to the mixture to discharge the red colour than to the two separate solutions used to make the mixture. In several experiments, using various quantities of caustic potash and of double zinc cyanide solutions, it has been found that the quantity of acid required for the mixture has been identical with the sum of the quantities required for the two separate solutions used to make the mixture, from which it would appear that the reaction probably does not take place.

To sum up this matter, either from experiments made or from reasoning, I still find it difficult to form a very decided opinion as to whether this reaction does or does not take place in the case of dilute solutions, but am inclined to the view that it does not.

Analytical Examination of Residual Solutions.—An exact direct method of determination of free alkaline cyanide in presence of double zinc cyanide and also carbonate and caustic alkali, one at least of which is almost invariably present in working solutions, is much required; but although I have made many attempts to find such, I must acknowledge to have failed in hitting upon a single one which can be considered altogether satisfactory when caustic alkali is present. In the presence of carbonate only, the addition of excess of barium chloride or nitrate decomposes this, and the filtrate from the barium carbonate contains now alkaline chloride or nitrate in place of carbonate, which does not interfere with the test. If caustic were also present, a very cautious addition of bicarbonate to carbonate it, followed by precipitation by barium salt as above, might be of some use; but the difficulty here is to avoid adding either too much or too little bicarbonate, leaving thus in the former case excess of bicarbonate, tending to liberate hydrocyanic acid, and in the latter a portion of the caustic uncarbonated.

Although as a rule the partial examination of residual solutions by determinations as indicated above is all that is often made, a fuller examination of them from time to time may afford valuable information relating to their condition and composition, especially after they have been in use for a considerable time; and possibly the cause of extractions of the gold by their use being not altogether satisfactory, when such is the case, might be thereby traced.

We are indebted to Bettel and Feldtmann for recent publications of their investigations relating to the analysis of cyanide solutions, &c.; and in contributing the following notes it is not in any way my desire to question the accuracy of the methods proposed by these chemists, but solely to offer a choice of alternative methods of determination of some of the various constituents of these solutions.

I will assume that the residual solution to be examined may contain any or all of the following, *viz.*, free alkaline cyanide, double zinc cyanide, alkaline ferrocyanide and sulphocyanide, cyanates, urea, salts of ammonia, cyanurates, oxalates, &c., in addition to hydrates, carbonates, chlorides, sulphates, and sulphides of the alkalis. In the first instance I assume the absence of copper compounds, except possibly in very minute quantity, as these interfere considerably with some of the methods of determination referred to below; but in a note at the end of this paper I refer shortly to this interference. Cases in which copper in considerable quantity finds its way into the cyanide solution are certainly not uncommon; in such cases, at least part of the copper will be removed on passing through the zinc extractors; but there are far more cases in which only a minute quantity, if any at all, is in solution.

(1.) *Cyanogen Compounds, &c.*—On shaking up the solution with excess of ferrous hydrate or carbonate and some caustic potash (which must be free from chloride), the cyanogen of the simple and double zinc cyanides is all brought into the condition of yellow prussiate, the reaction being complete in a very short time, thus:—

$$6KCy + FeO = K_4FeCy_6 + K_2O,$$

and

$$3ZnCy_2 2KCy + 2FeO + 4K_2O = 2K_4FeCy_6 + 3ZnOK_2O.$$

The solution is filtered from the excess of ferrous hydrate and divided into two portions, the one being used mainly for determining the yellow prussiate and sulphocyanide separately, and the other for determining yellow prussiate, sulphocyanide, and chloride all together. For these determinations an adaptation of Volhard's well-known method of titration with silver nitrate in presence of nitric acid has suggested itself to me, which for several reasons I prefer to employ to methods in which titration by permanganate is employed, as these latter are often so apt to have the accuracy of the determination of the particular body under consideration interfered with by the presence of other reducing agents in the solution. To the one portion a measured quantity of decinormal silver solution is run in in excess, the mixture acidified with nitric acid to dissolve any hydrate and cyanate of silver, &c. which may have been precipitated, and the remaining precipitate of ferrocyanide, chloride, and sulphocyanide of silver can then be filtered off, preferably through glass-wool or asbestos, and the excess of silver determined in the filtrate by decinormal sulphocyanide, using persulphate of iron as indicator; in place of filtering, the turbid mixture may be made up to a definite bulk with water, shaken up, and the precipitate allowed to settle, and the silver determined in an aliquot portion of the clear supernatant solution, the space occupied by the precipitate being too little to seriously affect the measurement. The necessity for removal of the precipitate at all is owing to the

presence of the silver ferrocyanide, which would react with the iron indicator, and thus interfere with the determination.

To determine the ferrocyanide alone in the other portion of the main solution, it is poured slowly into a warm solution of a per-salt of iron containing some free acid, the Prussian blue filtered off and washed, the precipitate decomposed with caustic potash, the iron hydrate filtered off, and the yellow prussiate in the filtrate determined, either by the silver method as above, or, if preferred, by acidifying and titrating with standard permanganate.

The filtrate from the Prussian blue will contain all the sulphocyanide, and after precipitation of the iron by caustic potash and neutralisation of the filtrate from the iron hydrate, in part of this solution the sulphocyanide can be determined by the method described by Barnes and Liddle (see the Journal of this Society, Vol. 2, p. 122, and also Sutton's book on Volumetric Analysis, p. 177), *viz.*, precipitating as sub-sulphocyanide of copper in presence of a reducing agent with standard copper solution, the presence of any excess of copper being tested for by bringing a drop of the solution in contact with a drop of yellow prussiate solution, a brown coloration forming as soon as there is a trace of copper in excess.

The cyanogen existing in the simple cyanide and double zinc cyanide may be determined by titration of the original solution with silver solution in presence of excess of caustic alkali and iodide of potassium, as indicated above. In carrying out this determination, I find it advisable to use as pure potash as possible, and even with this I have noticed a slight white opalescence form shortly before the yellow cloud, due to iodide of silver being formed; but, as far as I can judge from experiments with pure double cyanide of zinc, this slight white opalescence may be neglected, and the point when it changes to the yellow opalescence (quite a sharp point) be taken as the one indicating full conversion of all the cyanide into double cyanide of silver and potassium. The double zinc cyanide alone can be calculated from the determination of the zinc in the solution; by knowing the total cyanide, including the ferrocyanide, determined as stated above, all in the form of ferrocyanide, and the simple and double zinc cyanide, it is easy to calculate the ferrocyanide which existed as such in the solution being tested.

(1*a*.) An alternative method of determining the above constituents is as follows:—Place a suitable amount of the residual solution in a retort with some zinc sulphate solution; this latter is to ensure sufficient zinc being present to fix all the ferrocyanide, on acidifying, as zinc ferrocyanide, which is practically unattacked by moderately dilute sulphuric acid; then add an excess of dilute sulphuric acid, and distil through a condenser, catching the distillate in excess of caustic alkali; this latter can then be titrated with silver solution, and the whole of the hydrocyanic acid which had existed as simple cyanide and double zinc cyanide is thus determined. If the solution had contained any sulphide, this is preferably removed by shaking up with a little lead carbonate and filtering before placing in the retort. In the still there now remains all the hydroferrocyanic acid as ferrocyanide of zinc in the form of a precipitate insoluble in the weak sulphuric acid, and the sulphocyanic and hydrochloric acids, &c. in the solution. The precipitate is filtered off, decomposed by pure caustic potash, and the ferrocyanide determined by silver by the adaptation of Volhard's method as described above, the sulphocyanic and hydrochloric acids together by Volhard's method, and the sulphocyanic alone by Barnes and Liddle's method referred to above.

(2.) *Carbonates, Oxalates, and Sulphates.*—These can be determined as follows:—Three quantities of the solution are taken, and excess of barium salt added to one, and of calcium salt to the other two. The barium precipitate is treated with hydrochloric acid, and the barium sulphate left undissolved determined in the ordinary way. One of the others is ignited to convert the oxalate into carbonate, and the total carbonate thus produced determined by dissolving in a known quantity of standard hydrochloric acid in excess, and, after boiling off the carbonic acid, determining the excess of acid. The third precipitate is put in a large basin of water, and the carbonate and oxalate decomposed by a small excess of hydrochloric acid, and the oxalic acid determined by standard permanganate in the usual way, warming the solution to promote the reaction.

(3.) *The Bases in Solution.*—These are determined as described in an earlier part of this paper. During the course of making this determination, if desired, an approximate estimation of the total solids in the solution may also be made by evaporation to complete dryness and weighing the residue, though at best this will only give a rough approximation, as certain of the solid constituents of the original solution will have been decomposed or altered during the evaporation, and probably also some portion of them, such as the ammonia compounds, have been volatilised.

(4.) *Sulphide in Solutions.*—It is unusual for more than a small quantity of sulphide to be found in residual cyanide solutions, but there is fortunately a most delicate way of estimating an extremely minute quantity of it rapidly, even in presence of the various other constituents of these solutions. By means of a dilute solution of a lead salt, less than one part of sulphide in 1,000,000 parts of solution can be detected with comparative ease, and an approximate determination of the quantity in a solution as dilute as this be made. There are a variety of ways in which a lead solution of known strength can be employed to effect the determination. Of these I prefer the following:—A convenient strength of lead solution to employ is one of which 100 measured grains is equivalent to 0·01—0·02 of a grain of K_2S. The solution to be tested, if very highly coloured, is first decolorised by any suitable well-known means. This, however, is often unnecessary, a slight tinge of brown not seriously affecting the accuracy of the determination. A rough determination of the sulphide is then made colorimetrically by means of the lead solution, to find out if it contains more or less than about 1 part of sulphide in 20,000 parts. If the quantity is less than this, the solution is still further diluted with recently boiled and cooled distilled water until it contains = only 4—6 parts of potassic sulphide per million, which is about the most convenient strength to test. Two quantities, of 5,000 grains each, of the diluted solution are measured out into two 20-oz. flasks placed on a white surface. Successive quantities of 10 grains or so of the lead solution are added to each flask, until the last addition to one fails to make its contents darker than that of the other, when the sulphide is calculated from the quantity of lead solution employed, omitting this last addition. As each addition is made, it is easy at first to see a brown cloud forming at the point where the lead solution enters the one being tested; when this fails to be noticed, it is an indication that the end point is nearly reached. Working in this way, I find that the sulphide can be determined to within about 2—3 parts per 10,000,000 of solution, even when it is slightly coloured. If the original solution contains more than about 1 part of sulphide in 20,000, a suitable quantity of it, say from 100—1,000 grains, according to the amount of sulphide suspected to be in it, is put into a flask and the lead solution run in until the precipitate cannot be noticed increasing perceptibly; about 1—2 c.c. or so is then filtered through several thicknesses of filter paper, the filtrate generally being of a brown colour, half of it is placed in each of two small test tubes, a drop or two of the lead solution is added to one test tube, and if its contents become darker than that of the other, their contents are returned to the main solution and further lead solution added, and so on until this darkening no longer takes place. Of course, if on making the first test no darkening has taken place, too much lead solution has been added, and a new quantity of the original solution must be taken, and the test repeated, adding a smaller quantity of the lead solution at the first. The presence of the other constituents of the solution appear to have no effect on the accuracy of the determination made in this way.

(5.) *Cyanates, Cyanurates, Ammonia Salts, and Urea.*—From experiments on artificial mixtures of these in presence of cyanides, ferrocyanides, chlorides, &c., I find that their quantities can be approximately determined as follows:—

To the solution, barium nitrate solution in slight excess is added to decompose the carbonates, and cyanurates if

present in moderate quantity; if the presence of caustic soda is suspected, a little carbonic acid water in the least possible excess having been added to carbonate it before addition of the barium solution. To the filtrate from the barium carbonate and cyanurate, silver nitrate solution is run in, if the solution is almost neutral chromate being employed as indicator of full precipitation, if not, a slight excess of silver may be employed. The solution is filtered off and the filtrate is retained for estimation of the ammonia salts and urea, as described below. The precipitate, which may consist of silver cyanide, ferrocyanide, sulphocyanide, chloride, &c., in addition to cyanate, is shaken up with dilute nitric acid to dissolve the cyanate, and the quantity of silver in solution determined by Volhard's method, after removal of the insoluble portion of the precipitate, and the cyanic acid calculated from it. If cyanurate is suspected to be present, the above can be repeated, using calcium nitrate in place of barium nitrate; calcium cyanurate, being soluble in water, will then be in the filtrate from the carbonate precipitate, and can be determined along with the cyanate, as above. To the original filtrate from the total silver precipitate a little common salt is added, if excess of silver had been employed, to remove this excess, the silver chloride filtered off, and the filtrate placed in a retort and distilled, with a slight excess of caustic potash present, for a comparatively short time, keeping the liquid just barely boiling, the distillate being caught in a measured quantity of $\frac{1}{10}$ normal hydrochloric acid.

The ammonia in the distillate is then determined by nesslerising, or by titration of the acid left unneutralised. If the distillation is carried out at a low enough temperature, and is not too prolonged, and no great excess of caustic potash is employed, little, if any, of the nitrogen of the urea passes over as ammonia with the distillate, most of the urea remaining undecomposed; and its quantity can be determined in the solution left in the retort by the well-known method of decomposition by hypobromite of soda and measurement of the nitrogen evolved; or, if preferred, the ammonia and urea can be determined together by this method in a portion of the filtrate from the silver precipitate.

(6.) *Alkalinity*.—It is advisable to determine the alkalinity of the stock of working solution from time to time. This may be done, comparatively, by titration in the cold with acid, employing some one selected indicator; but, as Bettel has pointed out, the point of neutralisation of the various constituents of the solution differs with certain indicators, and therefore fuller information as to the character of the alkalinity may be obtained by making several titrations, using different indicators; also by titration before and after precipitation by barium or calcium salt, and removal of the precipitate. It may also be sometimes useful to determine the quantity of dilute acid consumed in decomposing certain constituents of the solution, by warming with an excess of it and titrating that remaining.

Note on the Effect of Copper in Solution.—As mentioned before, the presence of any considerable quantity of copper in solution tends to impair the accuracy of some of the methods of determination described above; this applies specially to those given in paragraph (1), *viz.*, the determination of the simple and double cyanides, as distinct from ferrocyanide, and also of the sulphocyanide. The reason for this is that cuproso-cyanide of potassium behaves in many ways differently towards certain reagents to zinc potassic-cyanide; for example, it does not yield ferrocyanide of potassium when treated with ferrous hydrate and caustic potash; its hydrocyanic acid cannot be determined directly by titration with silver solution, either in presence or absence of caustic potash; it is decomposed and gives off its hydrocyanic acid when distilled with dilute sulphuric acid, though not so readily as zinc double cyanide; if, however, any ferrocyanide, even of zinc, is present in the retort, the copper as set free from the cuproso-cyanide will displace the zinc and form a ferrocyanide of copper; ferrocyanide of copper is generally taken to be insoluble in dilute sulphuric acid, but it does not in fact resist its action when boiling nearly so fully as zinc ferrocyanide does, but is very slowly decomposed by it, giving off hydrocyanic acid; this decomposition is, however, so slow that to obtain all the hydrocyanic acid from it by distillation with weak acid entails prolonging the distillation for an almost impractical length of time; were this not so, the the addition of a copper salt to ferrocyanide of zinc and distillation of the mixture with weak acid might be employed as a means of obtaining hydrocyanic acid from the latter. The hydroferrocyanic acid in combination as a ferrocyanide of copper can, however, be determined just as readily and accurately as that in ferrocyanide of zinc by the adaptation of Volhard's method of titration with silver solution referred to above, the copper hydrate being filtered off after the decomposition by caustic alkali. The total hydrocyanic acid can, however, be determined, and also a roughly approximate separation of the hydrocyanic acid existing as simple and double cyanide from that existing as ferrocyanide be made, by distillation with weak sulphuric acid as described in paragraph (1*a*) above (a little of the copper ferrocyanide formed, if ferrocyanide is also present, probably being decomposed during the distillation). The cyanide found in the distillate will thus correspond to a little more than that which actually existed in the solution as simple and double cyanide, and the hydroferrocyanic acid estimated in the insoluble ferrocyanides left in the retort, by the silver titration method given above, to a little less than that which actually existed as ferrocyanide in the original solution.

I am still experimenting with solutions containing copper, with a view to arriving at some modifications of the analytical processes which will allow of the various determinations being made, in the case of such solutions, with the same degree of accuracy as if little or no copper were present.

In conclusion, I may remark that many of the points touched upon in this paper would probably afford suitable subject-matter for further investigation; and in reference to the latter part of the paper, *viz.*, that dealing with the analytical examination of these residual solutions, considering how complex these solutions often are, I think absolute accuracy in the determinations of all the constituents can hardly be looked for, except perhaps by the employment of long and tedious processes. The above scheme of examination only aims at allowing of the composition of such solutions being determined with a fair degree of accuracy, more or less expeditiously, and I trust it may at least prove of some little use to chemists employed in the industry of gold extraction by the cyanide process.

Discussion.

Mr. Alfred James remarked that some months since he had received a notice from the Executive of the Section calling for suggestions as to subjects for papers or for special investigation, and that he had at that time felt strongly inclined to suggest that special information as to the technical analysis of cyanide solutions was very urgently needed. He therefore deemed it a matter for special congratulation that the Society should have had such a paper presented to it as that to which they had just listened; the more so, as Mr. Ellis had been one of the earliest and foremost investigators into the theory and practice of extraction of gold by cyanide solutions, and had done a vast amount of work on the subject.

He had to confess, however, to a slight sense of disappointment, that whilst the author had given them some very interesting information as to the behaviour of the double cyanide of zinc and potassium in the presence of excess of caustic potash, and as to the similarity of the behaviour and reactions of this double cyanide to those of free cyanide of potassium, yet the paper did not supply any method for the determination of these two salts in the presence of each other.

This was a most important matter to the chemist in charge of a cyanide works. Unless he could tell how much of his nitrate of silver test was due to the free cyanide present, and how much to the double cyanide of zinc and potassium *plus* caustic potash, he had no means of knowing, beyond the actual weight of free cyanide added to the stock solutions, the amount of the latter actually present in his solutions. Mr. Ellis had shown them that the double-

cyanide *plus* caustic potash would give extractions of fairly high percentage, though perhaps inferior to those obtained by the use of simple cyanide alone; and he thought there was no doubt that in general practice it was found that the double cyanide was less effective, owing perhaps to impurities in works solutions.

Of course, if the equation adopted by Bettel was actually correct, and—

$$K_2ZnCy_4 + 4KHO = K_2ZnO_2 + 4KCy + 2H_2O,$$

then of course all the double cyanide was converted into free (or simple) cyanide, with the formation of oxide of zinc and potassium; but he was unable to accept this equation; for, although it had been proved that the double cyanide *plus* caustic potash (or soda) had a considerable action on gold, the regeneration theory was not necessary to account for this. One could expect that there would be some such action, as, if the silver in the $AgNO_3$ had the power to displace zinc from the double cyanide of zinc and potassium, gold in the presence of free oxygen might have that power also; thus:—

$$K_2ZnCy_4 + 2Au + 2KHO + O = 2KAuCy_2 + K_2ZnO_2 + H_2O.$$

To settle this point of "regeneration," if possible, he had made some experiments, and as direct analytical methods were inadmissible, owing to the similar behaviour of the two substances, he had tried the effect of dissolving molecular weights, as in the above first-mentioned equation, and crystallising out the product from the saturated solution.

As a result he had found that the solution formed had, on heating, not given out the slightest odour of HCy, so noticeable with cyanide solutions, and that the deposited crystals on being analysed were almost pure double cyanide of zinc and potassium (K_2ZnCy_4), and that they contained almost all the Cy originally taken; and he was therefore confirmed in the conclusion that with saturated solutions, at any rate, double cyanide of zinc and potassium and caustic potash did not combine to form free cyanide of potassium.

Having, however, got thus far, he thought it might be of advantage to test the converse equation, and see if free cyanide of potassium and oxide of zinc and potassium combined to form the double cyanide and caustic potash:—

$$K_2ZnO_2 + 4KCy + 2H_2O = K_2ZnCy_4 + 4KHO.$$

He accordingly took the molecular quantities shown in the above equation, but found some difficulty in forming the oxide of zinc and potassium, and had to use an excess of caustic potash. On crystalling out, however, he got all the zinc down as pure crystals of double cyanide of zinc and potassium; thus showing that, at least with saturated solutions and excess of cyanide of potassium, the latter is the correct equation, and that in place of free cyanide being formed by the combination of double zinc potassic cyanide and caustic potash, free cyanide actually combined with the oxide of zinc and potassium to form the double cyanide.

In conclusion, he ventured to hope Mr. Ellis might be induced to lay before the Society a further account of his investigations into this matter.

Mr. Beilby asked if Mr. Ellis could tell to what extent the dissolved oxygen in dilute solutions was used up in the wasteful oxidation of cyanide.

The Chairman said he had recently seen it stated that the action of an aqueous solution of potassium cyanide on gold in presence of atmospheric oxygen really takes place in two stages, and that one of the products of the first reaction was hydrogen peroxide. It was also suggested that as hydrogen peroxide converts hydrogen cyanide into oxamide, this formation of the peroxide during the dissolution of the gold would account for part of the loss of cyanide during the process. This seemed a point of some interest, and he would be glad if Mr. Ellis could give any further information.

Mr. Ellis said, in reply, that he quite agreed with Mr. James as to the want of a speedy and at the same time accurate method of determination of simple alkaline cyanide, as distinct from double zinc cyanide, when both were present in solution along with caustic alkali. By the crystallisation experiments to which Mr. James had referred, he thought that the former had settled the point that, in strong solutions, zinc double cyanide and caustic alkali together did not react to form simple alkaline cyanide and zinc potassic oxide, and, in fact, that the converse did take place; this, however, seemed to be a reaction which might depend much on the degree of dilution of the mixed solution, &c.; but that in any case these experiments of his offered one more piece of evidence in favour of the view that this reaction probably did not take place.

In reply to Mr. Beilby's question, he stated that he had not yet made any series of experiments on the point.

In reply to Prof. Henderson's remarks, he said he was unaware whether or not hydric peroxide was formed in the solutions in the way stated; if so, it might of course react as suggested to form oxamide, or possibly also sulphocyanide, if any lead sulphide were also present, as pointed out by Bettel and Feldtmann in another connection. It might thus be one factor in the consumption or destruction of cyanide, though he hardly thought one of any great practical importance.

New York Section.

Chairman: Charles F. Chandler.
Vice-Chairman:

Committee:

M. Alsberg.	E. G. Love.
T. Lynton Briggs.	Thos. J. Parker.
G. T. Bruckmann.	Wm. Jay Schieffelin.
Virgil Coblentz.	R. C. Schüpphaus.
H. Endemann.	J. H. Wainwright.
W. F. Fuerst.	D. Wesson.
Jas. Hartford.	

Hon. Treasurer: R. C. Woodcock.
Hon. Local Secretary:

Meeting held on Friday, January 22nd, 1897.

MR. R. C. WOODCOCK IN THE CHAIR.

The Chairman read the following letter from Dr. Edw. Schunck, F.R.S., President of the Society, to Dr. Chandler:—

"Dear Sir—

"As President of the Society of Chemical Industry, I may address you as Chairman of the New York Section of our Society without ceremony or any formal introduction. I wish you to be the vehicle of conveying to the members of your Section my sentiments of esteem and my best wishes for their happiness and prosperity during the year that is just commencing. I wish that during my Presidency it had been possible for me to cross the Atlantic and pay you a visit in New York; but at my age (I am now in my 77th year) this could not be. On the other hand, I shall be delighted to see you, and as many of the members of your Section as may find it convenient to come to our General Meeting next July in Manchester, where you and they will meet with a cordial reception. Please accept my best wishes for yourself individually, and believe me,

"Yours sincerely,

"Edward Schunck."

THE ANALYSIS AND CHEMISTRY OF ASPHALT.—II.

BY H. ENDEMANN, PH.D.

In my previous communication upon the same subject, I have shown that the old methods of analysis of paving asphalts (see this Journal, xiv. 315, 524, 960, and xv. 222, 298) are at fault by not giving us a correct idea of their composition. I have shown further that these asphalts consist of a compound containing oxygen and a series of

inert hydrocarbons. For the first I have adopted Boussingault's name "asphaltene," for the reason that it is the main constituent of the asphalt, though Boussingault's asphaltene was a different body and really a product of oxidation formed only during the analysis and not pre-existing in asphalt. For the second I have also adopted Boussingault's name "petrolene," for the reason of identity.

While the asphaltene is a somewhat easily changed body and entirely non-volatile and hard and brittle in the cold, the petrolenes were found to be comparatively stable compounds, volatile and liquid. They serve to soften the asphaltene and give it the desired elasticity, which makes asphalt so useful a material for paving purposes. I have also shown that the asphaltene of different asphalts has the same composition and identical properties, inasmuch as it will oxidise when heated in air to about 250° C., and form a body which is the same in all cases and evidently identical with Boussingault's mistaken asphaltene.

I have called this body, which I have since obtained from a series of other asphalts, "asphaltic acid," for the reason that I consider it the cause for the solubility of metallic oxides in chloroform, considering such solubility to be due to the formation of a salt. Its preparation is simple, and consists in heating asphalt bitumen or asphalt in air to a temperature of 250° C.

Asphaltene is at first liquid. It soon covers with a wrinkled skin, which has to be broken from time to time by stirring, to allow the air free access to the material. When the liquid has nearly all disappeared the mass becomes at 250° C. elastic like rubber, but soon after hard and brittle, and when repeatedly pulverised, hard and dry like sand. This is the stage when heating may be interrupted. Further heating will not change the product unless the temperature limit of 250° C. is grossly transgressed.

If this body is heated with caustic soda solution it dissolves, forming a deep brown liquid, which, on addition of an acid, gives a brown precipitate. This process of dissolving is an exceedingly slow one. Continued for weeks only a small portion of the material could be brought into the solution. Asphaltic acid I therefore had looked upon as an anhydride until the analysis of the body suggested another procedure for its preparation.

The analysis proved conclusively that the body was not the product of hydration only, but a product of oxidation. Hence the solution was effected thereafter by dissolving in soda solution, while a current of air was drawn through the solution. What could not be accomplished in weeks was then done in days.

The brown solutions obtained were precipitated by hydrochloric acid, the precipitate brought upon a filter and washed. For further purification it was re-dissolved in ammonia and re-precipitated, filtered, and washed.

The free acid is soluble in pure water, but insoluble in water containing salts. It is a strong acid, dissolving in ammonia with decided development of heat. The acid and salts are all dark brown.

Two elementary analyses were made of this body—(*a*) acid dried below 100° C.; (*b*) dried at 125° C.

—	(*a*.)	(*b*.)	Calculated for $C_{26}H_{20}O_{12}$
Carbon	57·09	59·40	59·54
Hydrogen	4·37	3·86	3·82
Oxygen	38·54	36·74	36·64

The ammonia salt, evaporated until odour of excessive ammonia is no longer noticed and then precipitated by sulphate of copper, produces a copper salt containing 15·95 per cent. CuO.

If evaporation is continued until some free acid is formed, *i.e.*, when the mass has become dry and the undissolved free acid thus formed has been removed by filtration of the water solution, the copper salt contains 12·0 per cent. CuO. Hence, one was perhaps a basic salt, while the other contained free acid. The free acid was then titrated with $\frac{1}{2}$ normal soda solution. The dark coloured solution prevented the use of an indicator in the solution. It had therefore to be neutralized by the use of litmus paper. But even then the colour of the solution interfered to such extent that it was not possible to get nearer to the point of neutralisation than about $\frac{1}{20}$ of all the quantity of soda solution required. All that could be seen by this test was that the neutral reacting salt corresponded about to a copper salt containing 13·9 per cent. of oxide of copper (calculated).

The point of neutralisation had to be guessed at by determining the point of actual acidity and actual alkalinity and putting the point of actual neutralisation arbitrarily at the average. These salts are not well suited for determination of actual molecular weight.

I have called attention to the fact that the conversion of asphaltic acid to this new acid—which I will call asphaltulmic acid—by means of soda is a very tedious operation, and, in fact, I have never carried the reaction so far as to dissolve all asphaltic acid. The difference in composition—

$C_{26}H_{20}O_4$ for asphaltic acid, and

$C_{26}H_{20}O_{12}$ for asphaltulmic acid,

might suggest that the substance called asphaltic acid were a mixture of a substance containing but little oxygen and another very much richer. Though I was always able, no matter when the process was interrupted, to again bring new material into solution by continuing the treatment, showing thereby that the substance still had the same property, I considered it safer to determine the composition of the residue, and thus affirm my belief into the uniformity of the substance called asphaltic acid.

0·1578 gave 0·0984 H_2O and 0·4484 CO_2—

Carbon	77·50
Hydrogen	6·92
Oxygen	15·85

which is the composition of asphaltic acid. This fractional treatment is also one of the best proofs of the uniformity of this substance. In fact, it has so far been the only one, inasmuch as other proofs are hardly available. The substances have a high atomic weight. Crystallisation as a means of separation cannot be used, inasmuch as all salts are amorphous. The ammonia salt and other soluble salts dry like gums, so does the free asphaltulmic acid. The free acid is insoluble in alcohol.

Altogether the hope of using this compound for the purpose of revising the assumption giving to the formulæ of these compounds 26 atoms of carbon had to be relinquished.

Soft and Liquid Asphalts or Maltha.—It is a well-known fact that besides the more or less hard paving asphalts, liquid asphalts are found, which, with characteristic asphalt odour, nevertheless show properties not in keeping with the composition of those previously mentioned.

They are more or less liquid and contain but little oxygen, or, in some cases, none at all. To this class belong the malthas. The investigation made in this direction was carried out on a Texas liquid asphalt, which on examination had been found to be entirely free from oxygen. It was therefore free from asphaltene. When such heavy oils are evaporated, it is with great loss, for such will lose on refining to harden as much as 80 per cent. of oil before they really become as hard as consistency of asphalt for varnish manufacture, and then only when absorption of oxygen is possible. The loss in current of carbon dioxide is quick at first, slower later, but it lasts a long while. When the stage had been reached that a slow continuous loss at 250° C. is noticed, the residue was analysed. The residue was found to be $C_{26}H_{36}$ with a varying but small quantity of oxygen. In two different treatments the following results were obtained:—

Carbon	87·77	86·89
Hydrogen	10·23	10·36
Oxygen	2·00	2·75

It is somewhat doubtful where in these cases the oxygen came from, inasmuch as the bitumen from which they had

been obtained was entirely free from oxygen. For instance, 0·1812 gave 0·1821 water and 0·5919 carbon dioxide.

Carbon	89·07
Hydrogen	11·16

Other analyses gave a like result.

Either the commercial carbon dioxide had contained oxygen or this hydrocarbon is capable of decomposing CO_2 at 250° C.

The former is the more probable, however. So low a percentage as 0·05 per cent. of oxygen, which is considered sufficient for causing the change is not easily found in carbon dioxide gas by an ordinary test, such as I have had occasion to make quite frequently.

I have not investigated this point as yet, inasmuch as it would have led me from the subject-matter of this investigation, which in the first place meant the establishment of a safe and rational method of analysis.

Inasmuch as the absorption of oxygen may have been preceded by a loss of hydrogen, I prepared this body on a different plan. I distilled the petrolenes from a larger quantity of asphalt in the vacuum. I then extracted the residue in the retort repeatedly with much boiling alcohol. The remainder was dissolved in ether, the ether removed and residue analysed.

0·1918 gave 0·1928 H_2O and 0·6363 CO_2—

Carbon	89·08
Hydrogen	11·00

calculated for $C_{26}H_{38}$.

Carbon	89·14
Hydrogen	10·80

The substance is a gum, entirely inodorous and in colour much lighter than asphaltene.

If we heat the asphalt bitumen of such asphalts in air, we find that they first lose rapidly in weight on account of the loss of petrolene, and that later on they gain in weight.

4·635 grms. after seven hours' heating in air-bath to 250° C. yielded 3·070, and after nine hours' further heating, 3·140 grms.

The liquid after the first rapid evaporation became covered with a skin, which, when pushed aside, gradually agglomerated to an elastic mass, and finally became hard and brittle.

The substance which was left behind was found to be asphaltic acid.

0·1739 gave 0·0982 water and 0·498 carbon dioxide—

Carbon	78·09
Hydrogen	6·27
Oxygen	15·64

It could be converted into asphaltulmic acid by soda solution.

This shows that these asphalts contain a hydrocarbon $C_{26}H_{38}$, which is capable by oxidation to pass into asphaltene and asphaltic acid; and furthermore, that this substance possesses a great affinity to oxygen, inasmuch as it will take it from such commercial carbon dioxide directly as will not pass any oxygen to asphaltene. I call this hydrocarbon "asphaltogen." Asphaltogen must be looked upon as an asphaltene preservative, in so far that as long as it is present the oxidation of asphaltene to asphaltic acid can be prevented.

This asphaltogen is a somewhat volatile hydrocarbon. Bitumens free from it will oxidise at 250° C. in a porcelain dish without discoloring the porcelain, except where the liquid comes in contact with it. If asphaltogen is contained in bitumen, the porcelain dish becomes coated above the surface of the liquid with a black mass of asphaltic acid. This behaviour may be looked upon as a test of the presence of asphaltogen.

It can thus be shown that the petroleum residuum oils contain asphaltogen (25 per cent.). (See this Journal, 1896, XV. 267).

Asphaltogen is solid at ordinary temperature, but melts already in the water-bath. It is soluble in ether, wherein asphaltene is insoluble.

Analysis of Soft Asphalts.—To determine asphaltogen in presence of asphaltene we may proceed as follows:—

Keep the bitumen for a considerable time at a temperature of about, but below, 200° C. At this temperature but little asphaltogen will evaporate, but it will readily pass into asphaltene. Then raise the temperature and convert into asphaltic acid. Thus we obtain the asphaltic acid obtained from asphaltogen as well as from asphaltene.*

Determine then the quantity of oxygen in the untreated bitumen, and calculate therefrom the asphaltene present (8·12 per cent. = 100 asphaltene), 380 asphaltene yield 402 asphaltic acid.

The whole asphaltic acid—
− Asphaltic acid from asphaltene
= Asphaltic acid from asphaltogen
402 Asphaltic acid = 350 asphaltogen.

Then bitumen—
− (Asphaltene + asphaltogen)
= Petrolene.

Review and Conclusions.—The formulæ of the substances, which have been described in this paper may require revision, inasmuch as I have so far not been able to obtain any compound which could be used for an accurate determination of molecular weights. Yet for practical purposes they will be found accurate enough for calculation of results. The substances described are—

$$\left.\begin{array}{ll} \text{Asphaltogen} \ldots\ldots\ldots\ldots & C_{26}H_{38} \\ \dagger\text{Asphaltene} \ldots\ldots\ldots\ldots & C_{26}H_{36}O_2 \\ \text{Asphaltic acid} \ldots\ldots\ldots & C_{26}H_{26}O_4 \\ \text{Asphaltulmic acid} \ldots & C_{26}H_{20}O_{12} \end{array}\right\} = C_{24}H_{18}O_8(CO_2H)_2.$$

† Melting point 210° C.

The first two are the useful constituents of natural asphalt; the latter two are products of oxidation of the first and not found in natural asphalts, except perhaps as products of accidental degeneration. Asphaltogen absorbs oxygen at elevated temperature with great avidity; asphaltene absorbs oxygen likewise, but not so easily. Crude asphalts containing asphaltogen may be oxidised so that they will contain asphaltene. Asphalts containing both will, by heating in air, become poorer in asphaltogen and richer in asphaltene. Asphaltene will oxidise only after the asphaltogen has been oxydised, the latter will therefore wield a protective influence upon the former. In smelting asphalt for the preparation of asphalt cement and for softening, oils containing asphaltogen should be added. Such oils are the malthas or petroleum residuum oil. The former are to be preferred on account of the nature of the petrolenes, i.e., the oils which will soften the asphaltene.

If we mix and heat asphaltic acid with petroleum residuum oil, or with petrolene from asphaltum, we obtain mixtures like sand and water.

If we mix and heat asphaltene with petroleum residuum oil, we obtain a somewhat plastic mixture.

If we mix and heat asphaltene with maltha petrolenes, we obtain a real asphalt.

This leads us to the consideration of the composition and nature of the substances which we call petrolene.

The hydrocarbons of petroleum residuum oils are to a large extent paraffins. But petroleum residuum oils form with asphaltene not advantageous mixtures; hence we must conclude that the petrolenes of asphalt cannot be paraffins—at least not predominantly.

Asphaltogen is a hydrocarbon of the general formula C_nH_{2n-14}.

From the analyses which I have made we can conclude that the petrolene hydrocarbons must contain more H than is contained in the easily oxidisible asphaltogen.

The indications derived from physical properties and chemical tests are for hydrocarbons of the general formula C_nH_{2n-4}, with perhaps others containing more hydrogen and nearing more and more the paraffins (C_nH_{2n+2}). To determine the series to which these hydrocarbons belong an elementary analysis of the substances is not sufficient, but will have to be supplemented by determinations of the vapour densities of the carefully purified products.

The liquid asphalts are best suited for the separation of the petrolenes, for the reason that they are richest. There is, however, a possibility that the petrolenes contain some asphaltogen, though the boiling points of asphaltogen and

* This method is applicable for the analysis of paving asphalts.

the petrolenes are far apart. In order to obtain them from Texas asphalt we proceed as follows:—

At first the water is driven out in an open casserole. Asphalt containing water foams to such an extent that it is hardly safe to undertake the boiling in a vessel more than a quarter filled. With the water some petrolenes are lost. When the foaming ceased 9 per cent. had been lost. The liquid mass is now filled into a retort, and carefully distilled over the naked flame under a partial (half) vacuum. The distillation, at first quite brisk, all at once stops, when the asphaltogen remains in the retort. Thus about 27 per cent. petrolenes could be collected. To learn something about their nature they were distilled under ordinary pressure with thermometer. Evenly with the time, the thermometer rose. Commencing from about 260° C. to 320° C. about one-third had passed over. These lower petrolenes possess a very strong asphalt odour. The higher petrolenes are almost odourless, unless rubbed between the hands, when they also develop the same odour, but much weaker.

The higher petrolenes were not distilled, for the reason that the thermometer commenced to work irregularly. Enough, however, had been learned, namely, that what we call petrolene is a complicated mixture. It contains a series of compounds, perhaps not even all belonging to a single of the aliphatic series, but comprising representatives of several such.

I have to leave this subject for a later communication, as likewise the relation of some petrolene compounds to asphaltogen, and the occurrence of nitrogenous and sulphur compounds with the petrolenes.

Remarks on Analysis of Paving Asphalts.—In my first paper I have shown that asphaltene is soluble in a petroleum ether solution of petrolene. Asphaltic acid in combination with bases is soluble in a chloroform solution of asphaltene. It is more soluble in a concentrated solution than in a dilute solution. We meet, therefore, also here the mistaken observation that "the fine silts will pass through the filter," only that in this case not only organic matter but really mineral matter will be found in the sediment. If this sediment be kept by itself and is examined, asphaltic acid, and from this asphaltulmic acid, may be separated and produced.

Much of the organic matter—not bitumen—found in refined asphalts is asphaltic acid in combination with the mineral matter. In the determination of total bitumen in paving asphalts it is indicated to decant or filter from any precipitate which may be produced by the addition of the chloroform washings to the first chloroform extract, inasmuch as the asphaltic acid and its compounds can no longer, and should not be, counted with the bitumen.

After concentrating the extract, and for the purpose of driving off the last traces of chloroform, we heat in an air-bath for a certain time (see this Journal, 1896, 874). I wish to add here that it is advisable to lay the flask containing the extract sideways, for the purpose of aiding the escape of the heavy chloroform vapours, especially if one desires to effect the escape of the chloroform within the prescribed time.

Practical Application of Analytical Results.—I have shown that in the analysis of asphalts by the old method, using petroleum ether as a solvent for the petrolene, not only the petrolene but a far greater quantity of asphaltene go into the solution. In the case of the analysis of the Mexican asphalt it has been seen that this so-called petrolene consists of—

26·51 petrolene, and
67·61 asphaltene.

All liquid asphalts containing petrolene and asphaltene in this proportion, down to those which contain no asphalttene or asphaltogen at all, give by this method identical results. Hence such results cannot give us any hints as to the applicability of these more or less liquid asphalts for the production of compositions of standard properties and consistency, while the results of analyses made according to my plan can be applied at once. For instance, it is proposed to imitate the bitumen of refined Mexican paving asphalt containing—

$$\left.\begin{array}{l}71 \text{ asphaltene} \ldots\ldots\ldots\ldots\ldots \\ 27 \text{ petrolene} \ldots\ldots\ldots\ldots\ldots\end{array}\right\} 98 \text{ bitumen};$$

by using Trinidad asphalt containing—

$$\left.\begin{array}{l}50 \text{ asphaltene} \ldots\ldots\ldots\ldots\ldots \\ 8 \text{ petrolene} \ldots\ldots\ldots\ldots\ldots\end{array}\right\} 58 \text{ bitumen};$$

and a maltha containing—

$$\left.\begin{array}{l}56 \text{ asphaltene} \ldots\ldots\ldots\ldots\ldots \\ 38 \text{ petrolene} \ldots\ldots\ldots\ldots\ldots\end{array}\right\} 94 \text{ bitumen};$$

then we find by a simple calculation that we have to mix these constituents in the following proportion:—

100 maltha,
149 Trinidad asphalt,

and for the purpose of making of this cement a 15 per cent. paving mixture we require for—

1,000 cement.
3,833 sand and limestone.

The correctness of the above calculations has been practically tested.

In working on a large scale, allowance must be made for oxidation of asphaltene and volatilisation of petrolene by the determination of a factory coefficient.

Discussion.

The Chairman said it was somewhat extraordinary, when one remembered that asphalt was undoubtedly used in the early days of the Egyptians, that the papers that Dr. Endemann had given them undoubtedly contained more chemistry and valuable information than all those that had been published before. If one searched through chemical journals, whether analytical or manufacturing, the knowledge one could obtain on this subject was very limited.

In Dr. Endemann's former paper all were struck by the statement that such very large quantities of mineral matter were extracted by chloroform; and he (the Chairman) took occasion to remark then that he thought it was most likely that the whole of this mineral matter must be in organic combination; and Dr. Endemann now stated that this chloroform-extracted mineral matter was probably combined with asphaltic acid. If he remembered correctly, Dr. Endemann stated in his previous paper that the chloroform was used directly on the crude asphalt as a means of extracting certain products, and with that chloroform extract he got these large quantities of mineral matter. Now, Dr. Endemann told them that this asphaltic oxide was only produced after the asphalt had become oxidised, and that it was not contained in any considerable quantity in the original asphalt. If such were the case, therefore, how was it that the original asphalt gave so much mineral matter in its chloroform extract? Dr. Endemann stated that this asphaltic oxide was not very soluble in chloroform. If this oxide (or acid, as he also termed it), when combined with mineral matter, dissolved to a large extent in chloroform, it seemed very extraordinary that the oxide, which was probably free anhydrous acid, should not dissolve in the chloroform to any considerable extent. There was one other question he would like to ask, and that was whether the asphalts that the author had been examining as they arrived at the present day contained any considerable quantity of arsenic?

Mr. Clifford Richardson said Dr. Endemann seemed to have neglected a very important element in the asphalts—the amount of sulphur present.

Dr. W. J. Schieffelin asked whether Dr. Endemann had proved his process of analysis on any mixture made up of known quantities of the different ingredients to which he had alluded.

Mr. Clifford Richardson said he had noticed in the last number of the Journal of the Society, in a paper by Dr. Endemann, at the reading of which he was unable to be present, that he gave an analysis of petrolene and asphaltene, so called, in which he included quite a large amount of oxygen. He (Mr. Richardson) was unaware that there was any oxygen in these substances, and he did not understand how Dr. Endemann found that there was any there. What were known as petrolene and asphaltene in the technical sense in which he first introduced the terms some seven or eight years ago, were substances obtained by the use

of different solvents on asphalt, the determination of which was convenient merely as denoting, from the relative amount of each found, the character of different asphalts, in exactly the same way that persons dealing in crude petroleum denoted its character by the relative amount of gasolene, burning and lubricating oils which could be obtained from it by distillation. Petroleums were distinguished in this way. They must also have some way of distinguishing asphalt technically, and they found it convenient and accurate to do so by determining the relative amounts soluble in naphtha and in carbon bisulphide or chloroform. There was no claim at all that there was any sharp separation of hydrocarbons by these solvents. It was only a technical one. A certain amount of the higher hydrocarbons, which they had called asphaltene, might go into solution in the naphtha, and a certain amount of the lower hydrocarbons might remain in the chloroform. The method was of analytical accuracy, and of value in the comparison of the properties of different asphalts. If they analysed the hydrocarbons petrolene and asphaltene, making an ultimate combustion, they found that they consisted of a certain amount of carbon and hydrogen in each case, but that the percentages of the two did not add up to 100. Carrying the examination still further, they found that in the petrolene, that is, in the hydrocarbons soluble in naphtha, there was about 3·8 per cent. of sulphur. Now, if that percentage of sulphur were added to that of the carbon and hydrogen it would bring the analysis up to 100—at all events in the case of Trinidad asphalt. In the same way with the hydrocarbons extracted with chloroform, after previous extraction with naphtha: if they determined carbon and hydrogen in them, they found that the percentages added up to even less than that obtained in the case of petrolene. That is to say, there would be a lack of as much as 10 per cent. from a total of 100. But the sulphur in these hydrocarbons also served to make up the necessary 10 per cent.

This was one of the most important features in connection with the hydrocarbons of asphalt. There were no asphalts anywhere, so far as he knew, which contained no sulphur, and as far as his experience had gone, none of the bitumens contained oxygen. Trinidad asphalt as refined, however, contained oxygen in the organic matter which was insoluble, and not of a bituminous nature. Sulphur was a very important thing in the presence of hydrocarbons, because of its relation to one of the interesting reactions of organic chemistry. If they heated with sulphur two molecules of a hydrocarbon—diphenylmethane, for instance,—two atoms of hydrogen were removed in combination with the sulphur to form hydrogen sulphide, and at the same time the two atoms of diphenylmethane united, or were condensed to form tetraphenylethylene. This reaction was well illustrated in the preparation of an artificial asphalt called "Pittsburg flux." It was made from the "residuum" of petroleum by heating it with sulphur. The sulphur removed some of the hydrogen atoms in the paraffin and olefine hydrocarbons, hydrogen sulphide was evolved, and the "residuum" was transformed into a substance of an asphalt type The presence of sulphur was therefore a very important element in the case which was under consideration, and he thought that Dr. Endemann had neglected one of the essential features of the chemistry of asphalt He would be glad to know if the author got his oxygen, in his analyses of petrolene and asphaltene, merely by difference. In the case of Trinidad and Bermudez asphalt, the bitumens contained no oxygens, but rather sulphur.

When they came to consider what petrolene and asphaltene were, they were not such mysteries after all. The petroleums of Ohio and Pennsylvania, as investigated by Mabery, consisted of hydrocarbons of the paraffin and the olefine series. They knew also, from the elaborate work of the Russian chemists, that the hydrocarbons of Russian petroleum were largely of the hexahydrobenzol series. It was with the asphalts exactly as it was with the petroleums; they must separate the hydrocarbons and sulphur derivatives in them, and determine what the series of hydrocarbons was to which they belonged. Asphalt was really nothing but another series of hydrocarbons, just like petroleum. In fact it was a petroleum in the widest sense of the word, since petroleum and asphalt merged into one another by gradual gradation.

Recent investigations which he had carried on showed that the most volatile hydrocarbon extracted by naphtha from Trinidad asphalt was probably one of the members of the hexahydrabenzol series. The first thing that attracted attention in obtaining the lighter oils from asphalt was that they had an enormously high specific gravity for their molecular weight. They could determine their molecular weight by the boiling or freezing point method, and could also control it by the density and index of refraction of the substance, according to the methods of Bruhl and Traube. This physical determination, which Dr. Endemann seemed to have neglected, showed at once that the lower members, at least, of the series of asphaltic hydrocarbons contained a closed ring, and that they were probably higher members of the hexahydrabenzol series, or something more complicated. There was no reason, then, why they should not continue the investigation in the same manner from this point through the higher members of the series of asphalt hydrocarbons and their sulphur derivatives. There was no necessity for all these empirical formulæ which Dr. Endemann had given, and which seemed to have very slight foundation upon a scientific basis. There were other methods for investigating asphalt, of a more scientific nature.

Dr. Endemann said, in reply, that the asphaltic acid was mainly produced during the refining process by either faulty treatment or unavoidable oxidation from asphaltene. The asphaltic acid might, in the presence of bases, saturate itself, for there was generally more or less inorganic matter present. He should not be surprised if asphaltic acid went into the solution of asphaltene in chloroform likewise, just the same as in combination with bases. He had not investigated this point, nor that of the amount of arsenic found.

There were two substances which he had mentioned in his paper—the sulphur and the nitrogen. Neither sulphur nor nitrogen were contained in the asphaltogen and asphaltene. He had especially stated in his paper that they pertained to volatile substances, and the nitrogenous bodies and sulphur compounds which had been obtained by himself and others were found with the petrolenes, and had not been investigated. He would investigate these substances hereafter.

As far as the names of petrolene and asphaltene were concerned, he thought that Boussingault, though long dead, had, so to say, a kind of a trade mark on them. Boussingault was the first to introduce them and describe them, and consequently, if anyone used the same names, he should use them as expressing the same things which the original inventor described; or, if one did not use them in the same sense as just explained, he should state why he did not use them in the original sense. In his (Dr. Endemann's) paper, he had stated that Boussingault had assumed the body asphaltic acid to be asphaltene, because he obtained it by heating the asphalt in air for a long period, but he really had not discovered asphaltene.

He adopted this name for real asphaltene, because he believed that Boussingault meant to call this body asphaltene. In the method of analysis which Mr. Richardson had introduced, there was absolutely no connection between the two substances.

In introducing this analysis by means of solvents, which he had proved in his paper to be absolutely incorrect and misleading, inasmuch as petroleum ether dissolved not only the petrolene, but likewise the asphaltene, Mr. Richardson's claim that this method, stated to be made up according to the plan by which petroleum oils were analysed, was absolutely incorrect and wrong, because using solvents was an entirely different thing from distillation. He (the author) had gone back to that original method of distillation, which was the one used in the examination of petroleum oils. Mr. Richardson had used an entirely new method, and now he likened his method to that which he (the author) had introduced.

In regard to the determination of the oxygen, the oxygen was determined by difference, after being assured that there was no sulphur present. Sulphur was in the distilled

portion. It was a fact that the sulphur compounds were among those substances which distilled, and, like the nitrogenous compounds, were found in the distillate. All the literature stated that these substances were first and always discovered in the distillate. He had recently found an article mentioning the presence of nitrogen in asphalt, where it was stated that with the petrolenes about 10 per cent. of pyridene compounds and substituted ammonias passed over. The author of this paper, whose name he did not remember, based upon this statement the useless hypothesis that this would show conclusively that the asphalt was the product of animal remains. From the fact that the asphaltene could be converted into asphaltulmic acid, he considered asphalt to be of vegetable origin. Ulmic acid, as obtained from sugar by the action of alkalis or acids, was of almost the same composition as the asphaltulmic acid. He had not been able to make the comparison as yet, but he might do it in the future. The substances in their composition were much the same. At any rate, they were very nearly related to each other.

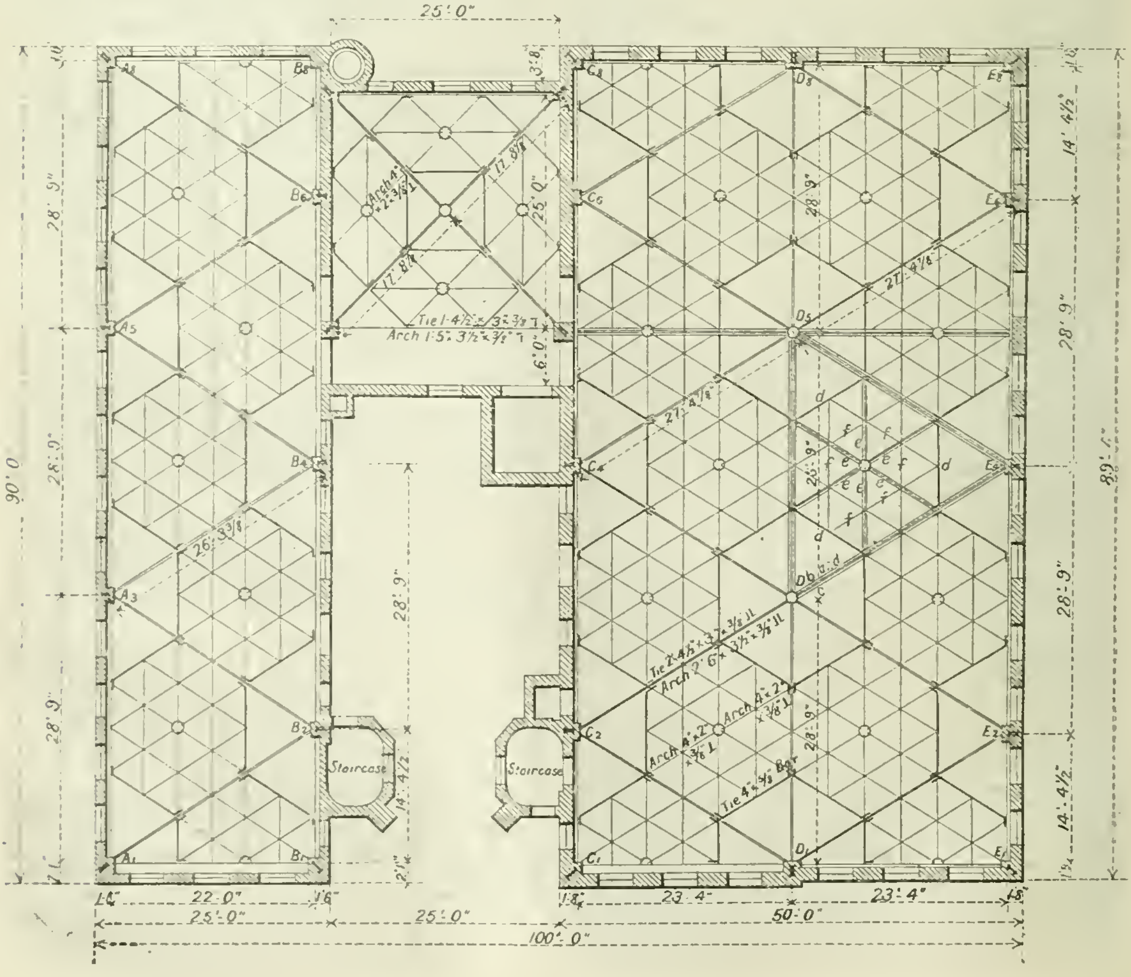
The presence of nitrogenous compounds, *i.e.*, pyridines or substituted ammonias in asphalt, did not indicate the derivation of the whole asphalt from animal origin. This was useless speculation. There was absolutely no necessity for such hypotheses, for the very reason that it was well known that nitrates were very easily decomposed into pyridene compounds and substituted ammonias. For instance, if water containing nitrates were distilled from an ordinary iron boiler, the water thus obtained contained a very much larger amount of ammonia than the water did originally in ammonia and albuminoid ammonia combination. This surplus was derived from the nitrates. It was notorious that the distilled water made in factories in New York always contained very much less ammonia than water made in Brooklyn. The reason was that the Ridgewood water in Brooklyn was richer in nitrates than the Croton water in New York; and in that way, by the infiltration of waters containing nitrates and sulphates, it was easy to account for the presence of nitrogen and sulphur in asphalt. A good many asphalts contained no nitrogen or sulphur, or only very small quantities. At any rate, he had determined that the bodies described in his paper did not contain any sulphur, and that therefore the substance given as oxygen was actually oxygen. It was not sulphur. Sulphur was among the volatile products. He intended to report hereafter upon the petrolenes, as well as the nitrogenous substances and sulphur compounds contained in the petrolenes or obtained with the petrolenes.

FIREPROOF LABORATORY CONSTRUCTION.

BY DR. WM. JAY SCHIEFFELIN.

To guard against fire, especially in a building intended to hold inflammable substances, it is not only necessary to have non-combustible materials of construction, but also to arrange the plan of the building so as to insure the complete isolation of its different parts, by avoiding large floor spaces (50 ft. × 50 ft. should not be greatly exceeded), and by having *no communication between the floors.*

It is better to have a low building over more ground than a high building on a small plot, as the latter is not so safe for the inmates and flames climb faster than they spread horizontally, but the rooms on each floor must be separated by fireproof partitions, and, if the building covers more than 50 ft. × 50 ft., enough continuous fire-walls must



be built (from cellar to roof) to divide it practically into separate buildings. Danger of damage by water is reduced to a minimum by having the floors watertight and graded to ample flush vents, and with the sills of the doors raised 3 ins.

A manufacturing laboratory requires large spaces for tanks and apparatus, with few columns to interfere with placing the same, and ample facilities for communication, such as elevators, dumb-waiters, schutes, speaking-tubes, and a variety of piping for conveying steam, compressed air, gases, and liquids. To comply with the fireproof requirements and yet not interfere with the essentials just mentioned, was the problem which we undertook to solve in 1894, when it became necessary for the firm of which I am a member to build a new laboratory, because the land on which our old laboratory stood had been taken by the City of New York to be made a public park. Thirty thousand square feet of room were required, and the system of construction selected is known as the Hecla fireproof construction. It consists of heavy steel columns supporting wide arches of concrete made from Portland cement. The concrete is cast in triangle forms between steel cantilevers and beams which extend between the columns, as per diagram, the beams are supported by curved brackets which, by virtue of the cantilever principle, allow a far wider span for a given weight of metal than does the ordinary rectangular fireproof construction; the spans in our laboratory are 30 ft., so that a room 50 ft. × 60 ft. has but one column. The floors have about 8 lb. of steel to the square foot, as compared to about 15 lb. for a like span of the usual rectangular construction.

The plan adopted is that of having three buildings around a courtyard, the largest building being divided by a continuous fireproof partition from the ground to the roof, thus substantially making the laboratory comprise four separate buildings under one roof, each building having three stories and a high cellar or basement with large windows. This arrangement of the plan admitted placing the stairways, elevator, dumb-waiter, and schutes outside the three manufacturing buildings, while they are all connected together, and direct communication with each other secured by means of the fourth building, which contains the boilers and drying rooms.

The building has a skeleton steel frame with skeleton cantilever steel floor construction filled in with concrete. The steel arches are grouped in triangles, the truss-beams transferring the loads immediately to the columns so that no intermediary system of girder and beam construction is required, thus economising steel and distributing the load evenly.

The work of putting in the floors took but a short time, and they were given six weeks time to set before heavy weights were put upon them. The fire partition from the ground to the roof in the large building is of rock plaster on expanded metal. It is 3 ins. thick and very strong. The other partitions are of rock plaster on jute bagging, they are 2 ins. thick and very rigid. The columns are protected from fire by being wrapped with $\frac{1}{4}$ in. thick asbestos paper and then with heavy wire laths, which in turn was covered with about $2\frac{1}{2}$ ins. of mortar composed of sand and lime, heavily gauged with plaster, and the floor beams being embedded in concrete and plaster are likewise protected. There are no hollow spaces between the floors and ceilings and the concrete becomes so firm that the floors are virtually on arches of artificial rock which would doubtless bear immense weight without the aid of the steel trusses. The specifications demanded that the floors support 250 lb. per square foot, but the actual strength is probably many times this amount. The floors are graded to flush outlets one to every 750 sq. ft., and the iron door sills are raised 3 ins. above the floor with inclined sides to facilitate the passing of trucks.

The doors are more fire resistant than those made of heavy iron, and are built according to specifications approved by the New York Board of Fire Underwriters, as follows:—Two thicknesses of 1-in. narrow-tongued and grooved, soft white pine boards, laid diagonally (both sides) and nailed with wrought nails driven through and clinched, then covered on both sides and edges with 10 × 14 sheets of "bright I.C." tin, joints flat-locked and securely nailed under laps (barbed wire nails used, $\frac{1}{2}$ in. in size, driven 2 ins. apart), but not soldered. All doors lap 3 ins. over openings, except where doors close in jambs or angle iron framework. The doors are arranged to close automatically in case of fire; they are sliding doors hung with heavy wrought-iron hangers and sheaves firmly bolted to and through the doors by carriage bolts. The runway or track is placed at an incline of $\frac{1}{2}$ in. to the foot and at such height that the doors when closed rest firmly on the raised sills. The doors are counterpoised by adjustible weights, hung on heavy sash cord in which is inserted over the centre of the door opening, a fusible link composed of two brass plates with rings for fastening, soldered together by the following alloy:—50 parts bismuth, 25 parts lead, 12 parts tin, and 13 cadmium. The melting point claimed is 68° C., but the one tested in the melting point apparatus of Roth melted at 78° C., which is probably low enough.

All the window frames are made of cast-iron. It is vital that windows opening on a courtyard should be fireproof, so that flames cannot be communicated from the outside; to attain this and avoid the trouble of fire shutters, the wire glass window panes were inserted; these are $\frac{1}{4}$ in. thick, and the wire mesh is in the centre of the glass, which is fused around it. The glass may be heated to a red heat on one side, and then cold water played upon the other without its breaking from the frame; it will crack in all directions, but none fall out, being held together by the wire; moreover, a brick can be thrown violently against it while hot or cold and fail to break through.

To avoid holes in the floors the following means were adopted:—The plumbing and gas pipes were put in place before the concrete, and collars extending 3 ins. above the floor were placed where steam pipes were to be fitted, then the concrete was filled in around them. Power for the machinery in the upper floors is transmitted by electricity from a dynamo in the basement, along a main wire hung in the elevator shaft, to motors located wherever needed, thus abolishing the dust, noise, reverberation, loss of power, and danger from belting or vertical shafts. Where it was necessary to run the return cables for the larger elevator through the building, they were entirely encased in an extra heavy galvanised iron box.

There are three fire escapes, one of iron at the back of the large building, and two of stone and brick in corresponding turrets at the entrance to the courtyard; these are used as stairways, and have fire-doors opening to the centre on each floor and windows to allow the escape of smoke. To subdue any interior fire in its incipiency, fire-hose, with high-water pressure, is placed at every stair landing outside the doors of the working rooms, and carbon dioxide extinguishers are at hand in every department.

In the smaller building, where the more inflammable materials are used in manufacture, the precaution has been taken to avoid any possible ignition from electric sparks by encasing the incandescent light sockets in rubber sleeves, and arranging them so that the current must be turned from a switch in the adjoining building, and motive power is supplied by steam engine instead of electric motor.

The composition of the surface of the floors varies with character of the materials used in the work: where they are dry, the floors are of concrete, sized with boiled linseed oil to prevent dust, and in the analytical department the floor is painted with two coats of oil-paint to make it absolutely dust proof. In a room where much petroleum is used as a solvent, the floor is covered with galvanised iron with soldered joints. Wherever water is apt to be spilt the floors are asphalt. The acid room has a floor of blue stone flag laid in tar and graded to an earthenware gutter and drain pipe.

The one disadvantage of the arched ceilings is the difficulty in hanging shafting, but by bolting wooden blocks to the cantilevers the hangers were adjusted.

The walls of the building are curtain walls, that is, enclosing walls sustaining only their own weight, and capable of being removed without disturbing the structure. There are flues built at intervals in the walls, and the main chimney from the boilers is outside the wall. The street walls are of washed brick trimmed with marble and buff-

brick and lined with hollow brick. The windows are on all sides of the building, so that the lighting and ventilation is excellent.

The cost of this system of fireproof construction depends on the price of steel and the skill and experience of the workmen who fill in the floor arches (much less time was required to construct the last of the floors put in than the first one). The cost is probably one-third more than ordinary wooden-mill construction, and one-fifth less than the rectangular beam and girder fireproof construction.

A marked advantage of this system is the stability of the structure, heavy machinery making but very slight vibration, and as the floors are braced in every direction and have windbraces in all the angles—like the knees of a ship—it would be useful in countries where earthquakes are prevalent. We have now occupied the building for 20 months, and it has proved satisfactory in every respect. The New York Board of Fire Underwriters has approved the building, and the Insurance Tariff Association has made a special rate upon it, which is one-third of what would be probable in ordinary construction, and is reported as the lowest rate on a laboratory in America.

Discussion.

Mr. Chas. M. Reubens asked of what material the tops of the tables of the laboratory were constructed.

Dr. W. J. Schieffelin replied that they had different tops for different purposes. The word laboratory would probably convey to most of their minds the place where analytical and experimental work was done. Only one department of the building was used for that, and the rest for manufacturing—and a druggist had to manufacture a hundred and one different things. In the room in the research department where the physical work was done, and where the different balances, the microscope, polariscope, and different instruments were placed, the table top for the dry work was made of plate glass, about 30 ft. or 40 ft. long, running around the different sides of the room. The plate glass was set on green baize, and it made a very clean surface to work upon. In the analytical room the table was covered with soapstone, albalene they called it, and it answered the purpose very well. Most soapstones, if butter or fat were dropped on them, would absorb them and stay dirty, but this did not, and one could scrape them off with a spatula. Soapstone was better than slate, for if one put a beaker down a little harder than one intended, it did not break as easily as upon slate.

The middle table of the analytical room was used solely for inflammable substances, when they were making experiments with naphtha, kerosene, ether, alcohol, or chloroform, and the rule was that these were to be made on that table. Generally they used the steam water-bath in the corner for heating. If that were not heat enough they could arrange to have an electric heater, so that these fluids were never where there was any fire. The top of that table was soaked in paraffin. It was a soft pine table, and he had a tailor's goose heated by electricity, and put shavings of paraffin upon the table, and, heating the goose, rubbed it around until the paraffin soaked in. But it was not very satisfactory. It collected the dust, and although it did not absorb water and turned most of the solvents that dropped on it, he would prefer the clean wood.

The one on the other side of the room was arranged in the same way. The sink was made as waterproof as possible by taking cherry wood and heating it in a kiln at the cabinet-makers, who put in all the woodwork in this department. All the cabinet woodwork was of oak. He had a drying kiln, and the specifications were that the sink should be made of cherry and the drip board also heated in the kiln with paraffin, so that the wood should absorb it. It was done twice and water did not affect it very much. The inside of the sink was caulked with white lead, and it made a rather nicer sink than the old lead one, because that was apt to get black and rub off.

Journal and Patent* Literature.

I.—PLANT, APPARATUS, AND MACHINERY.

Iron, Corrosion of, by Water containing Carbonic Acid. P. Petit. Comptes rend. 1896, **123**, 1278.

The waters of the Moselle, which are only slightly calcareous, act energetically on iron, sometimes piercing the iron pipes or reservoirs, to which they have access. A moderate addition of lime to the water causes deposition of a protecting crust of calcium carbonate on the iron surfaces. The iron is dissolved as ferrous carbonate, which decomposes in contact with air, forming ferric oxide and carbonic acid, which latter continues the attack. Thus a very small proportion of carbonic acid dissolved in water, is capable of great mischief.

The action on iron of very dilute solutions of calcium chloride, sodium chloride, potassium sulphate, and calcium nitrate, with and without carbonic acid, as compared with pure water, was also studied. In absence of CO_2, minute traces of iron were dissolved, though some was oxidised, sodium chloride and potassium sulphate acting most strongly. In presence of CO_2, iron was in each case dissolved—most in the potassium sulphate solution, and least in that of calcium nitrate. In closed vessels, the iron oxidised was about the same with or without carbonic acid; but with access of air, the oxidation proceeded very rapidly, and especially in a solution of calcium chloride.

—E. S.

Spectacles, New Protective. Chem. Ind. 1896, **19**, 554.

Dr. Thomalla, of Berlin, recommends the use of Schering's gelatoid in place of the wire-netting and glass at present used in spectacle construction. This gelatoid, specially hardened in amyl acetate, forms an elastic, solid, and perfectly transparent substance. The gelatoid pieces, serving as spectacle glasses, are perforated to allow passage of air to the eyes, whilst holes are also made in the side covers for the same purpose. The chief advantage in using gelatoid is that it is practically unbreakable, and, like horn, merely bends when struck by a foreign body, in cases where, under similar conditions, glass would splinter and perhaps cause injury.

* Any of these specifications may be obtained by post by remitting 8*d.*—the price now fixed for all specifications, postage included—to Sir Henry Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

The gelatoid is made in all colours; it is not combustible, merely carbonising when heated; and lenses made with it do not cloud over when the wearer passes from a cold to a warm atmosphere.—J. T. C.

PATENTS.

Evaporators, Impts. in and relating to the Discharging of Salts and the like from. J. Foster, Pollokshields, Glasgow. Eng. Pat. 928, Jan. 14, 1896.

The salts, &c., are withdrawn through a discharge valve in the lower end of the evaporator, into a pump barrel, and are delivered through a second valve in the barrel on the return stroke of the pump piston, the claim being for "the combination of a discharge valve at the lower end of an evaporator, working in time unison with an outlet valve in the pump barrel."—R. A.

Furnaces, Impts. in. [*Generator Furnace.*] W. P. Thompson, Manchester. From M. Herwig, Hesse-Nassau. Eng. Pat. 22,427, Oct. 9, 1896.

A generator-furnace having an "air superheater so constructed in the furnace that the outer wall of the latter forms also one outside wall of the air superheater, and the opposite wall of the latter encloses or limits the fire chamber, and is provided with discharge openings for branch flames derived from the generator furnace." The fourth claim is for "a form of construction of the generator furnace, in which the air superheater is extended round the entire wall of the furnace, and having, with the latter, its mouth at the top in the form of a tube-shaped extension. Thus, a most thorough utilisation of the radiated heat of the furnace," is said to be attained.—A. S.

II.—FUEL, GAS, AND LIGHT.

Peat, Production of Artificial Coal from. Glückauf, 1896, **32**, 923.

Owing to the low calorific value of peat in comparison with its bulk, and to the large percentage of ash, the cost of transporting it is prohibitive. Many attempts have therefore been made to convert it into coke. The largest bogs occur in Norway, and there Angel has carbonised peat by heating it to 250°—300° in open retorts for 10 hours, by which process gases, steam, and tarry constituents escaped and 50 per cent. of coke remained; but the method was too expensive. Rosendahl has now placed the matter on a practical footing. The peat is heated in closed iron retorts provided with a cock; the temperature is gradually raised to 250° C., and the cock, which is at first left open, is then closed, and the material kept at this temperature for seven hours. The result is that tar and gaseous products remain in the carbonised mass, and the yield of the artificial fuel thus produced, amounts to 80 per cent. of the peat employed. The analysis of the latter shows C = 65 per cent., O = 16 per cent., H = 6 per cent., H_2O = 3·7 per cent., and ash = 5 per cent. The artificial peat-coal has a calorific power of 6,500 cal., which, it is said, is nearly equal to that of ordinary coal. 1,000 kilos. can be sold at M. 7. Experiments at Krupp's works have shown that the material may be satisfactorily used in the iron foundry.—W. G. M.

Thorium, The Sources of. Rev. Chim. Ind. **7**, [84], 372—374.

The Norwegian minerals *thorite* and *orangite* contain a minimum of 20 per cent. of thoria, and their price is about 6*s.* to 8*s.* per kilo. per unit of thoria. Monazite is now the chief source of thoria, and, being found in large quantities, it has caused a reduction in price of pure ThO_2 from 240 dols. to about 36 dols. per lb. A few months ago the consuls of the United States were directed to report upon the trade in monazite, and the following is an abstract of their reports.

Monazite is a phosphate of cerium and lanthanum, containing small quantities of didymium and thorium; but the thorium is the valuable constituent, as it is used in the manufacture of incandescent mantles. The specific gravity of the mineral is 4·9 to 5·3, and it varies in colour from yellow to brownish-grey. It occurs in North America (in North and South Carolina, Quebec, and Connecticut), Brazil, and Russia. In North America the larger crystals of monazite are picked up by hand, but the sand is washed with water, as in the treatment of auriferous sands, yielding a product containing the monazite and particles of magnetite, ilmenite, garnets, and other impurities. After drying, the magnetite (but not the ilmenite) is removed with magnets. The sand then contains about 50 to 60 per cent. of monazite, and is sold at 25 to 30 cents per lb. The pure monazite crystals are worth 50 to 60 cents per lb. The Brazilian sand is found at Antigua, Bahia, Minas Geraes, Caravellas, and San Pedro, and is loaded directly into the ships, the cost amounting merely to that of the labour. In other districts—at the diamond mines of Rio Chico, Villa Bella, Cuyaba, and Goyaz—it is found as rounded nodules specked with orangite. The following table shows the amount of rare earths in average samples of the well-washed mineral:—

—	Oxide of Thorium.	Oxides of the Cerium Group.	Oxides of the Yttrium Group.
Quebec	1·10	50·2	4·5
Connecticut	1·40	61·0	..
North and South Carolina	0·23—0·80	39—63·3	0·1
Bahia	1·20	33·0	1·2
Minas Geraes	2·40	51·0	2·2
Rio Chico	4·80	53·0	3·2
Villa Bella	5·30	62·4	4·4
Goyaz	7·60	64·1	5·1

The price of monazite in the first shipments from Brazil, was quoted at 85*l.* per ton, but last winter it had fallen to 15*l.* per ton. Really the supply now exceeds the demand. Most of it is used at Vienna in manufacturing incandescent mantles.—H. B.

PATENTS.

Cyanogen Compounds, Impts. in Methods of Separating from Gas Liquor or other Solutions. H. Bower, Philadelphia, U.S.A. Eng. Pat. 361, Jan. 6, 1896.

Iron, or an iron salt, is added to ammoniacal liquor in sufficient proportion to form iron salts with the cyanogen compounds present. Lime is then added, and the ammonia is distilled off. The liquor, now containing calcium ferrocyanide and sulphocyanide, is treated with an acid solution of a copper salt, preferably cuprous chloride. The precipitate, containing copper ferrocyanide and sulphocyanide, is separated, and heated in an autoclave under pressure with iron filings or reduced iron, when iron salts are formed with separation of copper, which decomposes the sulphocyanide with production of copper sulphide and iron ferrocyanide, the latter being now the only cyanogen compound present. From the iron ferrocyanide, alkali ferrocyanides are readily obtained. Reference is made to the inventor's previous Eng. Pat. 8330, 1895 (this Journal, 1896, 274).—E. S.

Incandescent Gas Lighting, Impts. in the Manufacture of Mantles for. W. Mackean and The Incandescent Gas Light Co., Ltd., Westminster. Eng. Pat. 3681, Feb. 18, 1896.

Mantles are to be made by mixing oxides (either hydrated or anhydrous) or carbonates of suitable metals (*e.g.*, the rare earths) with a solution of an ammoniacal salt, such as the acetate, nitrate, or sulphate, impregnating a fabric with this mixture, drying the impregnated fabric, and burning off as usual.—H. B.

Incandescence Bodies for Illuminating Purposes, Impts. in the Manufacture of. W. Nicholls, London. Eng. Pat. 4000, Feb. 21, 1896.

Mantles are to be made by impregnating suitable fabric in the usual manner with a solution of the following mixture of salts:—Zirconium nitrate, 55 parts; calcium nitrate, 45 parts; yttrium nitrate, 3 parts; uranium nitrate, 0·5 part.—H. B.

Incandescent Lighting with an Intimate Mixture of Gas and Air, Impts. in. L. Denayrouze, Neuilly, France. Eng. Pat. 1576, Jan. 22, 1896.

Reference is made to Eng. Pats. 22,615 of 1894, 4491 of 1895, 7722 of 1895, and 14,053 of 1895, for methods of effectively mixing the gas and air before combustion in incandescent gas lighting. (1) When the coal-gas is taken at the service pressure and the air at atmospheric pressure, the mantle having the usual weight and volume, the combustible mixture should consist of 1 volume of gas and 3 to 5 volumes of air; the gases should be thoroughly mixed, and should have a velocity of 1·5 to 2·5 metres per second. (2) "Good mixture can be obtained by giving the gas and air an initial pressure higher than that in the service pipes and the atmosphere." Under these circumstances the aim should be to have a mixture containing 1 volume of gas with 3 or 4 of air, an intimate mixture, and a velocity of 6 to 12 metres per second. (3) When the incandescent bodies are not mantles of woven filaments, but are porous masses, relatively thick, such as may be obtained by using sponges, pumice stone, or retort carbon, impregnated with the usual solutions employed in making mantles from fabric, the quantity of combustible gas and the velocity of the mixture have both to be greater, the greater the mass of the body to be raised to incandescence, is, relatively to its volume.

The patentee claims "For incandescent gas lighting, the method of effecting an intimate mixture of air and gas under the combined conditions as to the composition, the pressure, and the velocity of the mixture herein-before stated."
—H. B.

Incandescence Bodies for Illuminating Purposes, Impts. in the Manufacture of. W. H. Wheatley, London. From F. Woernle, Stuttgart. Eng. Pat. 1681, Jan. 23, 1896.

Mantles are to be made in the usual manner, using as impregnating fluid a solution of one of the following mixtures of salts:—(1) Thorium nitrate, 12 parts; erbium nitrate, 2·5 parts; zirconium nitrate, 9·0 parts; magnesium nitrate, 1·25 parts. (2) Zirconium nitrate, 12·25 parts; erbium nitrate, 9·0 parts; thorium nitrate, 2·5 parts; strontium nitrate, 1·25 parts.—H. B.

Incandescence Bodies for Illuminating Purposes, Impts. in the Manufacture of. W. H. Wheatley, London. From C. Schmid, Brussels. Eng. Pat. 1682, Jan. 23, 1896.

Mantles are to be made in the usual manner, using as impregnating fluid a solution of one of the following mixtures of salts:—(1) Magnesia, 1 parts; zirconium nitrate, 85 parts; aluminium nitrate, 4 parts; erbium nitrate, 4 parts; strontium nitrate, 2 parts; didymium nitrate, 1 part. (2) Magnesia, 1·25 parts; zirconium nitrate, 12·5 parts; strontium nitrate, 1·25 parts; erbium nitrate, 10 parts; didymium nitrate, 0·25 part.—H. B.

Incandescent Mantles, Impts. in. P. Stiens, Clapham. Eng. Pat. 1841, Jan. 25, 1896.

Magnesium chloride which has been heated to a high temperature in an open vessel and then cooled, is ground and mixed with 2 to 3 per cent. of powdered burnt chalk and sufficient gum tragacanth or gelatin to form a paste with water, and is then "moulded to the desired shape." Or, preferably, the mantle is made in the usual way by impregnating a fabric with a solution containing one part of the burnt magnesium chloride in two parts of nitric acid. In either case the "mantle" is dried, burnt, and then sprayed with a solution of burnt or unburnt magnesium chloride, and finally exposed to a high temperature. Thoria and other mantles are given "greater luminosity and strength" by the addition of burnt magnesium chloride. The patentee disclaims the use of magnesium chloride, but makes the one claim: "the employment, in the manufacture of incandescent mantles, of magnesium chloride which has first been exposed to a high temperature, substantially as described."—H. B.

Liquid Fuel, Impts. in Apparatus for Burning. A. Quentin, Brussels. Eng. Pat. 3560, Feb. 17, 1896.

This consists of a burner in which is comprised a chamber containing a suitable finely divided material (metallic chips or turnings, &c.), through which the hydrocarbon passes, the said chamber being heated through its length by the flame from a nozzle, at which is burnt the vapour so produced.

The arrangement and construction of a battery of these burners is also claimed.—A. S.

Combustible Blocks [Briquettes] or Cakes from Particles or Dust of Waste Coal, Anthracite, and Lignite; A New Process for making. E. Jumeau, Pondicherry. Eng. Pat. 23,076, Dec. 2, 1895.

An agglutinative paste is made from pulverised tamarind seed by allowing it to slightly ferment, and then boiling. This paste is then mixed with particles of coal, anthracite, or lignite, in the proportion of about 10 per cent., and finally formed into blocks.—R. B. P.

Treatment of Coke so as to render it suitable for Transport; A New or Improved Method or Process for the. F. A. MacKinnon, South Kensington. Eng. Pat. 5916, March 17, 1896.

Coke, as it comes from the retorts, is treated with a spray of hot water or steam, holding in solution a binding cementitious material, preferably some cheap or waste matter containing glue or gelatin. The treated coke is next placed in moulds and subjected to pressure to form blocks of a convenient size for transport.—R. S.

Gas Producers; Improved Process of and Apparatus for Facilitating the Burning of poor Coal and the like, chiefly applicable for Use in. L. Bemelmans. Eng. Pat. 17,130, Aug. 1, 1896.

The claim is for a process for burning fuel which settles, and consists in withdrawing, at very short intervals or continually, small quantities of ashes, and introducing air from the furnace above the settled ashes. There is also claimed the arrangements for carrying into practice the process in a rotary gas producer, a rotary annular gas producer, and in a gas producer having a cylindrical furnace and a fixed casing.—A. S.

Gas Producers, Impts. in Safety Devices for. A. C. Humphreys and A. G. Glasgow, Victoria Street, London; and New York. Eng. Pat. 23,904, Oct. 27, 1896.

Consists in providing a water-gas apparatus (having air-blast pipes with valves, separate take-off pipes for products of combustion and water-gas, and a valve for opening and closing the take-off pipe for products of combustion), with an arrangement for absolutely locking one or more valves against movement when another valve or valves is in a determined position.—R. S.

Producing Gas or Vapour from Air and Liquid Hydrocarbons, Improved Process and Apparatus for. A. I. van Vriesland, Amsterdam. Eng. Pat. 25,498, Nov. 13, 1896.

The process consists in forcing air, at a suitable pressure and in a finely divided condition, through the liquid hydrocarbons or through fabrics saturated therewith, and leading off the mixture. Two forms of apparatus are shown, each consisting of a vessel having an internal chamber, into which the air is forced through a central tube ascending into it, and from which the air escapes through perforations, or through a fabric partition into the outer chamber, which contains the liquid hydrocarbon. The combustible gas escapes through an outlet at the top of the apparatus.
—R. S.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Uranium Carbide. H. Moissan. Bull. Soc. Chim. 1897, 12—18.

When uranium is heated in the electric furnace with excess of carbon, a definite crystalline carbide is formed

possessing the formula C_3U_2. When this substance is treated with cold water, it evolves about a third of its carbon in the form of a mixture of gaseous hydrocarbons rich in methane. The remainder is found in the form of a mixture of liquid and solid hydrocarbons and bituminous matters. Probably this decomposition is the result of polymerisations similar to those observed by Berthelot in his researches on the action of heat on hydrocarbons. The presence of the hydrogen in the gaseous mixture may, on the other hand, be due to a secondary action of a hydrated uranium oxide, which must be a powerful reducing agent. Péligot has shown that anhydrous uranium protoxide has a great avidity for oxygen, and that a suboxide exists which decomposes water.

The reaction of uranium carbide and water is interesting as affording a means of preparing a whole series of gaseous, liquid, and solid organic substances by the action of cold water on a metallic carbide.—T. E.

Mineral Oils, Desulphurising. W. Heinrici. Zeits. f. angew. Chem. 1897, 8.

Heusler (this Journal, 1896, 584) has stated that treatment with aluminium chloride removes all unsaturated hydrocarbons and sulphur compounds from mineral oils. The author investigates the applicability of this process to the lignite tar oils of Saxon Thüringia. A well-dried oil containing 1·2 per cent. of sulphur was treated with 5 per cent. of $AlCl_3$ for 24 hours: after separating the resinous matter formed, and washing free from acid and distilling, an illuminating oil was obtained containing 0·27 per cent. of sulphur. By employing 10 per cent. of $AlCl_3$ and continuing the treatment for 120 hours, the percentage of sulphur was reduced to 0·058, and by repeating the treatment it was possible to bring this down to 0·0085. According to Heusler, lubricating oils are obtained as a by-product; the author finds, however, that these heavy oils contain about 1·3 per cent. of sulphur and have a viscosity of only 4·5 at 20°, measured by Engler's viscosimeter. By distilling the resins which are formed, with steam, small quantities of an oil of disagreeable odour, and containing 4·4 per cent. of sulphur, were obtained: this oil was free from thiophene. It is to be noticed further that the oils may be desulphurised quite as efficiently and as cheaply by treating them with large quantities of strong sulphuric acid (66° B.). Thus, "Solar" oil treated with—

5 per cent. of Acid	gave	an oil	containing	0·796	per cent. S.
10	"	"	"	0·629	"
15	"	"	"	0·241	"
25	"	"	"	0·135	"
50	"	"	"	0·100	"
100	"	"	"	0·063	"
200	"	"	"	0·046	"
400	"	"	"	0·0074	"

Acid of 40° or 50° B. had little desulphurising action, while fuming acid (10 per cent. SO_3) gave no better results than acid of 66° B. The temperature at which the treatment took place was also found to be of little moment.

—T. E.

Thiophene: Removal from Benzol by Means of Aluminium Chloride. F. Heusler. Zeits. f. angew. Chem. 1896, **11**, 750.

Benzol (120 c.c.) which had been purified commercially by means of sulphuric acid and contained much thiophene, was heated to boiling in a reflux apparatus with aluminium chloride (5·8 grms.) and distilled, moisture being excluded. The distillate, after being washed with caustic soda and dried with calcium chloride, was found to be free from thiophene. This is in accordance with the results of Haller and Michel (this Journal, 1896, 706).

In his earlier experiments (*ibid.* 584), in which the benzol, after treatment with aluminium chloride, was steam-distilled, or the decanted product distilled, the author failed to remove all the thiophene. It seems probable that at the low temperature of the reaction, only a portion of the thiophene is converted into an insoluble resin, the rest forming an intermediate product which is soluble in benzol, and which regenerates thiophene in presence of water. In the case of xylol, the intermediate product would be decomposed at the high temperature of the reaction, so that the decanted product may be treated with water without regeneration of thioxene.—N. H. J. M.

Coal-Tar Bases. F. B. Ahrens. Ber. 1896, **29**, 2995—2999.

The author has isolated a new lutidine and collidine from coal tar, which he found to boil at 163·5°—164·5° and 165°—168° respectively.—D. B.

Iron Carbide, A Pure. E. D. Campbell. Amer. Chem. J. 1896, **18**, 836.

See under X., page 145.

IV.—COLOURING MATTERS AND DYES.

Dyestuffs, An Attempt to classify Organic. S. de Kostanecki. J. Soc. Dyers and Colourists, 1897, 27.

The organic colouring matters are characterised according to their formulæ by the presence of chromophors, which all contain double bonds. By arranging them according to the nature and the number of these chromophors, the author arrives at the following classification:—

A. Dyestuffs containing a Single Chromophor.

C : C. Dibiphenylene-ethene.
C : O. Oxyketones, oxycoumarins, oxyflavones, oxyxanthones.
C : N. Auramine, thioflavine, quinoline yellow.
$N\langle{}^{O}_{O}$ Nitro-colouring matters.
N : N. Azo colours.
N : N : O. Azoxy colours.

B. Dyestuffs containing Several Chromophors.

(a.) Streptostatic Chromophors (Ketone Type).

$\left.\begin{matrix}C:C\\C:C\end{matrix}\right\}$ Unsaturated oxyketones, indogenides, oxyindogenides, indigo.
$\left.\begin{matrix}C:O\\C:O\end{matrix}\right\}$ Oxydiketones, oxydixanthones.
$\left.\begin{matrix}C:N\\C:N\end{matrix}\right\}$ Hydrazone dyestuffs.
$\left.\begin{matrix}N:N\\N:N\end{matrix}\right\}$ Disazo dyestuffs.

(b.) Cyclostatic Chromophors (Quinone Type).

	C : C	C : O	C : N
C : O...	Aurins ...., Benzeins ...., Phthaleins	Oxyquinones.	.. ..
C : N...	Basic colouring matters of the triphenylmethane group ....., Pyronines	Indophenols. Nitrosophenols.	Indamines. Azines. Safranines. Indulines.
C : N : O	....	Resazurine.	.. ..

(c.) Streptostatic and Cyclostatic Chromophors.

This group comprises several complicated colouring matters, such as Alizarin Blue, Styrogallol, &c.—I. S.

Colour Bases of the Triphenylmethane Series, Constitution of. H. Weil. Ber. **29**, 2677—2678.

In contradiction of the statement of v. Georgievics (Monatsh. Chem. **17**, 10), the author observes that the red precipitate obtained by the addition of the calculated amount of soda-lye to a concentrated magenta solution is not a coloured base, but under all circumstances a mixture of rosaniline with its hydrochloride, the latter preponderating, the more concentrated the cold magenta solution employed, and the smaller the excess of soda lye. The presence of magenta is easily proved by the fact that the precipitate, washed free from inorganic salts, begins to dissolve in water with the

characteristic colour, and contains chlorine (see this Journal 1896, 535). The author, moreover, points out that no conclusion can legitimately be drawn as to the constitution of the "red rosaniline base" from that of the "imidoxide base," which is certainly different, and can be obtained from it by washing with hot water. As already stated (*loc. cit.*), owing to the little solubility of magenta in salt solutions, the alkali partly salts out some of the magenta, together with the rosaniline base. The following experiment supports the statement as regards the stability of magenta towards alkalis. If carbonic anhydride be passed into water warmed to 50°—60° C., in which rosaniline base is suspended, a solution is obtained containing rosaniline carbonate, which is neutral to litmus. On the addition of salt, rosaniline hydrochloride is precipitated and the colourless solution then contains sodium carbonate, and reacts strongly alkaline.—T. A. L.

Parabenzoyltriphenylmethane and its Derivatives. P. Bourcet. Bull. Soc. Chim. 1897, 81—85.

Parabenzoyltrinitrotriphenylmethane—

$$C_6H_5CO.C_6H_3NO_2.CH(C_6H_4.NO_2)_2$$

is prepared by adding dry finely powdered parabenzoyltriphenylmethane to a large excess of fuming nitric acid at 0°. After three hours the liquid is warmed gently, and finally poured into excess of cold water. The nitro derivative thus obtained is purified by precipitating its solution in acetic acid with water, or may be crystallised by spontaneous evaporation of its solution in nitric acid. Fused with caustic potash, it yields benzoic acid, confirming the above formula. It softens at 70° and melts at 74°—75°.

The corresponding *carbinol* is obtained by oxidation with chromic acid in acetic acid solution. The carbinol is colourless, but becomes bright red when exposed to light; it softens at 85° and melts at 88°.

By reducing the acetic acid solution of the carbinol with the theoretical quantity of zinc dust and precipitating with water, a green powder is obtained, the greater part of which, is the *acetate of parabenzoylpararosaniline*—

$$C_6H_5CO.C_6H_3NH_2.C\begin{cases}O.COCH_3\\(C_6H_4NH_2)_2\end{cases}$$

This is not pure, because the hydroxyl group of the carbinol is reduced to some extent. The acetate of parabenzoyl pararosaniline dyes silk, wool, and cotton without a mordant; the colour produced is a pale green, which is not very fresh, but is fast to water, soap, and light.

When excess of zinc dust is employed in reducing the nitrated carbinol, the solution loses its red colour after a time, becoming pale yellow; it then contains the *leuco-base* $C_6H_5CO.C_6H_3NH_2CH(C_6H_4NH_2)_2$, which may be crystallised from alcohol in greyish crystals. The last two compounds decompose without melting.—T. E.

Methyl Iodide on Aqueous Solutions of Crystal Violet, Malachite Green, and Methylene Blue, Action of. Hydrolysis of these Colouring Matters. A. Rosenstiehl. Bull. Soc. Chim. (3), **15**, 1299—1313.

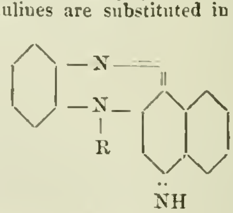
The alcoholised di- and triamine derivatives of triphenylcarbinol combine directly with methyl iodide in the cold, even in presence of much water, forming iodammonium derivatives, which can be titrated alkalimetrically after hydroxylation. The presence of acids retards the change, but does not prevent it, so that aqueous solutions of the magentas corresponding to the carbinols behave like the latter. The colouring matters above named, on solution in water, become partially hydrolysed into free acid and free base, which latter combines with the methyl iodide. This partial hydrolysis causes an increase in the electric conductivity of the solution.—G. H. B.

Safranines, Constitution of. R. Nietzki. Ber. **29**, 2771—2773.

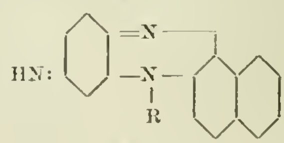
Kehrmann's discovery of phenylphenazonium (this Journal, 1896, 802) having at length definitely settled the question as to the safranine formula, the author again refers to his position in the matter, pointing out that a condensation (*e.g.* in safranone) between the ammonium group and a hydroxyl group is in reality only an inner salt formation which frequently occurs between acid and basic groups, even between meta substituents, as, for instance, in *m*-diazobenzene sulphonic acid and in *m*-diazobenzoic acid. That aposafranine can be diazotised has been shown meanwhile by Kehrmann. The strong basicity of the safranines, which the author has always considered a strong argument in favour of the azonium formula, is also borne out by Fischer's discovery that the hydrochloride of acetyl-aposafranine gives a carbonate with dilute sodium carbonate solution, for only relatively strong bases form stable carbonates. In the light of our present knowledge, only two views are tenable as regards the anhydrous safranine bases: either a condensation takes place (Nietzki), or there is a molecular change into the tautomeric *p*-quinone formula (Kehrmann). The latter appears improbable, since the shade of the safranine colouring matters is but little altered by alkalis, although in the less basic members of the series—for instance, acetylsafranine, which changes from yellow to violet—such a transformation may take place. In conclusion the author points out that Jaubert's analyses (usually nitrogen determinations) are not sufficiently in accord with theory to be accepted in support of this investigator's views and nomenclature proposals.—T. A. L.

The Isorosindulines. O. Fischer and E. Hepp. Ber. **29**, 2752—2760.

The name isorosinduline has been given (Annalen, **272**, 306; this Journal, 1893, 669) to substances isomeric with the rosindulines, differing from these latter in containing the substituents (:NH, :O, :NC_6H_5) in the benzene nucleus, whilst the rosindulines are substituted in the naphthalene ring—

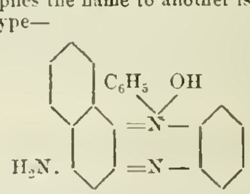


Rosinduline.



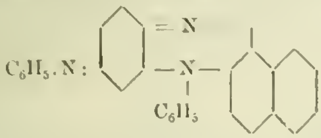
Isorosinduline.

Kehrmann, however (Annalen, **290**, 247; this Journal, 1896, 536), applies the name to another isomeride of rosinduline of the type—

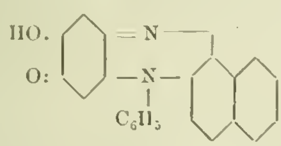


for which the authors propose the name pseudorosinduline. In view of the relation between aposafranine and the simplest induline, it follows that the compound obtained by Nietzki and Otto (Ber. **21**, 1600) from quinone dichlorodiimide and phenyl-β-naphthylamine must be an isorosinduline. The present investigation was undertaken in order to determine in what respects this dyestuff is similar to aposafranine, and more especially as regards the indone, and also to study the action of bases such as aniline, *p*- and *o*-phenylene diamine, the action of alcoholic potash and of nitrous acid. When isorosinduline is treated with nitrous acid in presence of strong sulphuric acid, it is converted into a diazo compound, which, on decomposition with alcohol, yields, to all appearances, the same azonium compound as rosinduline when treated in a similar manner (Kehrmann, Ber. **29**, 1316). The azonium compound from isorosinduline is

converted by the action of ammonia into rosinduline, so that the conversion of the one isomeride into the other can in this way be effected. *Isorosinduline*, prepared according to Nietzki and Otto (*loc. cit.*), was purified by direct conversion into the well-crystallised nitrate by adding potassium nitrate to the alcoholic solution of the melt, diluted with water. Alcoholic solutions of the salts show a faint brownish fluorescence, and they are decomposed by alkaline carbonates. The isorosinduline base is very unstable to caustic alkalis and turns dirty bluish-violet. The dark bluish-violet sparingly soluble product gives blue salts with acids, and cannot be reconverted into isorosinduline. Much more stable to alkalis is *phenylisorosinduline*—



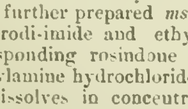
obtained by condensing, in presence of hydrochloric acid, nitrosodiphenylamine (3 mol.) with phenyl-β-naphthylamine (2 mol.) in an alcoholic solution. The nitrate crystallises well from 50 per cent. alcohol; the base dissolves with a blue colour in alcohol, benzene, or ether, and crystallises from benzene-petroleum ether. On heating with a mixture of acetic and hydrochloric acids to about 235° C. for 10 hours, *isorosindone* is obtained, but it can be produced much more easily by condensing nitrosophenol with phenyl-β-naphthylamine. The substance is a very weak base, and after crystallisation from alcohol, melts at 224° C. The solutions of the salts are yellow. When heated with alcoholic potash, isorosindone is converted into *oxyisorosindone*—



as safranone is into oxyaposafranone (Ber. 28, 2287). On heating with caustic potash and methyl iodide in methyl alcoholic solution, it is converted into methoxyisorosindone, which crystallises from alcohol in greenish needles, melting at 274° C. This substance dissolves in concentrated sulphuric acid with a bluish-violet colour, which turns brownish-red on dilution. By the action of aniline and aniline hydrochloride on isorosinduline in a concentrated alcoholic solution, *anilido-isorosinduline* (melting point 152° C.) is obtained, but the mode of conversion is not yet fully investigated.

The authors have further prepared *ms*-ethylisorosinduline from quinonedichlorodi-imide and ethyl-β-naphthylamine, and also the corresponding rosindone from nitrosophenol and ethyl β-naphthylamine hydrochloride. The base, which melts at 178° C., dissolves in concentrated sulphuric acid with a reddish-violet colour, which, on dilution, passes through brownish-yellow to light yellow.

It would appear that the same *phenylnaphthophenazonium chloride*—



is obtained both from rosinduline and isorosinduline by means of the diazo reaction, and further investigation will show whether the isorosindulines alkylated in the azine ring, behave similarly.—T. A. L.

Quinone-imide Dyestuffs, Configuration of. W. Vaubel. J. für prakt. Chem. 1896, 292—304.

The quinone-imide colouring matters may be arranged in two groups, one of which comprises such of them as do not contain azine rings—for example, the indamines and indophenols—and the other, the azines, that is, the eurhodines, oxazimes, thiazimes, safranines, and indulines.

The second group may be subdivided into the following classes:—

1. Compounds of which one form only is possible; for example, phenazine.

2. Colouring matters which are capable of existing in two forms, namely, with a central ring like that of phenazine, and in a quinonoïd form. The eurhodines and eurhodols belong to this class. Although the former of these have not yet been shown to exist in the quinonoid form, still it is probable that they do so, since the analogously constituted eurhodols so exist, yielding, as they do, with methyl iodide two monomethyl derivatives.

3. Colouring matters which exist in two quinonoïd forms. This group comprises the Safranines, Magdala Red, Mauveïne, and the Rosindulines.

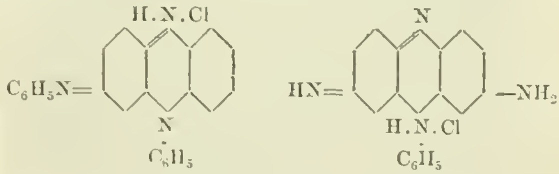
4. Quinonoïd colouring matters which have one form alone—the indulines.

Safranines and indulines differ in the colours of their bases and salts, thus:—

	Safranines.
Colour of base and salt	Red.
" concentrated HCl solution	Blue.
" " H_2SO_4 "	Green.

	Indulines.	
	Phenylindulines.	Indazines.
Colour of base	Brown-yellow.	Red.
" salt	Violet.	Blue.
" concentrated HCl solution.	Blue.	Green.

The fundamental difference in constitution between the two classes of colouring matters lies in the different valencies of the nitrogen atoms in the azine rings, as is seen in the formulæ—



Indulines. Safranines.

5. Oxazime, oxazone, thiazime, and thiazone colouring matters, which may be regarded as derived from indulines and indones by the substitution of oxygen and sulphur for the inactive meso-phenylimido groups.—E. B.

Quinone-imide Dyestuffs, Bromination of. W. Vaubel. J. für prakt. Chem. 1896, 289—291.

Phenosafranine is powerfully acted upon by bromine, apparently with the production of a tetrabromo derivative, although the end of the reaction is difficult to perceive, owing to the intense red colour of the solution. It is probable that the bromine atoms take up the free positions ortho to the amido- and imido groups.

Indazine and bromine react slowly, the monobromo compound separating out from the solution.

Rosinduline also absorbs one atom of bromine, the completion of the reaction being in this case easily observed.

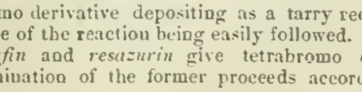
Rosinduline disulphonic acid reacts energetically with bromine. It was not determined how much of the latter was absorbed, or whether sulphuric acid was liberated.

Rosindone behaves similarly to the last compound. The product is precipitated in red flocks.

Meldola's Blue is likewise precipitated, but the termination of the reaction is seen with difficulty.

Methylene Blue reacts with two atoms of bromine, the monobromo derivative depositing as a tarry red mass and the course of the reaction being easily followed.

Resorufin and *resazurin* give tetrabromo derivatives. The bromination of the former proceeds according to the equations—



$$BrO.C_6H_3 \left\langle \begin{matrix} N(Br) \\ O \end{matrix} \right\rangle C_6HBr_2OH + (2HBr + 2Br) =$$

$$HO.C_6H_2Br \left\langle \begin{matrix} N(Br) \\ O \end{matrix} \right\rangle C_6HBr_2.OH =$$

$$O = C_6H_2Br \left\langle \begin{matrix} N \\ O \end{matrix} \right\rangle C_6HBr_2.OH (+ HBr),$$

further action in the same way giving finally—

$$O = C_6HBr_2 \left\langle \begin{matrix} N \\ O \end{matrix} \right\rangle C_6HBr_2.OH.$$

—E. B.

Pyronine Dyestuffs, Constitution of. J. Biehringer. J. für prakt. Chem. 1896, 217—258.

The pyronines have the general formula—

$$RC \left\langle \begin{matrix} C_6H_3—NR'_2 \\ >O \\ C_6H_3{=}NR'_2Cl, \end{matrix} \right.$$

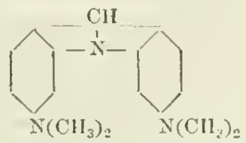
in which R′ represents an alkyl group, and R an alkyl, phenyl, or other group, or hydrogen.

They are obtained by condensing together aldehydes and *m*-hydroxydialkylanilines and oxidising the resulting compounds (Eng. Pats. 13,217 of 1889 and 18,606 of 1891; this Journal, 1890, 934, and 1892, 808). Only the compounds obtained from formaldehyde are at present manufactured. These appear in commerce under the names and shade marks Pyronine G (prepared from dimethyl-*m*-amidophenol), Pyronine B (from diethyl-*m*-amidophenol), and Pyronines O and GO, and are powders of a dark red colour with a green surface-lustre. Their aqueous solutions have a red colour and yellow fluorescence.

The constitution of the class is established by the formation of Pyronine G in the following way:—Dimethyl-*m*-phenylenediamine (2 mols.) when treated in alcoholic solution with hydrochloric acid ($\frac{1}{8}$ mol.) and formaldehyde (1 mol.), gives tetramethyltetramidodiphenylmethane (m.p. 139°—141°), identical with the compound formed by nitrating tetramethyl-*p*-diamidodiphenylmethane, according to the method patented by Gerber and Co. (in sulphuric acid solution at 0°; Ger. Pat. 60,505), and reducing the dinitro compound. It has, therefore, the constitution—

$$N(CH_3)_2 : NH_2 : CH_2 = 4:2:1\ ;\ 4:6:1,$$

which is confirmed by its conversion, when heated with fuming hydrochloric acid, into an acridine derivative—



a compound (m.p. 181°—182°) dissolving in water with an orange-yellow colour, in aqueous acids with a red colour, and in concentrated sulphuric acid with a yellow colour and the blue-green fluorescence characteristic of acridine compounds. This is presumably the colouring matter known as Acridine orange (Ger. Pat. 59,179; this Journal, 1891, 537), the tetramethyltetramidodiphenylmethane requisite for the production of which is obtained by the condensation of *m*-amidodimethylaniline and formaldehyde.

The tetramethyltetramidodiphenylmethane obtained by either of the foregoing methods, gives by the diazo reaction the dihydroxy leuco base of Pyronine G.

Benzaldehyde condenses with dimethyl-*m*-amidophenol, yielding tetramethyldiamidodihydroxytriphenylmethane (Ger. Pat. 62,574), which, when oxidised, loses the elements of water and forms an Oxy-malachite-green Pyronine, a pink colouring matter formerly prepared from dimethyl-*m*-amidophenol and benzotrichloride (Ber. 22, 3001), and named Rosamine. The dihydroxy leuco compound of this, after being benzoylated, gives on oxidation, a green dyestuff, which is easily saponified and condensed into the Pyronine.

When oxidised in alkaline solution, the Pyronines yield xanthone derivatives; thus, Pyronine G gives tetramethyldiamidoxanthone—

$$CO \left\langle \begin{matrix} C_6H_3—N(CH_3)_2 \\ >O \\ C_6H_3—N(CH_3)_2 \end{matrix} \right.$$

a compound crystallising from alcohol in yellow prisms, which lose alcohol of crystallisation at 130° and melt at 240°—242°. It dissolves readily in alcohol, acetone, chloroform, carbon bisulphide, and xylene, less readily in benzene, and with difficulty in ether and petroleum spirit. Its acetone, chloroform, alcohol, and concentrated sulphuric acid solutions display a blue fluorescence, its aqueous acid solutions a fine green fluorescence and yellow colour; the concentrated acid solutions stain the skin yellow. On reduction and subsequent oxidation in acid solution, the xanthones are reconverted into the corresponding Pyronines.

—E. B.

Thiobenzophenone. L. Gattermann and H. Schulze. Ber. 29, 2944—2945.

In a previous paper (Ber. 28, 2869), the authors have described the preparation of thiobenzophenone by heating benzophenone with phosphorus pentasulphide in benzene. The product obtained, however, always contained more or less benzophenone, and purification, owing to the instability of the thio-compound, was not possible. According to the present paper, the pure product can be obtained by the action of alcoholic potassium sulphide on benzophenone chloride. This latter is prepared by heating 12 grms. of benzophenone and 20 grms. of phosphorus pentachloride under an inverted condenser in an oil-bath to about 230° C., and subsequently fractionating in a vacuum, when the ketone chloride distils at 193° C. under a pressure of 30 mm. For the preparation of the thiobenzophenone, 1 grm. is mixed with 4·5 c.c. of alcoholic potassium sulphide solution (1 c.c. contains 0·1124 potassium sulphide). The mixture heats spontaneously, turns blue, and potassium chloride is precipitated. After cooling, water is added, the thioketone is extracted with ether and distilled under a pressure of 14 mm., when it passes over at 174° C. as a dark blue oil, solidifying to blue needles. It melts at a lower temperature than benzophenone, and can only be preserved in an atmosphere of carbonic acid in the dark. On warming with alcoholic potash it is quickly converted into benzophenone. The same change takes place gradually when exposed to the air. With hydroxylamine it yields benzophenone oxime, and on heating with finely divided copper, it gives quantitatively tetraphenylethylene.

—T. A. L.

Naphthacetol (p-Acetamido-α-Naphthol). O. N. Witt and J. Dedichen. Ber. 29, 2945—2954.

The azo dyestuffs from α- and β-naphthol possess very different properties, for whilst β-naphthol yields compounds in which the azo group enters ortho to the hydroxyl, α-naphthol can give two series of azo dyestuffs, according to whether the azo group enters para or ortho to the hydroxyl. The former most frequently takes place, and the resulting dyestuffs usually possess such strongly marked phenolic and basic properties, giving salts not only with metallic oxides but also with acids, that, technically, they are of little value, owing to the fact that the formation and decomposition of these salts is accompanied by a change in shade. The case is, however, otherwise with those derivatives of α-naphthol in which the azo group enters ortho to the hydroxyl. The dyestuffs having this constitution possess a brightness and stability superior even to that of the β-naphthol derivatives, and, moreover, they are bluer in shade, which renders them still more valuable. These ortho derivatives are, however, as a general rule, only formed directly when the para position is occupied—such as is the case, for instance, with α-naphthol-*p*-sulphonic acid. With the object of obtaining non-sulphonated ortho derivatives, the authors have prepared the acetyl compound of *p*-amido-α-naphthol and describe some of its reactions. The compound is obtained by mixing 100 grms. of dry *p*-amidonaphthol hydrochloride with 50 grms. of fused sodium acetate, and adding a mixture of 50 grms. of acetic

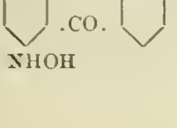
anhydride and 100 grms. of glacial acetic acid. The melt becomes warm and solidifies to a crystalline mass of naphthacetol (1.4-hydroxyacetnaphthalide). After distilling off a part of the acetic acid, the product is washed with water and re-crystallised from alcohol, when it forms thin white needles, melting at 187° C., soluble in the ordinary solvents. It is easily soluble in caustic alkalis and also dissolves in sodium carbonate solution and in ammonia. The shades obtained by combining it with diazo compounds are similar to those of β-naphthol, and by impregnating cotton cloth with its solution in sodium carbonate, various shades can be obtained by the use of different diazo compounds.

Diazo Compound from	Naphthacetol.	β-Naphthol.
Aniline.	Scarlet.	Orange-red.
ψ-Cumidine.	Bluish-red.	Yellowish-scarlet.
α-Naphthylamine.	Reddish-violet.	Bordeaux.
β-Naphthylamine.	Bordeaux.	Bluish-red.
Benzidine.	Violet.	Reddish-violet.
Tolidine.	Bluish-violet.	Violet.
Dianisidine.	Indigo blue.	Violet.

The dyestuff obtained from aniline has been examined, and crystallises from pyridine in matted needles, melting at 268° C. It was not found possible to hydrolyse the compound, and the methyl ether (m.p. 220° C.) was also stable. Naphthacetol also combines with diazosulphonic acids, yielding dyestuffs giving shades on wool and silk from an acid bath similar to but bluer than those from β-naphthol. All the azo compounds from naphthacetol can be reduced by means of stannous chloride (Ber. **21**, 3468; this Journal, 1889, 276), but the resulting amidonaphthacetol could not be purified, and it was therefore oxidised with ferric chloride to the corresponding quinone, which melted at 220° C. This compound, which on reduction with sulphurous acid gives the hydroquinone (m.p. 187° C.), reacts with o-phenylene diamine, yielding acetylphenonaphtheurhodine, and this, on hydrolysis with concentrated hydrochloric acid, forms dark red needles of phenonaphtheurhodine hydrochloride. The base crystallises from xylene and melts at 294° C. As a further proof that the azo group enters ortho to the hydroxyl, it is stated that the acetyl quinone obtained above is easily converted by alcoholic potash into oximidonaphthol and hydroxynaphthoquinone, and, moreover, it gives, like phenanthraquinone, a compound with bisulphites. The acetyleurhodine can also be obtained by the joint oxidation of naphthacetol and o-phenylene diamine with potassium ferricyanide. The nitroso derivative of naphthacetol crystallises from glacial acetic acid in golden needles, melting at 203° C., and gives an emerald-green precipitate with ferrous and a blackish-violet precipitate with cobalt salts. If in the above preparation of naphthacetol, the acetic anhydride be replaced by an equivalent weight of other anhydrides, corresponding derivatives are obtained. Thus, for instance, *butyrylamidonaphthol* is formed by treating a mixture of 30 grms. of amidonaphthol hydrochloride and 15 grms. of sodium acetate with 23 grms. of butyric anhydride and 100 grms. of glacial acetic acid. The compound crystallises from alcohol in colourless needles, melting at 161° C. Similarly obtained, *valerylamidonaphthol* melts at 205° C., and *benzoylamidonaphthol* melts at 229° C. This latter compound can also be obtained by the Baumann-Schotten method by cautiously adding an alkali to the aqueous solution of equimolecular proportions of amidonaphthol hydrochloride and benzoylchloride. All these derivatives in their behaviour to alkalis and diazo compounds act similarly to naphthacetol.—T. A. L.

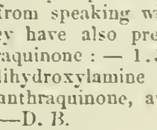
Anthraquinone, Hydroxylamine Derivatives of. R. E. Schmidt and L. Gattermann. Ber. 1896, **29**, 2934—2943.

1.5-dihydroxylamine anthraquinone—



is obtained by the reduction of 1.5-dinitro-anthraquinone with stannous chloride in an alkaline solution. On acidification a brownish-red precipitate is obtained, which is extracted with cold acetone and recrystallised from hot pyridine. The pure product forms dark brownish-red needles showing a green metallic lustre. It is sparingly soluble in acetone, alcohol, and wood spirit, but dissolves easily in hot pyridine. The dibenzoate crystallises in long garnet-red needles melting at 188°, and the tribenzoate forms orange-yellow crystals melting at 228°. For its conversion into amidohydroxyanthraquinones, 10 grms. of the hydroxylamine derivative are dissolved in 850 grms. of concentrated sulphuric acid and 150 grms. of water. The mixture is heated rapidly to 100°—110°, the reaction being completed as soon as the solution assumes a yellow colour. The chief products of the reaction are two isomeric diamido compounds, called α and β by the authors, a third substance, γ, being produced in small quantity. Their separation is effected by the difference in their behaviour towards solvents. The β-compound dissolves easily in hot dilute sulphuric acid (15 per cent. H_2SO_4), and separates out again on cooling. α and γ are more insoluble in sulphuric acid than β. The latter is readily soluble in alkalis, whilst α and γ are almost insoluble; γ is easily soluble, α sparingly soluble, in acetone. The following are the distinctive properties of the three compounds:—α dissolves in hot nitrobenzene with a violet-blue colour, and forms long lustrous needles resembling brass in appearance; β is readily soluble in acetone, forming a red solution, and crystallises in green lustrous crystals. Both compounds have the composition $C_{14}H_4O_2(OH)_2(NH_2)_2$. They dissolve in sulphuric acid with a yellow colour, and on adding boric acid to the solution, α assumes a pure blue, and β a violet colour, exhibiting characteristic absorption spectra, consisting in the case of α of a sharp line situated in the red and a fainter line in the green, whilst the lines given by β are situated in the blue rays; γ gives a yellow colour with sulphuric acid, which boric acid does not alter. However, on heating it to 80°—100° in sulphuric monohydrate with a 10 per cent. solution of boric acid, a bluish-red solution is obtained, showing a cinnabar-red fluorescence and exhibiting an absorption spectrum in the green rays, the blue being entirely extinguished.

The authors have prepared the same compounds by the action of 1.5-dinitro-anthraquinone on sulphur sesquioxide (sulphur dissolved in fuming sulphuric acid). Having obtained the compound α by synthesis from anthrarufin, they conclude that it is di-p-amidoanthrarufin, its constitution being represented by the formula—



Analyses of the compound γ gave numbers pointing to the formula for diamidotrihydroxyanthraquinone. However, owing to the difficulty experienced in the purification, the authors refrain from speaking with absolute certainty on this point. They have also prepared the following derivatives of anthraquinone: — 1.5-nitrohydroxylamine anthraquinone, 1.8-dihydroxylamine anthraquinone, 1.8-nitrohydroxylamine anthraquinone, and 1-monohydroxylamine anthraquinone.—D. B.

Coal-Tar Dyestuffs [for Colouring Eatables, &c.]; The Behaviour of, towards the Process of Digestion. H. A. Weber. J. Amer. Chem. Soc. 1896, **18**, 1092—1096.

See under XVIII. A., *page* 158.

Chrysoidine, Behaviour of, towards Cholera Bacilli. A. Blachstein. Münchener med. Wochenschr. 1896, **43**, 1067.

See under XXIV., *page* 168.

PATENTS.

Azo Dyes, Impts. in the Manufacture of. S. Pitt, Sutton. From L. Cassella and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 2446, Feb. 3, 1896.

The azo dyestuffs formed from salicylic acid have the power of forming sparingly soluble lakes with chrome mordants, by virtue of their possessing a hydroxyl and a carboxyl group ortho to one another. The lake-forming property is, however, not so well defined as in the alizarin dyestuffs, and with a view to increasing it, the patentees have introduced two salicylic acid groups into an azo dyestuff by combining with *m*-diamines, diazo compounds derived from substances having the general formula—

$$(1.2.4)C_6H_3.OH.CO_2H.N:N.X.NH_2$$

where $X.NH_2$ represents an amine like α-naphthylamine. The combination takes place in such a way that the *m*-diamine is combined either with two molecular proportions of the above substances, or else first with diazosalicylic acid and subsequently with the diazo-azo compound. The most valuable products are obtained from nitro-amidosalicylic acid, since the introduction of the nitro group increases the fastness to light and also produces a yellower shade. Moreover, no change in shade is to be observed on chroming, which is the case when salicylic acid itself is used. By varying X in the above general formula the shades may be varied in the direction either of the red or violet end of the spectrum, α-naphthylamine, however, giving the darkest shades. Easily soluble dyestuffs are obtained by employing 1.3′ or 1.2′-naphthylamine sulphonic acid in place of α-naphthylamine. The following is a typical example :—The diazo compound from 39·6 kilos. of (1.3.4.5) nitro-amidosalicylic acid and 14 kilos. of sodium nitrite is combined with 28·6 kilos of α-naphthylamine in a solution faintly acid with hydrochloric acid. When the combination is complete, the dyestuff, after filtering, is dissolved in soda-lye, 14 kilos. of sodium nitrite added, and the mixture is acidulated at about 0° C. The sparingly soluble diazo compound thus formed is filtered off and added to a solution containing 10·8 kilos. of *m*-phenylene diamine kept alkaline with sodium carbonate. The dyestuff separates as a dark brown precipitate.—T. A. L.

Disazo and Trisazo [Cotton Browns and Blacks] Colours or Dyes, Impts in the Manufacture of. T. R. Shillito, London. From J. R. Geigy and Co., Basle, Switzerland. Eng. Pat. 2771, Feb. 6, 1896.

The specification relates to the production of dyestuffs from the alkyl derivatives of 2.1′.3′-amidonaphthol sulphonic acid by combination with tetrazo compounds. The usual *p*-diamines are employed, such as benzidine, diamidodiphenyltolyl, tolidine, ethoxybenzidine, diamidodiphenol ether, *p*-phenylene diamine, 1.4.3′-naphthylene diamine sulphonic acid, &c. Either symmetrical or mixed disazo dyestuffs can be obtained, and in the case of the latter, certain of them can be further diazotised and combined. The shades obtained from the disazo compounds vary from brown to violet, greyish-blue, grey, and black, and dye unmordanted cotton. The following example gives the quantities employed in the formation of a trisazo dyestuff :—75·6 kilos. of the mixed disazo compound obtained from equimolecular proportions of tetrazodiphenyl, 2.1′.3′-methylamidonaphthol sulphonic acid, and 2.1′.3′-amidonaphthol sulphonic acid, is suspended in 1,000 litres of water together with 35 kilos. of hydrochloric acid (21° B.) and diazotised with 7 kilos. of sodium nitrite. The diazotisation is complete after several hours, and the resulting compound is poured into 300 litres of water containing 11 kilos. of resorcinol and 30 kilos. of sodium carbonate. The dyestuff forms a black precipitate, which is filter-pressed and dried. It gives silver-grey to black shades on unmordanted cotton.—T. A. L.

New Sulpho Acid [1.4.2-Naphthylene Diamine Sulphonic Acid] and of [Blue] Colours derived therefrom, Production of a. Levinstein, Lim., and I. Levinstein, Manchester. Eng. Pat. 2946, Feb. 10, 1896.

The 1.4.3′- or 2′-naphthylene diamine sulphonic acid of Eng. Pat. 15,444 of 1893 (this Journal, 1894, 801), is decomposed by nitrous acid. The acid of the present specification, however, gives a diazo compound which reacts with amines, phenols, and their derivatives, and the resulting products are capable of being again diazotised, and give, on combination with various components, disazo dyestuffs yielding yellow to deep black shades. This last operation can also be performed on the fibre. In addition to its employment for the production of azo dyestuffs, the new 1.4.2-naphthylene diamine sulphonic acid can also be used in the manufacture of safranines, indulines, and allied compounds. The acid is obtained by combining aniline or other suitable aromatic amine with 1.2-naphthylamine sulphonic acid and reducing the resultant product with iron and hydrochloric acid. After making alkaline, and blowing off the aniline with steam, the new acid crystallises out on acidifying the filtrate. It oxidises easily in a neutral or alkaline solution, forming a yellow crystalline substance, which has a greenish-yellow fluorescence in an aqueous solution. The new acid is converted into a diazo compound by dissolving 23·8 kilos. of it in 200 litres of water containing 5·3 kilos. of sodium carbonate, precipitating with 100 kilos. of 30 per cent. acetic acid, and diazotising with 7·2 kilos. of sodium nitrite. The compound so obtained, is gradually added to an alkaline solution of 35 kilos. of β-naphthol disulphonic acid R. The dyestuff, after salting out, is filter-pressed and dried. It gives blue shades on wool from an acid bath. This dyestuff can then be further diazotised by dissolving 62 kilos. of it in 300 litres of water, acidulating with 70 kilos. of hydrochloric acid (20° B.), and diazotising with 7·2 kilos. of sodium nitrite. The solution, which originally was violet-blue, changes to a deep red, and the new diazo compound, which can, if required, be salted out, is combined with an alkaline solution of β-naphthol. The disazo colour produced, forms after salting out, a dark blue precipitate, which dyes wool deep blue to black from a neutral or acid bath.—T. A. L.

Colouring Matter [Bluish-Green] from Substituted Fluoresceïnes, Manufacture of. O. Imray, London. From "The Farbwerke vormals Meister, Lucius, and Brüning," Höchst a/M., Germany. Eng. Pat. 3,000, Feb. 10, 1896.

According to Baeyer (Annalen, **183**), when tetrabromofluoresceïn is strongly heated with concentrated sulphuric acid, it is converted into bromocœruleïn, but the yield is poor. Dyestuffs of the nature of cœruleïn and in good yield, may, however, be obtained by the employment of monohydrated or fuming sulphuric acid. Thus, 20 kilos. of tetrabromofluoresceïn are added to 100 kilos. of fuming sulphuric acid containing 20 per cent. of SO_3, and the mixture is kept at 125° C. until a sample neutralised with alkali and mixed with brine no longer gives a coloured filtrate. The excess of fuming sulphuric acid is then decomposed by adding 40 kilos. of sulphuric acid of 66° B. and 100 kilos. of 60° B., after which the dyestuff is precipitated by pouring the melt into ice and water. An almost black paste is thus obtained which can be used directly for dyeing. It is insoluble in water, very sparingly soluble in alcohol, but dissolves in sulphuric acid with a cherry-red, and in alkalis and alkaline carbonates with a bluish-green colour. The alkali salts can be salted out from these solutions. The product dyes chrome-mordanted wool bluish-green, very fast to air and fulling. The dyestuff undergoes a further change and gives much greener shades by heating the dry substance with 10 times its weight of ordinary sulphuric acid to 130° C. for 3—4 hours and subsequent precipitation with water, when it is filtered off and washed. The two processes can, of course, be combined, if it be desired, to at once obtain the greener shade. Other substituted fluoresceïns suitable for this invention are : disubstituted bromofluoresceïn, di-iodo-α-tetra-iodo fluoresceïn, tetrabromo- and tetra-iodo-dichlorofluoresceïns, tetrabromo- and tetra-iodo-tetrachlorofluoresceïns.—T. A. L.

Polyazo Dyestuffs [Blacks], Impts. in the Production of. S. Pitt, Sutton. From L. Cassella and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 3028, Feb. 10, 1896.

This is an extension of Eng. Pats. 16,699 of 1889; 5141 of 1893; 9529 and 25,018 of 1894 (this Journal, 1890, 935;

1894, 246; 1895, 478 and 1041). The dyestuffs there mentioned are obtained by using *m*-phenylene diamine, *m*-tolylene diamine, and resorcinol as end-components. It has now been discovered that these substances can be replaced by their azo derivatives, and if these be suitably chosen, new dyestuffs having valuable properties may be obtained. The general formula of the new compounds is—

$$P \left< \begin{array}{l} N:N.\gamma\text{-amidonaphthol sulphonic acid} \\ N:N.m\text{-diamine-}N:N.R.OH \end{array} \right.$$

where P is the radicle of a *p*-diamine, and N:N.R.OH the diazo compound of an amidophenol or amidonaphthol sulphonic or carboxylic acid. The following is a typical example. The tetrazo derivative from 24·4 kilos. of dianisidine is combined with 24 kilos. of γ-amidonaphthol sulphonic acid in an alkaline solution, and the intermediate compound thus obtained is run into a solution in sodium carbonate of the product obtained by the diazotisation of 15·3 kilos. of amidosalicylic acid, and combination with 11 kilos. of *m*-phenylene diamine in an acetic acid solution. A black precipitate gradually forms. The product dyes unmordanted cotton a deep black, and can be fixed with chrome mordants. As *p*-diamines, the most useful are benzidine, tolidine, diamidodiphenol ether, diamidodiphenylmethane, and *p*-phenylene diamine, whilst R can be either γ-amidonaphthol sulphonic acid, 2.1′.3.3′-amidonaphtholdisulphonic acid, amidosalicylic acid, or *p*-amidophenol sulphonic acid.—T. A. L.

New Phthaleïne Colouring Matters [*Yellows, Reds*], *Manufacture of.* O. Imray, London. From "The Farbwerke vormals Meister, Lucius, und Brüning," Hoechst a/M., Germany. Eng. Pat. 3966, Feb. 21, 1896.

Certain phthaleïns, when treated with ammonia or primary bases of the fatty or aromatic series, are converted into new and valuable dyestuffs. The resultant products still possess an acid character and give alkaline salts, and are obtained from the nitrofluoresceïns and their derivatives. For example, 10 kilos. of dinitrofluorescein and 10 kilos. of water are stirred to a paste with 7·5 kilos. of 22—24 per cent. ammonia The mass heats spontaneously to about 40—45° C., and after a short time thickens, and shining laminæ separate out. Brine is added, and the mass is filtered, the residue consisting of the ammonium salt of the new product, which can be purified by dissolving in water and precipitating with salt. The product dyes wool a pure yellow, unaffected by alkalis. The colour dyes evenly and is fast to light. If, in the above example, the water and ammonia be replaced by 15 kilos. of aniline and the melt be heated to 120° C., condensation with separation of water takes place, and the resultant dyestuff, after removing the excess of aniline with hydrochloric acid, dyes wool a reddish-yellow. A brick-red dyestuff for wool is obtained, as in the first example, by employing the same quantity of dichlorodinitrofluorescein in place of the dinitrofluorescein.

—T. A. L.

Greenish-Blue Colouring Matters or Dyes of the Malachite Green Series, Impts. in the Manufacture of. T. R. Shillito, London. From J. R. Geigy and Co., Basle, Switzerland. Eng. Pat. 5068, March 6, 1896.

In Ger. Pat. 80,982, the patentees described a process for the production of the orthosulphonic acid of Malachite Green, and observed that the position of the sulphonic acid group ortho to the methane carbon conferred upon the product, properties of dyestuffs belonging to the Patent Blue series. In the present specification a method is described for the direct production of similarly constituted colouring matters from o-benzaldehyde sulphonic acid. This product can be obtained by heating together *o*-chlorobenzaldehyde with an aqueous solution of neutral sodium sulphite in an autoclave to 190°—200° C. for about 8 hours. The formation of the new dyestuff is carried out as follows:—186 kilos. of a 10 per cent. solution of *o*-benzaldehyde sulphonic acid is boiled with 30 kilos. of dimethylaniline and 3 kilos. of sulphuric acid for about 15 hours. Sodium carbonate is then added, and the excess of dimethylaniline is distilled off. After dilution to 5,000 litres, the solution is made acid with hydrochloric or acetic acid, and the resulting leucosulphonic acid is oxidised with lead peroxide. The lead is precipitated by means of sodium sulphate, and the filtered solution is made alkaline with ammonia and evaporated to dryness. If the dimethylaniline in the above example be replaced by 60 kilos. of ethylbenzylaniline sulphonic acid and the leucosulphonic acid obtained, be oxidised with 100 kilos. of a 30 per cent. lead peroxide paste, the resulting colouring matter forms a bronzy powder, soluble in water with a blue colour, and dyeing wool a pure greenish-blue from an acid bath.—T. A. L.

New Colouring Matters Dyeing from Blue-Green to Blue, and of New Chloro Derivatives and Sulphonic Derivatives of Benzoic Aldehyde therefor; Manufacture of. O. Imray, London. From "The Society of Chemical Industry in Basle," Basle, Switzerland. Eng. Pat. 25,128, Nov. 9, 1896.

A process has been formerly described for obtaining bluish-green colouring matters, which dye from an acid bath, by condensing benzyl-*o*-toluidine or benzyl-*o*-toluidine sulphonic acid with benzaldehyde and oxidising the resultant leuco compound. According to the present specification, the benzaldehyde is to be replaced by a whole series of chloro and sulphonic derivatives of benzaldehyde, yielding in a similar manner derivatives of triphenylmethane, which on oxidation give bluish-green to blue dyestuffs. The new sulphonic acids of benzaldehyde are obtained either by sulphonating o-chlorobenzaldehyde, when chlorobenzaldehyde sulphonic acid ($CHO:Cl:SO_3H$ = 1:3:6) is formed, or else by the action of sodium bisulphite on this latter product or on dichlorobenzaldehyde under pressure in an autoclave. The products so obtained are condensed with two molecules, the same or different, of secondary or tertiary aromatic amines, such as methyl-*o*-toluidine, dimethyl- or diethylaniline, methylbenzylaniline sulphonic acid, &c., forming leuco compounds, which on oxidation with peroxides or chromic acid yield greenish-blue to blue dyestuffs. The following may serve as an example of the method employed:—120 kilos. of an 8 per cent. solution of *o*-benzaldehyde sulphonic acid and 28 kilos. of benzyl-o-toluidine sulphonic acid are heated on the water-bath for about eight hours. The melt is dissolved in 200 litres of water and the leuco trisulphonic acid is precipitated with salt. After drying, 20 kilos. of the dry product are dissolved in 500 litres of water and oxidised after adding 40 kilos. of 10 per cent. sulphuric acid with the requisite quantity of lead peroxide. When the reaction, which takes about one hour, is complete, the mixture is neutralised with chalk, filtered, and evaporated. The dyestuff forms a dark green powder easily soluble in water to a blue solution, and gives blue shades on wool from an acid bath. (See also this Journal, 1893, 672.)

—T. A. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

Pentosans, Proportion of, in Cotton-Wool. H. Suringar. Chem. Zeit. Rep. 1896, 290.

See under XXIII., *page* 167.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Defects in Dyeing, and their Remedies. J. J. Hummel. J. Soc. Dyers and Colourists, 1897, 2—12.

Defects due to Fibre.—The white specks occasionally noticed in plain-dyed calicoes have been shown by Crum to be due to unripe fibres ("Dead cotton"), which have no affinity for certain colouring matters. The remedy of this defect lies mainly in the hands of the spinners. The ginning, scutching, carding, &c., should be as perfect as possible, not only to remove "dead cotton," but also to get rid of bits of cotton seed, the oil of which is apt to cause similar defects (see also this Journal, 1885, 720). In some cases, however, the dyer may hide the defect by selecting such dyestuffs as possess the property of covering even unripe cotton fibres.

In wool, too, there are occasionally abnormal fibres known as "kemps," which have little or no affinity for colouring matters, and cause imperfections in dyed woollen cloth similar to those due to "dead cotton" in dyed calicoes. Here the remedy rests partly with the sheep farmer and partly with the wool sorter. Here, again, the dyer may occasionally cover the defect by choosing such colouring matters as tend to dye the kemps, by chlorinating the wool, or by slightly modifying the dyeing process (see J. Soc. Dyers and Colourists, 1896, 141).

But, apart from such structural defects, even different qualities of the same fibre often possess a different attraction for colouring matters, and an irregularity in the dye of woollen cloth is often due to an injudicious blending of different qualities of wool. Irregular weaving, the introduction of yarns not uniform in diameter, of different tensions, or twisted in opposite directions, are equally liable to cause irregularity in the appearance of the dye.

Again, many colouring matters, *e.g.* induline, dye the individual fibres so irregularly that the cloth or yarn exhibits a speckled appearance. This defect may be overcome by oxidising the fibre previous to dyeing, either by means of potassium bichromate or a solution of bleaching powder.

Brown discolorations or dark spots have been traced to the well-known presence of sulphur in wool, which produces dark-coloured sulphides of copper, tin, or lead, with which metals the material had accidentally come in contact. These spots are formed the more readily when the wool is in an alkaline condition. In special cases it may be desirable to remove the active sulphur from the wool by repeated successive treatment of the wool with calcium hydrate, hydrochloric acid, and water, as proposed by Chevreul.

Defects caused by Operations preliminary to Dyeing.—Special care must be exercised by the bleacher to prevent rust stains, by the immediate removal of the cotton from the kier after the soda-boil, by a careful search for iron nails in the roof or elsewhere, by periodical whitewashing of kiers, and painting of iron pillars, brackets, &c., and the use of wooden pegs instead of nails in wooden benches, &c. Holes, cuts, and tears in the cloth must be prevented by avoiding tremulous boiling in the kiers, or the casual entrance of cinders into the washing machines, and by the renewal of cracked pot-eyes or wooden rails, when the latter have worn to a cutting edge by the friction of the travelling cloth.

Cloth is often tendered by an insufficiency of liquor in the kiers, by undissolved particles of chloride of lime, and allowing piles of cloth soaked with acid standing too long or exposed to air currents, which concentrate the acid in certain portions. The author mentions an interesting case investigated by Witz, who traced the brown spots, frequently accompanied by minute holes, which often appear in bleached calicoes after steaming, to the presence of undissolved particles of bleaching powder on the cloth. Witz also found that with strong solutions of bleaching powder, oxycellulose is formed, which greatly increases the affinity of the cotton fibre for certain basic colouring matters, notably so for Methyl Violet; and his investigations led him also to the discovery of the oxidising action which bleaching powder exerts on animal fibres, thereby increasing their affinity for dyestuffs.

In connection with wool, the all-important preliminary operation is that of scouring. Caustic soda, and all so-called substitutes, should be avoided, and only well-recognised scouring agents, such as sodium carbonate, soap, ammonia, &c., should be used. Excessive concentration or high temperatures in the solution, and an unduly prolonged application, are to be avoided. Equally important is the necessity of completely removing the scouring agent from the wool by thorough washing, as in many cases the presence of alkali in the scoured wool may lead to defects in mordanting or dyeing.

Skin-wool, which has been removed from the pelt by lime, and invariably highly charged with calcium carbonate, can only give poor, dull, indifferent colours, unless the lime is previously removed by rinsing such wool with dilute hydrochloric acid, followed by washing and scouring. Wool, which has been submitted to the action of sulphuric acid in the process of "carbonising," has acquired, by reason of this treatment, a much greater attraction for colouring matters than ordinary wool; so that an imperfect blending of "extracted" with ordinary wool may lead to irregularity of colour. The same may happen if the acid has not been completely removed from the "extracted" wool by washing, or, better still, by the use of sodium carbonate solution.

Defects due to Water.—The necessity for soft water, or its correction by well-known methods where calcareous waters only are available, is insisted on.

Defects due to Mordanting.—Full and well-nourished colours can only be obtained with mordant colours, when the mordant used has been in proper condition and rightly applied. With wool, if the mordanting bath is too acid, comparatively little mordant is fixed on the fibre; and if "too neutral," the mordant is insufficiently, irregularly, or superficially fixed, giving poor, speckled-looking colours which rub off. The right proportion of mordant to the amount of dyestuff, the quantity of material and water employed, should be experimentally determined and then rigidly adhered to.

Over-chroming is another frequent cause of poor and badly fixed colours, which defect can never occur if the chromium is fixed as a chromic oxide salt. The author deprecates the long-continued use of the same chrome-bath with the customary freshening up.

Similar defects as with chrome may also occur with aluminium, tin, and iron mordants, if an insufficiency of tartar or similar assistant be employed.

Other defects connected with bichrome mordanting are the irregularities caused by the reducing action of light on exposed portions of the mordanted material, and by allowing the mordanted goods to lie in a heap before washing for an undue length of time, which causes "feeding" in the lower portions of the mass, subsequently dyeing to a darker colour.

Alum for mordanting purposes should be free from iron; and if iron vessels only are available, a block of zinc should be introduced into the bath to prevent any iron from being dissolved. In calico-printing, the saddening influence of iron, perchance present in the alumina employed, is counteracted by the addition of stannous chloride, which, by its reducing action, maintains the iron in the soluble ferrous state. The substitution of the neutral aluminium sulphocyanide for the acetate has removed the possibility of iron being dissolved from the steel "doctors" during printing.

Defects due to the Dyestuffs employed.—As an instance of mutually incompatible dyestuffs the author mentions a case where a mixture of nitro-alizarin and an alizarin bisulphite colour was used. The mixture had often been used with success, but now and then the colours came up entirely off shade. An inquiry into the circumstances elicited the fact that if the colours were mixed beforehand, and allowed to stand for some time before being added to the dye-bath, the nitro-alizarin was affected by the reducing action of the sulphurous acid of the bisulphite colour—a decomposition which did not take place if added in the dilute state direct to the dye-bath.

Similar defects, which are either inherent in the dyestuffs employed, or are due to the methods of application, are then discussed in connection with aniline black, indigo, &c., and methods are suggested which may prevent the rubbing off of vat-dyed woollen cloth.

Defects due to Machinery usually comprise streaks, spots, stains, cuts, tears, &c. Spots are frequently caused by water drops from the roof or from the interior of a hood, *e.g.* over drying cylinders, which should be avoided by suitable precautions. Listing may be due, among other causes, to the arrangement of the dye-vessel, the position of the steam-pipe, &c. Other marks in the pieces may be due to friction, or too vigorous working with a wooden rod.

Defects due to the Methods employed.—These are of the most varied character, and must, as a rule, be ascribed to ignorance or want of experience on the part of the dyer. Full shades on wool with many dyes are apt to bleed and rub off if not boiled long enough during the dyeing process. This is the case, for example, with the Congo and the Alizarin

colours. On the other hand, long boiling must be avoided when dyeing delicate shades with logwood, otherwise the colours are rendered dull and off shade, owing to the oxidation and destruction of the colouring matter. By slightly acidifying the bath with acetic acid, this defect may be avoided.

Entering the goods into a dye-liquor which is too hot, tends to a superficial fixing of the colouring matter, which is usually accompanied by rubbing off or irregularity of colour. On the other hand, entering at too low a temperature may, with certain dyestuffs, *e.g.* Acid Violets, cause the production of spots, due to a separation of the colour acid in a tarry form. By entering at about 70°—100° C., the colour acid remains in perfect solution, and at the same time dyes level shades.

No fixed rule as to the most suitable temperature at which to enter the goods can be given, as this varies with the character of the dyestuffs employed. But it is advisable always to enter at the highest temperature which is consistent with the production of bright and level shades.

Bronzy patches with aniline colours, or irregularity of shade, may be caused by freshening up the bath with dyestuff without lifting the goods from the liquor, or by omitting the filtration of the colour solutions.

Defects caused in the Finishing Operations.—A very common one is that of pieces being "listed," *i.e.*, appearing of a paler or darker colour at the selvedges, which is due to "feeding," by the pieces having been left to drain in a heated and unwashed condition, or to the operation of blowing steam through the pieces while wrapped tightly on perforated rollers. The steam, in tending to force its way between the folds of cloth towards the selvedges, causes there an accumulation of any loose impurities that may have been present in the cloth, and are carried along with the steam.

A common source of annoyance experienced with many colours during the scouring or milling of dyed pieces is that of "bleeding," or the staining of neighbouring fibres in the cloth. To prevent this, only such colouring matters should be used as are not liable to this defect. Certain of the Congo dyestuffs, *viz.*, those containing the salicylic acid group, have the property of forming lakes; and with these the defect of "bleeding" is cured by passing the dyed cotton through various metallic solutions, whereby the colouring matter is fixed as an insoluble salt. Alizarin colours and some of the basic colours fixed with tannic acid, &c., may be prevented from bleeding on to neighbouring parts by adding to the soap-bath some finely divided tannate of antimony precipitate, which immediately attracts any loose basic colour in the soap-bath, and renders it insoluble. (See also this Journal, 1896, 590.)—I. S.

Crimp Patterns on Mousseline de Laine, A Method of Producing. E. Knecht. J. Soc. Dyers and Colourists, 1895, 21—22.

By subjecting wool to the treatment of certain chemical agents, its felting properties may be either increased (*e.g.*, by means of mercury nitrate) or decreased (*e.g.*, by means of chromic acid or free chlorine).

Based on this well-known fact, the author has worked out the following process for producing permanent crimps on light woollen cloth. The scoured mousseline de laine is printed in stripes of ½ in. or more with sulphuric acid of 10°—20° Tw., suitably thickened with British gum. It is then dried and passed for about two minutes through a solution of bleaching powder at about 2° Tw. The material is well washed to remove acid, &c., and is then milled with soap until the crimp is sufficiently prominent. The crimped cloth may subsequently be dyed any colour, but with some colouring matters there will be a distinct difference of shade between the plain and raised portions. If the printed stripes are too narrow, the crimping does not succeed well. Equally unsatisfactory were the experiments made with thick material.—I. S.

Soupled Silk, The Dyeing of, by the Aid of Formaldehyde. H. Silbermann. Färber Zeit. **7**, [30], 479—480.

Hitherto it has been impossible to dye a full black with logwood from a soap-bath on soupled silk, because, as is well known, silk which has been soupled is made worthless by even very dilute soap solutions at temperatures above 65° C. The author has now found that the process patented by Ris-Kummer (Eng. Pat. 15,152, 1895; this Journal, 1895, 965), of rendering the gum insoluble by means of formaldehyde in hot water, is also applicable to soupled silk, which absorbs the formaldehyde very energetically. He has treated the silk for two hours in a bath with 3 per cent. of a 40 per cent. solution of aldehyde (commercial "formaline"), rinsed, and dyed it with logwood in a soap-bath. The results are stated to have been most satisfactory: the fibre has lost very little, whilst the lustre and "feel" have been improved.

The treatment with aldehyde must take place subsequently to the weighting, as the absorbent power of the silk is considerably lowered by this process.—I. S.

Milling with Acids. Leipziges Monatsschr. für Text. Ind. Through Färber Zeit. **7**, [29], 466—468, and [30], 486—487.

The article is based on occasional notes, and embodies the experience of the author whilst working the process. The following are the chief points of interest:—

Cloth dyed in such a manner that it may be subjected to the acid treatment without detriment to its colour, is not much inferior in general appearance and weight to goods milled with soap. But goods that had been dyed in the wool (*i.e.* before spinning) are liable to have their shade slightly altered. This, however, can be guarded against in the preliminary process. The author is of opinion that acid-milled cloth feels more spongy and less firm than cloth milled with soap; the latter is also better felted. Pieces milled with acids are certainly not better than those milled with soap.

Grey pieces that have to be "carbonised" and are to be dyed in the piece are milled directly after carbonising, requiring only the requisite quantity of water. Cloth that has been dyed in the wool, and which does not require carbonising, is first carefully scoured, and, after removal of all surplus moisture by the hydro-extractor, is milled with sulphuric acid of 2°—3° B. In milling by the acid process it is essential that the goods should previously be well scoured, as they cannot be subsequently treated with soap without deterioration. Care must be taken, therefore, not to soil the pieces during or after the milling process.

With the acid process it is more difficult to mill lengthwise than with the soap process. Hence, when desiring to mill lengthwise, the soap process is preferable. It is also of importance that the goods should not be allowed to remain long after milling before the acid is removed, since the continued action of the acid may affect the shade of colour; moreover, continued contact of the goods with metals, whilst saturated with acid, may cause stains.

The author says that, on the whole, the acid process of milling has so far given satisfactory results. There is a considerable saving in soap, whilst in the case of pieces that have to be carbonised, no additional acid even is required, that already in the cloth, being sufficient.

The relative merits of the two processes are then discussed, and, with the exception of cost, which is in favour of the acid process, the author is inclined to think that the soap process has many advantages over the former. The colours, in the case of dyed goods, are more brilliant by the latter process, and there is less risk of stained or soiled goods, which, in the case of the acid process, may be a serious matter, since the goods cannot be subsequently scoured in soap liquors without deteriorating their quality.

The most suitable acid is sulphuric acid. There should not be an excess of moisture, as better and more uniform results are obtained when the cloth is not too wet. Of late, acid of only 1½° B. has been used, with better results, the shades being less liable to injury, if the goods were allowed to lie in the wet state than with the stronger acid used formerly.—I. S.

Paranitraniline Red in Dyeing and Printing. A. G. Green. J. Soc. Dyers and Colourists, 1897, 17—21. (See also this Journal, 1896, 590, 649, and 901.)

Until quite recently it was believed that a very low temperature was a necessary condition for the diazotisation of

p-nitraniline, and that the diazo compound was very unstable. This is now known to be erroneous, for by proper conditions the diazotisation can be readily performed without the employment of ice, or taking any particular care as to temperature. Moreover, instead of being easily decomposed, the diazo solution is very stable, and will keep almost unchanged for two or three weeks. It is only when mixed with sodium acetate that it becomes unstable.

The following points must be observed for successful diazotisation :—(*a*) The mixture of *p*-nitraniline and hydrochloric acid must be fairly concentrated. (*b*) The nitrite should be added without intermission, but slowly. (*c*) Vigorous stirring during the operation. (*d*) A sufficient excess of acid. (*e*) Copper vessels must not be used. (*f*) The hydrochloric acid should be as pure as possible.

10 lb. of *p*-nitraniline and 2 gals. of cold water are briskly stirred in an enamelled or wooden vessel to form a perfectly even paste. $2\frac{1}{2}$ gals. of hydrochloric acid of 28° Tw., are then added, stirring for another 15 minutes, after which 6 galls. more of cold water are added. To this mixture is added, *all at once*, with constant stirring, a solution of $5\frac{1}{2}$ lb. of sodium nitrite in $1\frac{1}{2}$ gals. of water. If, after 15 or 20 minutes' stirring, the solution does not give a blue colour when tested with "iodine paper," a little more nitrite solution is added, until the latter is present in slight excess.

Directions for printing are given.—I. S.

Lactic Acid as an Assistant in Mordanting Wool. C. Archer. Färber Zeit. **7**, [30], 477—479. (See also this Journal, 1896. 196 and 540.)

As an acid, lactic acid is weaker than either tartaric or oxalic acids, and ranks about equal with the very feeble chromic acid. This is argued from the fact that whilst lactic acid can liberate chromic acid from neutral chromates, chromic acid can also liberate lactic acid from neutral lactates. All the chromic acid, however, is not liberated by the lactic acid (unless the latter be in large excess), and the mixture consists of an acid chromate and a lactate. These salts do not decompose each other, even on boiling. But if both acids be present in the free state, they react very energetically, the solution turning green in a very short time even at ordinary temperatures.

The action of tartar, tartaric acid, and more especially of oxalic acid on chromic acid, is very slow and feeble. By boiling, the action of tartaric acid is increased, the solution turning green. But a mixture of the solutions of oxalic and chromic acid retains its yellow colour, even after prolonged boiling.

It follows from this that lactic acid is a much better reducing agent for chromic acid than either oxalic or tartaric acid, but in order to use it to best advantage, both acids should be present in the free state. This may be effected by adding to the mordanting bath the requisite quantity of sulphuric acid to liberate the chromic acid from the salt employed.

Under such conditions the whole of the chromic acid present may be quantitatively reduced to chromic oxide. Thus, if a solution of 30 parts of lactic acid (50 per cent.), 15 parts of potassium bichromate, and 5 parts of concentrated sulphuric acid in 1,000 parts of water, be boiled for two hours, there results a green precipitate of chromic oxide, and a clear, colourless liquid, free from any trace of chromic acid, the originally strongly acid solution having become almost neutral.

If an excess of sulphuric acid be taken, then chromium sulphate or chrome alum would be formed instead of chromic oxide. On the other hand, it is necessary that the lactic acid should be present in excess; and it has been found that for the complete reduction of the chromic acid, the quantity of lactic acid (50 per cent.) to be used, should be twice the weight of that of the bichromate. If less be taken, the reaction does not proceed as above stated. If, for instance, a solution of 25 parts of lactic acid (50 per cent.) and 30 parts of potassium bichromate be boiled, a brown, unalterable precipitate of basic chromium chromate is speedily formed, leaving potassium bichromate in solution.

In the presence of wool the reactions proceed as above described; but inasmuch as the wool (being of a basic character) absorbs some of the acid, it is necessary to increase slightly the quantity of sulphuric acid.

Based on these results, the following proportions and mode of working are recommended. The well-scoured and rinsed wool is worked for 20 minutes in a bath at 75° C., to which are gradually added 1·5 per cent. of concentrated sulphuric acid, 1·5 per cent. of potassium bichromate dissolved in water, and 3 per cent. of lactic acid (50 per cent.). The temperature is then raised to boiling, and so maintained for half an hour, when the wool will be found to be of a pure green colour, and the bath perfectly colourless. The above quantities are said to be sufficient for the fullest shades.

For thick felts, army cloths, &c., which are difficult to penetrate, the following modification of the process is recommended. The goods are first boiled for half an hour with the bichromate and sulphuric acid only, when the steam is shut off, the lactic acid added, and the goods run for half an hour longer. The temperature is then again raised to boiling, and the goods worked for another 20 minutes.—I. S.

Albumins, Putrefaction of. O. Emmerling. Ber. **29**, 2721—2726.

Wheat-flour glutin was separated from starch and fatty matters, and mixed with water, calcium carbonate, and a little calcium phosphate and magnesium sulphate. After sterilising, a pure culture of *proteus vulgaris* was added, and the whole maintained at a temperature of 37°. After four days a considerable quantity of gas was evolved, containing 46 per cent. of CO_2, 38 per cent. of hydrogen, and 16 per cent. of nitrogen. After the sixth day a smell of putrefaction was noticed, and in 14 days the whole mass was in solution. The following substances were recognised in the liquid: phenol, ammonia, trimethylamine, betaïne, formic, acetic, butyric, and small quantities of higher fatty acids. No ptomaïnes could be detected.

Egg albumin was examined in a similar way, except that *staphylococcus pyogenes aureus* was used in place of *proteus* and the experiment was conducted in an atmosphere of hydrogen. Little gas was evolved, and in 14 days the albumin was converted into a thin liquid. Phenol, indole, scatol, trimethylamine, ammonia, formic, acetic, propionic, butyric, oxalic, and succinic acids, with small quantities of higher fatty acids, were the only products isolated.

Experiments destined to decide whether staphylococcus contains an enzyme capable of splitting up polysaccharides, showed that this is not the case for cane sugar, glycogen, maltose, and milk sugar.—T. E.

PATENTS.

Textile Fabrics, An Improved Method of and Apparatus for Printing upon. A. S. Young and W. Rumney and Co., Ltd., Ramsbottom. Eng. Pat. 5153, March 7, 1896.

With the object of printing metallic or other dry powders on textile fabrics, an adhesive medium suitable for carrying the powder is first printed by means of an engraved copper roller, and this is followed by a second roller on which a facsimile of the design engraved on the first roller has been cut in relief. This second roller is furnished with the dry powder from an ordinary colour box, and is adjusted so that the design falls exactly on the top of the previous impression. By using two or more relief rollers, the fabric may be printed with several differently coloured powders.

—R. B. B.

Textile Fabrics and Materials, Impts. in Kiers for Bleaching and similarly Treating. E. Deakin, Bolton. Eng. Pat. 6028, March 18, 1896.

In the bottom of the kier is inserted an inverted hopper-shaped casting, which, together with a perforated grating, forms the false bottom of the kier. Near the bottom of this casting are small openings communicating with the space below the false bottom, and inside the casting is a coil of

steam pipes. Above the casting, and connected with it, is a vertical circular pipe, and inside this and concentric with it, is a smaller tube consisting of a series of short tapering pipes fitting loosely over one another in such a way as to allow of a passage of liquid from the inner pipe to the space between the two pipes. When steam is first admitted and circulation set up, the liquor ascends in the outer vertical pipe and descends through the inner tube into the casting described; when, however, the whole of the liquid reaches the boiling point, it flows over the top of the spurt pipe and is deflected on to the fabric in the kier.—R. B. B.

Dry Materials [more particularly Fibrous Substances], Improved Process for Treating, with Liquid. A. Vogelsang, Dresden, Germany. Eng. Pat. 26,195, Nov. 23, 1896.

A VACUUM is produced in a hermetically closed vessel, in which the dry fibrous materials are placed, and liquid is rapidly conveyed into the vacuum chamber by means of a high-pressure pump, and immediately brought to a pressure of at least 50 atmospheres. The process is stated to be applicable to the dyeing and bleaching of dry, raw, unboiled fibrous material, such as loose cotton, cops, twilled hanks, or meshed fabrics.—R. B. B.

VII.—ACIDS, ALKALIS, AND SALTS.

Sulphuric Acid: Cleaning Transport Tank-Waggons. R. Nörrenberg. Chem. Ind. 1896, **19**, 553.

THE wrought-iron cylindrical vessels, used as tanks (Rhenania Fabrik), are flushed with a good stream of water introduced through the manhole, and this flushing water, with the suspended mud, is drawn off through a tube inserted in the manhole and reaching to the lowest point in the tank, to a partially exhausted reservoir. Drainage taps, which are liable to leakage, are thus dispensed with.

The reservoir should be fitted with a vacuum gauge and a tube to show the level of the contained liquid.

The cleaning of each tank can be done without risk, by two workmen in from half an hour to one hour.—J. T. C.

Permanganic Acid, Reduction of, by Manganese Peroxide. H. N. Morse. Ber. **30**, 48—50.

THE evolution of oxygen, noticed by Meyer and Recklinghausen, in acidified solutions of potassium permanganate which have been employed to absorb hydrogen or carbonic oxide, is due, according to the present author, to the manganese peroxide formed in the oxidation, and now present in the solution. The same evolution is produced by all reducing agents, and also by the addition of a small quantity of precipitated manganese dioxide to acid permanganate solutions. The rapidity of the evolution in this latter case is dependent upon the temperature, the amount of oxide present, and the degree of acidity of the solution.

Investigation of the reaction led to the following conclusions:—

1. Permanganic acid and potassium permanganate are reduced by precipitated manganese dioxide, with liberation of three-fifths of the active oxygen of the permanganic acid.

2. The observed instability of standard potassium permanganate solution is attributable to this cause. Such solutions should be carefully filtered through asbestos, and then stoppered up.

3. Permanganate solutions free from suspended oxide show a high degree of stability in the dark or in diffused daylight; in direct sunlight however, decomposition occurs.

4. The oxide resulting on complete reduction of a neutral potassium permanganate solution contains the whole of the potassium originally present in the salt, and the supernatant solution is therefore neutral.

5. Whether the precipitated peroxide is formed by the gradual decomposition of a neutral permanganate solution or by the addition of manganese sulphate to an acidified solution of permanganic acid, the ratio of oxygen to manganese only remains normal (*i.e.*, 2:1) so long as unreduced permanganate or permanganic acid is present. In the absence of permanganic acid or its salts, the oxide loses oxygen even at ordinary temperatures.

6. In the presence of permanganate or permanganic acid, this lost oxygen is again taken up.

The lower oxides of manganese produced in these reactions appear to form members of a homologous series, and may be represented by the following formulæ:—$MnO.5MnO_2$, $MnO.10MnO_2$, $MnO.15MnO_2$, $MnO.20MnO_2$, &c.

The author has also studied the reaction of lead peroxide on permanganic acid, in presence of nitric acid, and represents it by the equation—

$$2HMnO_4 + 3PbO_2 = H_2O + 2MnO_2 + 3PbO + 3O_2$$

This reaction is complicated by the presence of the manganese peroxide, and is not so simple as that described above, which is represented as follows:—

$$4HMnO_4 + xMnO_2 = 2H_2O + xMnO_2 + 4MnO_2 + 3O_2$$

The reaction with lead peroxide forms a good method for the preparation of oxygen, and possesses the advantage that the gas current can be regulated by suitable heating of the mixture, or by altering the rate at which the permanganate solution is added to the mixture of lead peroxide and nitric acid.—J. T. C.

Corrosion of Cast and Wrought Iron by Fused Caustic Soda under Pressure. Scheurer-Kestner. Bull. Soc. Chim. **15**, [24], 1250—1252.

THE author relates the circumstances attending the explosion of an iron boiler in which caustic soda was being heated to 250° C., at a pressure of 4 atmospheres. Laboratory experiments showed that pressure increases the rapidity of corrosion of cast or wrought iron by fused caustic soda.—G. H. B.

Alkali Sulphates, Action of Hydrochloric Acid Gas on. A. Colson. Comptes rend. 1896, **123**, 1285.

EXPERIMENTS were made in which relatively large quantities of sodium sulphate were subjected to the action of dry HCl in tubes having their open ends dipping into mercury, so that the tension attained after prolonged contact could be determined for different temperatures. Under these conditions, the mercury rose slowly in the tubes, until it attained a stable height, showing, at 10° C., a tension of 2 mm.; at 100°, of 15 mm.; at 120°, of 23·5 mm.; and at 175°, of 77 mm. Doubling the proportion of the gas (the salt being still in large excess), gave practically the same results. Hence it is concluded that the tension of the HCl gas is independent of its mass, this being characteristic of heterogeneous dissociation. It was also found that a somewhat rapid rise of temperature in the experiments increased the pressure far beyond the stable tension for the temperature attained. In one case a rise to 175° in the tube gave a tension exceeding 200 mm., which after a time fell to 78 mm., showing a change of phase in the reactions. From these and other experiments described, the conclusions are drawn, first, that sodium sulphate is attacked cold by dry HCl; and, second, that there are several series of tensions of the gas, corresponding to reactions expressed by—

	Calories.
(1.) $SO_4Na_2 + 2HCl = SO_4H_2 + 2NaCl$	+ 17·2
(2.) $2SO_4Na_2 + 2HCl = 2SO_4NaH + 2NaCl$	+ 33·4
(3.) $SO_4NaH + HCl = SO_4H_2 + NaCl$	+ 10·1

Of these, the second equation, answering to the greatest liberation of heat, represents the more stable effects. Finally, the author concludes that the action of hydrochloric acid gas on sodium sulphate involves a succession, or rather, a superposition of phenomena of heterogeneous dissociation.—E. S.

Iron Salts from Pyrites, Manufacture of [for Sewage Purification]. A. and P. Buisine. Rev. Chim. Indust. 1896, 297.

THE salts of iron, particularly the ferric salts, have of late years increased in importance as precipitants proposed for sewage purification, the ferric salts yielding the best results (see this Journal, 1893, 354 and 1052). For the manufacture of ferric sulphate by this process 100 parts of sulphuric acid at 60° B., in the hot state in which it comes

from the Glover tower, are run into a cast-iron boiler which can be heated by direct heat. To this hot acid, from 40 to 50 parts, according to the desired acidity of the finished product, of very finely ground burnt pyrites are added, and the mixture well agitated. A reaction sets in, which is finished by the application of heat, and results in conversion of 95 per cent. of the burnt pyrites into the ferric salt. The finished product is a dry greyish powder of anhydrous ferric sulphate, slowly soluble in cold, readily soluble in hot water. Ferric chloride in the solid state is obtained by passing hydrochloric acid gas containing very little moisture—*i.e.*, in the state in which it leaves the salt-cake furnace—over burnt pyrites, placed in moderately thick layers in suitable chambers. The action starts in one point and proceeds rapidly through the whole mass until practically the whole of the ferric oxide has been converted. For the preparation of ferrous salts, incompletely burnt pyrites, containing still at least 10 per cent. of sulphur, are treated with sulphuric acid at 60° B. in the manner above described for the production of ferric sulphate. The residual sulphur in the pyrites is eliminated during this treatment in the form of sulphuretted hydrogen, which reduces the ferric sulphate formed at the same time into ferrous sulphate, free sulphur separating out at the same time. The crude product thus obtained is dissolved in boiling water for crystallisation. The insoluble residue consists almost entirely of sulphur. —C. O. W.

Sulphuretted Hydrogen, Absorption of, by Liquid Sulphur. H. Pélabon. Comptes rend. 1897, **124**, 35—37.

The author summarises his results as follows:—

1. Hydrogen begins to combine with sulphur at about 250° C.
2. Between 250° and 350°, combination is not complete.
3. Liquid sulphur, maintained at a temperature above 170° C., in presence of sulphuretted hydrogen, absorbs a considerable quantity of this gas.
4. The quantity of gas absorbed is greater the higher the temperature, pressure remaining constant.
5. In all cases the gas is liberated at the moment of solidification of the sulphur; "the disengagement of the gas is a consequence of the solidification."
6. Pure hydrogen is not absorbed (in the sense of solution) by liquid sulphur.

It is interesting to compare this result with the observation that liquid selenium, heated in the presence of a mixture of hydrogen and seleniuretted hydrogen absorbs a considerable proportion of this latter gas.—J. T. C.

Electrolysis of Salts and Bases in Presence of Ammonia. S. M. Losanitsch and Z. Jovitschitsch. Mitteil. d. königl. Serb. Akad. d. Wiss. (Ber. **29**, 2436.)

See under XI. A., *page* 148.

Boric Acid, Volumetric Determination of. G. Jörgensen. Zeits. f. angew. Chem. 1897, 5.

See under XXIII., *page* 163.

Limestones, Estimation of Iron and Alumina in. Désiré de Paepe. Chem. Zeit. 1896, **20**, 1004.

See under XXIII., *page* 163.

Alkalis and Alkaline Carbonates, Volumetric Estimation of, with Phenolphthaleïn and Methyl Orange as Indicators. F. W. Küster. Zeits. anorg. Chem. 1896, **13**, 127.

See under XXIII., *page* 163.

PATENTS.

Cyanogen Compounds, Impts. in Methods of Separating, from Gas Liquor or other Solutions containing Cyanogen Compounds. H. Bower, Philadelphia, U.S.A. Eng. Pat. 361, Jan. 6, 1897.

See under II., *page* 129.

Chloride Solutions for Disinfecting and like Purposes, An Impt. in the Preparation of Electrolysed. E. Hermite, E. J. Paterson, and C. F. Cooper, London. Eng. Pat. 2197, Jan. 30, 1896.

The electrolysed chloride solution prepared according to specifications 15,384 of 1887 (this Journal, 1888, 764), 8177 of 1889 (1890, 733), 18,370 of 1893 (1894, 170), 22,279 of 1893 (1894, 271), and 6,497 of 1894 (1895, 492), loses strength and deteriorates with time, and it is claimed that this may be prevented by the addition to the chloride solution, either before or after electrolysis, of small proportions of alkali, preferably on account of cheapness, caustic soda or milk of lime.—G. H. R.

Ferrocyanides from Sulphocyanides, Impts. in the Manufacture of. J. T. Conroy, F. Hurter, and J. Brock, Liverpool. Eng. Pat. 3869, Feb. 20, 1896.

A mixture of calcium sulphocyanide and of ferrous chloride solutions is heated in an autoclave under pressure, with iron, preferably reduced iron, in excess of the proportion indicated by the equation—

$$Fe(CNS)_2 + 2Fe = Fe(CN)_2 + 2FeS.$$

When the reaction is completed, the precipitate, consisting of ferrous cyanide and ferrous sulphide, may be digested with caustic soda solution if it be desired to obtain sodium ferrocyanide; but if the potassium salt be wanted, the precipitate is first treated with strong hydrochloric acid to dissolve out the iron sulphide, the hydrogen sulphide evolved being collected. The ferrous cyanide is then washed, and digested with potassium hydroxide solution to obtain potassium ferrocyanide. If the first-named process be used, the ferrous sulphide left after the digestion with soda, is treated with hydrochloric acid to obtain ferrous chloride for application in decomposing calcium sulphocyanide, the hydrogen sulphide given off being collected as before.—E. S.

Zinc Oxide, Impts. in the Manufacture of [*from Zinc Sulphate*]. Dr. W. Hampe and Dr. C. Schnabel, Clausthal, Germany. Eng. Pat. 3906, Feb. 20, 1896.

Anhydrous zinc sulphate and carbon, both finely divided, are mixed in the proportions indicated by the equation—

$$ZnSO_4 + C = ZnO + SO_2 + CO$$

and heated for about two hours at a regulated temperature approximating to 650° C. The sulphurous acid evolved, may be utilised in obtaining sulphuric acid for acting on zinc ores to produce the zinc sulphate required in the process.—E. S.

Nitrites of Soda and Potash, Impts. in the Manufacture of. L. G. Paul, London. Eng. Pat. 4743, March 3, 1896.

To 100 parts of sodium nitrate, melted in an iron pan, 40 parts of caustic soda and 14 parts of sulphur are added in successive portions, with agitation, the temperature being kept too low for the sulphur to inflame. After mixture, the temperature is raised until the melt froths, when the agitation is renewed. The increased temperature is maintained until the melt clears, when it is run off. The same process is used in the reduction of potassium nitrate, equivalent weights of the nitrate and hydroxide being taken, answering to the proportions used in the former case. The nitrite obtained is separated from the sulphate simultaneously formed by fractional crystallisation.—E. S.

White Arsenic, Impts. in the Manufacture of. T. P. Sims and W. Terrill, Swansea. Eng. Pat. 9076, April 30, 1896.

Certain by-products, consisting chiefly of iron arsenide, and known as "slurrie" and "hardhead," are melted and run into a vessel which is fixed; instead of the usual movable vessel known as the "Bessemer converter." This vessel has a tap hole, nozzles for introduction of an air-blast, and it is surrounded by a water-jacket. The charge is melted in an adjacent furnace and then allowed to run into the fixed vessel or converter. The blast of air is introduced just before the molten charge is run in, and is maintained

throughout the operation. When arsenious anhydride (which is collected) ceases to be evolved, the molten contents are run out. The slag, consisting chiefly of ferric oxide, is separated from the metal or speiss, containing iron, copper, silver, &c.—E. S.

Evaporators, Improvements in and relating to the Discharging of Salts and the like from. J. Foster, Pollokshields, Glasgow. Eng. Pat. 928, Jan. 14, 1896.

See under I., *page* 129.

Nitric Acid Regenerator. Dr. J. Walker, Basel, Switzerland, and C. Lehmann, Muskau, Prussia. Eng. Pat. 20,290, Sept. 14, 1896.

The apparatus for regaining nitric acid from its products of reduction is a cylindrical chamber constructed in two sections, the upper section resting on the lower, near the bottom of which is an inlet for the gases. A series of superposed porous earthenware plates, having holes, each vertically over that beneath it, is arranged within the chamber, the gas outlet being immediately below the highest plate. Each hole is occupied by a tube, resting on its flange, and covered by a cap in the form of an inverted cup, provided with a series of notches at the rim, on which it rests, allowing passage for water in one direction and for the gases in the opposite direction. Water enters the chamber through a spray head in the cover. Several modified forms of caps to the depending tubes are described and shown.—E. S.

VIII.—GLASS, POTTERY, ENAMELS.

Crude Kaolin, Saarau (Silesia). H. Seger and E. Cramer. Thonind.-Zeit. **20**, [56], 819.

Saarau kaolin is derived from a deposit of kaolinised granite. Its fusing point approximates to 35° of the Seger cone, and its proximate composition is :—Quartz, 38·05 ; felspar, 0·61 ; clay substance, 61·34 per cent. ; the ultimate constituents being silica, 67·09 ; alumina, 23·77 ; and iron (reckoned as oxide), 0·70 ; with 8·50 per cent. of loss during calcination. The clay—assuming the iron to be present as a mechanical admixture, probably from pyrites—is composed of alumina, 39·8 ; silica, 46·3 ; water, 13·9 per cent. Analyses of the kaolin from the upper workings of the same deposit, made by G. Richters in 1868, showed 75·88 per cent. of total silica, and 17·31 per cent. of alumina, so that the present material is much richer in clay substance.

Most of the quartz is removed by sedimentation washing, the purified kaolin then consisting of silica (combined), 40·89 ; quartz, 7·38 ; alumina, 36·3 : iron oxide, 1·16 ; loss during calcination, 12·57 per cent., which—expressed in percentage terms of burnt kaolin—is equivalent to silica, 55·0 ; alumina, 41·4 per cent.—C. S.

Temperature Determinations. [Pottery Furnaces.] Hecht. Centralbl. f. Glas-Ind. 1896, **11**, 311 and 321.

When employing the Le Chatelier pyrometer the chief points to remember are, first, that the resistance of the wire leading to the galvanometer should not greatly exceed 1 ohm. The instrument may be ordered with sufficient length of wire to reach from the furnace to the manager's office (whereby the progress of the heating can be readily observed), and the resistance of this will be taken into consideration in standardising the apparatus. Secondly, the platinum and platinum-rhodium wire must be of such a length that its temperature at the point of connection with the copper wire leading to the galvanometer is not appreciably higher than that of the room. The wires of the elements, which are insulated from each other by porcelain capillaries, must be protected from the heating gases, and particularly from carbon, by a porcelain tube glazed on the outside. The galvanometer must be firmly fixed on to a table, wall bracket, or other support, so that the perpendicular coincides with the intersection of the fixed lines on the lower ring, and the checking screw must be loosened gradually, otherwise the suspensory threads of the frame may be broken or strained and give rise to inaccuracies. The crank handle on the case is for the adjustment of the pointer against the zero point of the scale after the instrument is in position and before the current is passed.

The instrument enables the manager to control, from a distance, the work of the furnace men, and to see that they are performing their task properly.

In considering the feasibility of replacing the Seger cones by the Le Chatelier pyrometer for pottery work, it should be remembered that time as well as temperature forms an important factor in the production of the pyrochemical effect to be attained in the kiln, and that the Seger cones undergo, in the processes of sintering and fusing, similar changes to those sustained by the ware, and therefore constitute a valuable means of control. The two methods in conjunction, however, afford more complete information respecting the firing temperature of various kinds of ware than has hitherto been obtained.

The author submits a table of the fusing points of the Seger cones from No. 022 to No. 36, calculated from data obtained by testing No. 022 and others up to No. 10, which reduces the figures at which the higher numbers were formerly estimated. It is stated that the Le Chatelier instrument is employed in conjunction with the d'Arsonval galvanometer and the Seger cones at the Royal Porcelain Works at Berlin, Dr. Schott's glass laboratory in Jena, Messrs. Villeroy and Boch's works, and at the Mahlstadt Cement Works, with satisfactory results.—C. S.

Glass, Calculation of the Yield of a Charge of. R. Hohlbaum. Sprechsaal, **29**, [46], 1273—1274.

The shrinkage of the charge is due to the volatilisation of acids, water, and other impurities, alkalis, and (where fluorspar is added as a flux) silicon fluoride, the two first-named being, however, all that have usually to be considered in practice. The actual percentages of loss sustained by the chief substances used in glass-making are : of the sodium sulphate employed, 62 per cent. as SO_3 ; sodium carbonate, 53 per cent. as CO_2 ; potassium carbonate, 45 per cent. as CO_2 ; calcium carbonate, 46 per cent. as CO_2 ; barium carbonate, 25 per cent. as CO_2 ; strontium carbonate, 32 per cent. as CO_2 ; silica, *nil* ; carbon, the whole.

Assuming the following quantities to be taken for a charge : sand, 120 kilos. ; sodium sulphate, 60 ; calcium carbonate, 39 ; coke, 3 kilos. ; total, 222 kilos. ; then the loss will be : on the sodium sulphate, 37·2 ; on the calcium carbonate, 17·49 ; coke, 3 kilos. ; total, 58·14 kilos. ; so that the yield of glass will be 222 − 58 = 163·86 kilos. (see this Journal, 1897, 43).—C. S.

Plate Glass, Rough-Etching. Sprechsaal, **29**, [49], 1349.

Before treating with hydrofluoric acid, the glass is rough-etched, by immersion in an acid bath, if the sheets are small. In the case of large sheets an edging of wax—prepared by incorporating ½ kilo. each of shoemakers' wax and beeswax, and 50 to 80 grms. of oil of turpentine—is carefully placed all round the part of the sheet to be treated, forming a trough to contain the etching fluid. The background and all portions of the sheet, except those to be occupied by the design, are coated with a preparation which must be sufficiently fluid to apply with the brush (or by lithography), and cover well without spreading, dry throughout in 6 or 8 hours, and resist the action of acids. The following mixtures answer well :—

——	I. Thin.	II. Medium.	III. Thick.
	Grms.	Grms.	Grms.
Rectified oil of turpentine ..	1,000	1,000	1,200
Gum dammar............	300	..	..
Venice turpentine..........	300	200	200
Powdered Syrian asphalt ...	600	500	500
Rosin......................	..	150	200
Burgundy pitch	..	150	..
Tallow	..	100	100
Beeswax....................	..	100	..

They are prepared by heating the materials over a sand-bath, taking precautions against boiling over. The product

is stored in closed vessels to prevent the evaporation of turpentine, which latter substance is also used for thinning down the (warmed) mass when required.

To apply the preparation, long, fine brushes of various sizes are used, the design, drawn on paper, being laid under the small sheets of glass, or indicated by laying the paper on the larger sized sheets, and dusting coloured powder through perforations made along the outline.—C. S.

Clear Glass. [*Materials.*] A. M. Sprechsaal, **29**, [48], 1326.

In the production of clear glass, purity of materials, cleanliness, and care in the mixing and fusing of the constituents are of prime importance. Decoloration is effected either by the complementary colouring action of manganese and other oxides, or by oxidising agents, such as nitrate of soda and arsenic. The use of coloured cullet should, as far as possible, be avoided, and regularity as to purity of the materials should be secured.

The following proportions are recommended for the several grades of glass specified:—

(*a.*) *Lead Crystal for Sonorous Drinking Glasses and Finest Cut Ware.*—Quartzsand, 100; potash (90 per cent.), 33·33; minium (free from iron or copper), 50; pulverised limestone, marble, calcspar, or Danish chalk, 8·66; nitre, 3·66; manganese oxide, 0·10 to 0·15; crystal cullet, 100 parts.

(*b.*) *Crystal for Fine Table-Ware.*—Quartzsand, 100; soda (90 per cent.), 35; minium, 8; nitre, 2; lime (as under *a*), 10; arsenic powder, 0·5; manganese oxide, 0·15 to 0·2; red cobalt oxide, 0·0066; crystal cullet, 100 parts.

(*c.*) *Semi-Crystal for Ordinary Ware.*—Quartzsand, 100; soda (90 per cent.), 33·5; lime (as above), 20; nitre, 2; arsenic powder, 0·5; manganese oxide, 0·2 to 0·25; red cobalt oxide, 0·001; semi-crystal cullet, 100 parts.

(*d.*) *Bohemian Crystal I.*—Quartzsand, 100; best potash, 35; calcined lime, 19 (or marble, &c., 26); nitre, 1·25; green nickel carbonate, 0·007 to 0·009; Bohemian crystal cullet, 100 parts.

(*e.*) *Bohemian Crystal II.*—Quartzsand, 100; best potash, 25; soda (90 per cent.), 5; powdered marble, &c., 28; minium, 3·25; arsenic powder, 0·75; Bohemian crystal cullet II., 100 parts.

(*f.*) *Bohemian Crystal III. Hard Soda-Glass.*—Quartzsand, 100; best potash, 25; soda (90 per cent.), 10; powdered marble, &c., 25; arsenic powder, 1·5; nitre, 0·5; manganese oxide, 0·05; soda crystal cullet, 100 parts.

(*g.*) *Bohemian Crystal IV. Soft Soda-Glass.*—Quartzsand, 100; best potash, 20; soda (90 per cent.), 13·33; powdered marble, &c., 20; arsenic powder, 3; minium, 1; manganese oxide, 0·080 to 0·1; soft soda crystal cullet, 100 parts.

(*h.*) *Crystal Glass for Open Furnace with direct Wood Fire.*—Quartzsand, 100; molasses potash, 28; soda (90 per cent.), 4; powdered marble, &c., 24; nitre, 1; litharge, 2; green nickelous carbonate, 0·008 to 0·01; cullet from similar glass, 100 parts.

(*i.*) *Moulding Crystal.*—Same as *a* and *b*.

(*k.*) *American Moulding Crystal.*—Quartzsand, 100; soda (90 per cent.), 39; minium, 51; nitre, 3; calcined borax, 0·1; nickelous carbonate, 0·008 to 0·01; red cobaltous oxide, 0·0005; cullet from similar glass, about 100 parts.

(*l.*) *Ordinary Moulding Glass for Household Ware.*—Quartzsand, 100; soda (90 per cent.), 42; limestone, &c., 25; nitre, 2; arsenic powder, 0·5; calcined borax, 0·5; nickelous carbonate, 0·009 to 0·012; red cobaltous oxide, 0·0005 to 0·0007; cullet from similar glass, 100 parts.

(*m.*) *Plate Glass.*—Quartzsand, 100; soda (90 per cent.), 33·33; calcspar, &c., 30; nitre, 2; arsenic powder, 0·5; borax, 0·5; nickelous carbonate, 0·009 to 0·012; red cobaltous oxide, 0·0005 to 0·0008; plate glass cullet, 100 parts.—C. S.

Enamels for Iron Plates, &c. Clauss. Berg- u. Hüttenm. Zeit. 1896, **55**, 34.

The enamel consists of 130 parts of felspar, with 125 of borax, 70 of quartz, 25 of soda, 17 of nitre, 10 of fluorspar, 4 of antimony, and 0·5 of cobalt. The large proportion of felspar makes the enamel less fusible, but it adds to its covering power and to its resistance to chemical action.

—W. G. M.

Glazes and Enamels, Part played by Boric Acid in. L. Grenet. Comptes rend. **123**, [21], 891—893.

The coefficient of expansion of a glaze or enamel is sometimes increased by the addition of boric acid, and, on the other hand, is sometimes diminished, although the coefficient of expansion of boric acid itself is greater than that of any glass. The author finds that if boric acid be used in successively increased quantities, the first effect is to lower the coefficient, and afterwards to progressively increase it. This result appears to be connected with the circumstance that if the proportion of bases be large, devitrification occurs; whereas if the proportion of boric acid be large, a separation of nearly pure boric acid takes place.—V. C.

PATENT.

Improved Kiln for "Firing" Colours, Gold, Enamels, and the like, on all Kinds of Pottery and Glass; applicable also to other Purposes, such as Baking Bread and the like. D. Chapman and W. Illingworth, Longton. Eng. Pat. 23,831, Dec. 12, 1895.

The articles are placed on a frame, which revolves so as to carry the articles to be fired into the furnace while withdrawing from the heated chamber, the articles which have been fired. The inventors claim improved construction of kiln and means of securing continuous firing.—V. C.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Slag Bricks, Manufacture of. Berg- u. Hüttenm. Zeit. 1896, **55**, 340.

Blast-furnace slag is too brittle and too sharp-cornered for road-metal; and if the hot slag, whilst still pasty, is worked in a brick press, the moulds are not properly filled, and the resulting slag block becomes brittle, like quickly cooled glass. At the Karl Emil-Hütte in Königinhof (Bohemia), bricks are made from the cinder produced in a coke furnace smelting an oolitic impure clay ironstone. The slag, having the percentage composition of from 25·8—27 SiO_2, 1·5—1·7 FeO, 17·3—19·3 Al_2O_3, 51·5 CaO, 0·4—2·5 MgO, and 1·3—1·8 S, is run from the cinder-notch into water, where it is granulated to a clear grey-coloured sand. It is then mixed in a mill with milk of lime and pressed into bricks, which must be left for eight days to harden before they will bear transport. The bricks should contain about 26·5 SiO_2, 17 Al_2O_3, and 52 CaO per cent., and should bear a pressure of 18 kilos. per sq. cm. (256 lb. per sq. in.), though they are only guaranteed to carry two-thirds of this amount. The whole width of the brick should withstand a load of 4,870 kilos. (nearly 5 tons). Another furnace, producing a slag of the composition 33 SiO_2, 1 FeO, $4\frac{1}{4}$ MnO, $18\frac{2}{3}$ Al_2O_3, 40 CaO, $2\frac{1}{2}$ MgO, and $1\frac{1}{2}$ S per cent., gives less satisfactory bricks, that will not bear transport for three months.

—W. G. M.

Limestones, Estimation of Iron and Alumina in. Désiré de Paepe. Chem. Zeit. 1896, **20**, 1004.

See under XXIII., *page* 163.

PATENTS.

Burning Cement-making Materials, Lime, Chalk, and the like; Impts. in Apparatus for, and the Obtainment therefrom of Carbonic Acid Gas. W. R. Taylor, Rochester. Eng. Pat. 68, Jan. 1, 1896.

The inventor claims the obtaining from the above materials of practically pure carbonic acid gas, and the combination in apparatus therefor of "a drying floor on which wet slurry is partially dried, a bed of retorts in which the remaining moisture and carbonic acid is driven off from the material, and a fusing kiln in which the dried material is burnt or fused."—V. C.

Artificial Stone, A New or Improved Manufacture of, for Building, Decorative, and other Purposes. F. W. Maxwell and W. Beer, Manchester. Eng. Pat. 2614, Feb. 5, 1896.

A MATERIAL produced by admixture of a stone base with oxide of magnesium, and successive treatment with magnesium chloride, oleate soap, and acetate of aluminium.
—V. C.

Veined, Coloured Marble; Improved Process for Producing Artificial M. Bernstein, Berlin. Eng. Pat. 17,281, Aug. 5, 1896.

THE material consists mainly of the waste produced in polishing stones, which is mixed with a binding material, such as a mixture of magnesite and magnesium chloride, and allowed to harden under slight pressure. In order to produce veins, "the colouring matters are previously mixed with the suitable quantity of pulverised white marble and brought into the fundamental mass in this mixed state."
—V. C.

Artificial Stone, Impts. in the Manufacture of. A. Cléry, London. Eng. Pat. 24,212, Oct. 30, 1896.

A PLASTIC material, hardening to the consistency of stone, suitable for application to and protection of stony surfaces. The plastic material is prepared by adding about one part of the following liquid mixture, *viz.*, zinc 35 per cent. and hydrochloric acid 65 per cent., to three parts of a mixture composed of about 30 per cent. of zinc oxide mixed with sandstone, Portland stone, sand and marble, or granite.
—V. C.

Piles and the like, Impts. in, and in Means for Protecting them from Water Insects, and Preserving them generally. H. Gallinowsky, Longbeach, Miss., U.S. Eng. Pat. 25,851, Nov. 17, 1896

A FIBROUS material, such as jute, is soaked with boric acid, magnesium chloride, hydrated magnesium oxide, and sal ammoniac, and wrapped round that portion of the pile which is to be immersed in water, which portion has a diminished cross section, and the whole is then subjected to a creosote bath.—V. C.

X.—METALLURGY.

Calcium Carbide: A New Reducing Agent. H. N. Warren. Chem. News, 1897, **75**, 2.

THE author gives the results of a series of experiments in which calcium carbide was used as a metallurgical reducing agent.

An excess of litharge, when heated to redness in contact with calcium carbide in a clay crucible, was reduced, with formation of metallic lead and calcium oxide, CaO, the reaction being accompanied by vivid incandescence.

On heating a mixture, in which the proportion of carbide exceeded that of the litharge, to various temperatures, alloys of calcium and lead of varying composition were obtained, together with the expulsion of carbon dioxide. The alloys thus formed are all more or less brittle, their melting point ranking below that of pure lead, and are slowly but completely decomposed in contact with aqueous vapour. Stannic oxide, cupric oxide, and also ferric oxide, at correspondingly higher temperatures, were readily reduced, yielding results of no practical value; in the case of the cupric alloys, those samples containing less than 1 per cent. of calcium being rendered cold-short, and breaking under very small strain, whilst iron containing calcium approaches in appearance that of ferro-manganese, being even more brittle and very oxidisable in contact with water. Oxides of manganese, nickel, cobalt, and even chromium, molybdenum, and tungsten were also readily reduced, yielding calcium alloys.—A. S.

Iron Carbide, Pure. E. D. Campbell. Amer. Chem. J. 1896, **18**, 836—847.

CAREFULLY annealed bars of crucible cast steel (1·29 per cent. of C) were suspended from a copper ring in a 4 per cent. solution of hydrochloric acid. The ring was connected to the positive pole of a series of storage cells, a platinum cup being cathode, and an electric current of 1 ampère at 8 volts, passed through for 15—16 hours. Each day the deposit on the strips was brushed off and collected, and the process renewed. From 576 grms. of steel which was dissolved, 55 grms. of carbide was collected, or 9·66 per cent., or about half the total carbon in the steel (assuming the formula of the carbide to be Fe_3C). The carbide obtained consists of minute bright steel-grey scales of sp. gr. 6·944, gradually but completely converted by moist air into carbon and ferric oxide, and completely soluble in moderately strong hydrochloric acid. Analyses of the body gave 6·646 and 6·625 per cent. of carbon; Fe_3C requires 6·67 per cent.

The gases arising from the solution of the carbide in hydrochloric acid were examined. About 150 c.c. of gas was yielded by 0·600 grm. of the carbide. Determinations were made of the olefines (by absorption with bromine), the carbon dioxide formed by explosion, first of the original gas and then of the gas without olefines, and the hydrogen (by absorption with palladious chloride). Calculated on 1 grm. of the carbide, the average result was: volume of gas, 250 c.c.; hydrogen, 210 c.c.; olefines, 9·6 c.c.; total CO_2, 72 c.c.; CO_2 from paraffins, 33 c.c.; CO_2 from olefines (by difference), 39 c.c. The figures show that about 60 per cent. of the total carbon of the carbide appears in the gases, the rest clearly occurring as some hydrocarbon which either condensed or remained in solution. The liquid in the flask smelt strongly of butane, in which form no doubt most of the missing carbon existed. The ratio of the volume of the olefines to that of the CO_2 they produced shows that butylene must have been present in quantity. The author suggests that these iron carbides have the general formula C_nFe_{3n}, being olefines in which H_2 is substituted by —Fe.Fe.Fe—; when these are dissolved, the corresponding olefine is formed, which partly polymerises and partly picks up hydrogen, which is simultaneously formed by the action of the iron group on the acid.
—J. T. D.

Carbide in Steel. F. Mylius, F. Foerster, and G. Schoene. Zeits. anorg. Chem. 1896, **13**, 38.

THE results arrived at by the authors are as follows:—(1.) Ignited steel is a mixture of crystallised iron and crystallised iron carbide. (2.) Iron carbide is a definite compound of the composition CFe_3, and resembles and is analogous to manganese carbide, CMn_3, but is more indifferent to solvents. (3.) Iron carbide, is not altogether insoluble in dilute acids. (4.) Iron carbide dissociates on strong heating into carbon, and iron poorer in carbon, and from this latter iron carbide again separates on slow cooling. (5.) Iron carbide enters into chemical reaction with iron at a bright red heat.—L. T. T.

Gold, Cyanide Process: Comparison of Zinc and Electrical Methods of Precipitation. A von Gernet. Johannesburg Chem. and Metall. Soc., Oct. 1896.

THE most important points for comparison are the relative strengths of solution and consumption of cyanide in the two processes. In regard to the former, the solution of gold depends more upon mechanical conditions than upon strength of solution, as even a 0·05 per cent. solution contains over 88 times the amount of cyanide actually required. A 0·3 per cent. solution is used on the Rand because, although the gold might be dissolved in a weaker liquor, the zinc would fail to precipitate it. Thus, in the zinc process, the strength of solution must be adapted to the precipitant, whilst in the electrolytic process any strength may be used according to the nature of the ore to be treated. The crudeness of the zinc method is one of its prominent faults, great care being needed in a clean-up, and no means being available for checking loss. "Then, taking zinc cyanide bullion as averaging 70 per cent. fine gold, and Siemens-Halske cupelled bullion as 89 per cent., each oz. of fine gold in the latter case is worth 6*d.* more than in the former." The cost of electrical precipitation for a plant capable of working 500 tons per diem may be taken at (per ton):—Filling, 10*d.*; cyanide, 3·4*d.*; lime, 0·5*d.*; white labour, 5*d.*; native labour, 1·9*d.*;

fuel and power, 4*d.*; lead, 1·5*d.*; iron, 0·3*d.*: charge due extra cost plant, 0·5*d.*; store and general charge, 3·2*d.* Total, 2*s.* 6·3*d.* For a 7,000-ton plant, four boxes 4½ ft. × 3 ft. × 30 ft. would be used, containing 3 tons of iron per box.

Treatment of Slimes.—For this purpose the electrical process has no rival. It is now for the first time possible to treat all ore products—free gold, concentrates, tailings, and slimes: and since the last-named may now be treated separately, there is no need to leave as much pulp as possible in the tailings vat, with the risk of rendering the sands slimy and unleachable. Only the leachable portion of the mill-pulp will be retained, and extractions should therefore be higher. Early in 1897, plant, capable of treating over 50,000 tons of slimes monthly, will be at work, and such a plant will soon be a necessary part of every mill. At present every ton of water introduced into the stock cyanide solution in the tailings necessitates the waste of an equal volume of dilute solution to make way for it in the tanks. The gold and cyanide in this solution were formerly lost, but they will now be recovered by applying this solution to the treatment of slimes, for which a very dilute liquor is appropriate. In a slimes plant the pumping and precipitating capacity must be very great, since 7 tons of solution are handled for each ton of slimes treated. The precipitation must be very perfect, since the dilute liquor displaced, bulk for bulk, by the slimes under treatment, must run to waste, and any gold and cyanide left in it is lost. The rate at which the slimes settle depends upon their nature, upon the depth of the vat, upon the ratio of slimes to solution in the mixture, and upon the influence of the agents (such as lime) added to accelerate settling. The treatment of slimes fresh from the mill, causes difficulty owing to the ready solubility of the gold; this happens through some of the gold in the overflowing slimes from a newly charged vat being dissolved by the agency of a little cyanide left in the vat after discharging. Old slimes from slimes dams give trouble owing to the presence of sulphates and free acids from oxidised pyrites, and from decomposed animal and greasy matter from the mine and the mill. These complications are not observed in laboratory trials, but only in actual working. The whole may be summed up by saying that while zinc has in its day done good service, it no longer meets modern requirements, and must be superseded by electricity.—W. G. M.

Silver, Extraction of. [*Treatment of Sulphides with Sulphuric Acid.*] P. Truchot. Revue Chim. Ind. **7**, [84], 380—383.

In the extraction of silver by the Russell process in the United States, a double hyposulphite (thiosulphate) of sodium and copper is employed, and this solution gives much better extractions than hyposulphite of sodium alone. The drawback of the process was the difficulty of treating the sulphide precipitate, which takes the form of a dry powder, difficult to handle, causing loss and being difficult to sample and assay. The following successful method of treatment has been adopted at the Marsac Mine of the Dewey-Walter Refining Co.:—

A charge of about 150 kilos. of the sulphide precipitate and 450 kilos. of sulphuric acid of 66° B. is placed in an iron pot, carefully stirred, and heated to boiling. Sulphur dioxide, mixed with sulphur, is violently disengaged at first. The reaction may be hastened by means of a jet of steam. When the mixture begins to get pasty, about 1,350 kilos. of acid are added in quantities of 45 kilos. at a time. As the boiling proceeds, the precipitation of granular insoluble sulphate of copper necessitates frequent stirring. The pots are not rapidly attacked, nine having been employed during 1894. After about an hour's violent boiling, the operation is stopped and the charge allowed to cool, after which it is led into the dissolving vat, containing water, and boiled by a jet of steam, and filtered. Almost all the copper is contained in the first liquid. The heavy white mud is washed with weakly acid solutions to remove the silver; after washing, it contains about 5 to 19 ozs. of silver and 50 to 100 ozs. of gold per ton, and consists principally of sulphate of lead, and is smelted directly. The solutions are passed through a filter of washed quartz sand, upon which and in the dissolving vat considerable quantities of metallic silver are deposited; about 10,000 ozs. of silver per year accumulate on the filters. The filtered liquors are heated by steam in a vessel containing copper (usually electrolytic cathodes), with a current of air passing over, about 4 to 18 hours being required for the precipitation. After 20,000 ozs. of silver have accumulated in the vat, it is transferred to a special vat and washed with cold water and cold acidulated water till no further trace of copper is found in the washings; this operation takes 15 hours. The silver is dried, pressed into cakes, again dried, and melted under borax as usual. The sulphate of copper is recovered from the liquors by crystallisation.

A foreman and three men are able to work a plant capable of treating over 52,000 kilos. of the sulphides per annum.—H. B.

Cadmium with Silver and Copper Metallic Combinations, A New Mode of Alloys of. J. B. Senderens. Bull. Soc. Chim. **15**, [24], 1241—1247.

A bar of cadmium, placed in a solution of sulphate or acetate of silver, precipitates the latter metal, but if the action be allowed to go on for a long time, the loss in weight of the bar of cadmium is much more than equivalent to the silver precipitated, and as this excess was not present in the solution, it was found in the metallic deposit, which had been slowly transformed into a very oxidisable alloy of the formula $AgCd_3$. Similar experiments were tried with solutions of chloride, sulphate and acetate of copper. Pulverulent alloys were formed, but containing less cadmium.—G. H. B.

Steel, Alloys of, with Molybdenum and Chromium. Oesterr. Zeits. Berg- u. Hüttenw. 1896, **54**, 590.

The Schneider Company at Creusot have lately experimented with regard to the production of molybdenum-chrome steel for armour plates. They find that the addition of 0·2—0·3 per cent. of chromium and a like amount of molybdenum to steel greatly increases its resistance to penetration, without rendering it brittle. This steel may be made in the converter or in the crucible, with the aid of ferro-chrome and of pure metallic or of alloyed molybdenum. It may be forged, rolled, hardened, or cemented.—W. G. M.

Tellurium Production at the Royal Hungarian Lead and Silver Smelting Works at Schemnitz. J. Farbaky. Zeits. f. angew. Chem. 1897, 11.

It has long been known that the presence of tellurium gives rise, by volatilisation, to a loss of gold and silver during their cupellation. Experiments made with the object of removing tellurium from the gold and silver obtained at the Pertuzola works in Italy were published last year by C. and F. Heberlein (Berg- und Hüttenm. Zeit.), and C. Whitehead (this Journal, 1896, 202) has worked out a process for separating tellurium from the silver and copper contained in the silver mud formed in copper electrolysis. Applications of tellurium in medicine, porcelain painting, and thermo-electricity are heard of from time to time, but it would appear that so far no satisfactory results have been obtained, and that tellurium is at present only valuable on account of its rarity. Partly in the hope that by making tellurium more accessible some practical applications of it will be discovered, it has been decided to manufacture it at Schemnitz from the Siebenbürgen ore used there. The ores used contain calcium and magnesium carbonates, silica, lead, copper, and zinc compounds, and gold, silver, and tellurium. The ore is first dissolved, as far as possible, in concentrated sulphuric acid; the mass is then extracted with hot dilute hydrochloric acid, and filtered. The residue contains all the gold and silver, the filtrate the tellurium. The latter is precipitated by saturating the liquid containing it with sulphur dioxide, which is employed in the liquefied form. The precipitated tellurium is washed, dried, and finally fused and cast in sticks or granulated. It contains from 72 to 85 per cent. of tellurium, the principal impurities being copper and tellurium dioxide, the latter formed by the oxidation of the finely divided metal during the drying process.—T. E.

Cobalt and Nickel, Sulphides of. G. Chesneau. Comptes rend. 1896, **123**, 1068—1071.

Alkaline polysulphides saturated with sulphur, give with cobalt salts a black persulphide, CO_2S_7, insoluble in alkali monosulphides, but soluble in sulphides saturated with sulphur. Nickel salts give a corresponding persulphide, which, however, is soluble in alkali monosulphides, but only with difficulty in polysulphides.—J. T. D.

Tungsten, The Reduction of, from Wolfram by Carbon in the Electric Furnace. E. Defacqz. Comptes rend. 1896, **123**, 1288.

Wolfram from Zinnwald, in Bohemia, was used in the experiments. It contained about 72 per cent. of tungstic acid, and about 16 per cent. of manganous oxide, the remainder consisting of ferrous oxide, lime, and silica in variable proportions. A mixture of the mineral with sugar carbon, subjected to the action of an electric arc of 1,000 amperes and 60 volts for 12 minutes, gave a melted metallic mass, easily detachable from the scoria. Analysis of the tungsten thus obtained showed it to be entirely free from manganese and calcium, the only impurities being about 5 per cent. of carbon, rather more than 2 per cent. of iron, and 0·5 per cent. of silicon. The author concludes that the direct treatment of native minerals in the electric furnace is capable of yielding metals sufficiently pure for industrial application.—E. S.

Iron, Corrosion of, by Water containing Carbonic Acid. P. Petit. Comptes rend. 1896, **123**, 1278.

See under I., *page* 128.

Corrosion of Cast and Wrought Iron by Fused Caustic Soda under Pressure. Scheurer-Kestner. Bull. Soc. Chim. **15**, [24], 1250.

See under VII., *page* 141.

Platinum, The Action of Phosphorus on. A. Granger. Comptes rend. 1896, **123**, 1284.

See under XXIV., *page* 169.

Copper, Electrolytic Analysis for Technical Purposes; Estimation of Arsenic, Antimony, Sulphur, and Foreign Metals. A. Hollard. Comptes rend. 1896, **123**, [24], 1063.

See under XXIII., *page* 164.

Slag Bricks, Manufacture of. Berg- u. Hüttenmänn. Zeit. 1896, **55**, 340.

See under IX., *page* 144.

PATENTS.

White Arsenic, Impts. in the Manufacture of. T. P. Sims and W. Terrill, Swansea. Eng. Pat. 9076, April 30, 1896.

See under VII., *page* 142.

Galvanising, An Impt. in. [*Tinning.*] R. Heathfield and W. S. Rawson, London. Eng. Pat. 24,703, Dec. 24, 1895.

Any imperfection in the coating of galvanised iron or steel articles may be remedied by cleansing them from all traces of oxide, and then immersing in a bath composed of about 2 parts of chloride of tin (fused), and 10 parts pyrophosphate of soda to 600 parts of water. To prevent too rapid deposition of tin, the bath is preferably started cold and then gradually raised to boiling point An immersion of about 30 minutes suffices to give a coating of tin to the bare iron surfaces as well as to the galvanised portions, but if the articles are to receive a second coating in the zinc-depositing vat, a 10 minutes' immersion in the tin bath will suffice.—G. H. R.

Treatment of Iron and Steel, and Production of Alloys [producing Chromated Iron or Steel]; Impts. in the. E. Placet, Paris. Eng. Pat. 203, Jan. 3, 1896.

Chromium is deposited upon bars or tubes of iron or steel by electrolysis, and the bars or tubes are afterwards either melted, hammered, rolled, drawn, or subjected to cementation.—R. S.

Auriferous Antimony and other Ores, Smelting; Impts. in Plant and Process for. C. C. Longridge, Leigh, and G. T. Holloway, London. Eng. Pat. 303, Jan. 4, 1896.

At least four furnaces are arranged in cascade form, one above the other; in the first, antimony sulphide ores are liquated; the second is for reducing, the third for refining, and the fourth for "starring." For the extraction of gold from antimony ores an extra furnace for mixing is introduced between the first and second. The refining and starring may both be conducted in the third furnace if desired. The last three furnaces are separately fired with gas, and the products of combustion may be used to fire the mixing and liquating furnaces. In applying the process, the auriferous antimony ore is liquated, and is run through a launder into the mixing furnace, in which it is agitated with about half its weight of fused metallic antimony until all the gold has alloyed with the metal. The agitation is effected by rotating the mixing furnace, which is made in the form of a horizontal cylinder mounted on rollers. The sulphide is then run on to the other furnaces, but the antimony is used to treat three other batches of ore, or until it assays about 100 ozs. to the ton. In other cases the ore is treated with so much metallic iron that the reduced antimony is in quantity sufficient to dissolve the gold from the whole charge of ore, and the mixture is rotated as before. In treating other gold-bearing substances, the material is first mixed with fused antimony sulphide, which dissolves the gold, and then the auriferous sulphide is agitated with antimony as above.—W. G. M.

Gold, Amalgamation of, and of like Metals; Improved Apparatus for effecting. E. L. Oppermann, London. Eng. Pat. 1469, Jan. 21, 1896.

Mercury vapour generated in a closed retort is injected, with steam at a suitable pressure, into shallow covered channels, through which the ore is passing from the stamps, or from a rotating mixer into which steam may be injected if desired. From the closed channel, the ore passes over amalgamated plates to a rotating separator provided with electrodes. The amalgamated plates themselves, and rods supported at a short distance above them, also form electrodes to the liquid passing between them. Ammonia or other gases may be injected in addition to the steam and mercury vapour. The apparatus is especially designed to carry out the process specified in Eng. Pat. 17,020 of 1895 (this Journal, 1896, 120).—W. G. M.

Gold, Impts. in Apparatus for Use in the Extraction of, from Auriferous Material by the Aid of Chemical Solvents. J. Y. Johnson, London. From J. J. Deeble, Bendigo, Victoria. Eng. Pat. 23,954, Oct. 27, 1896.

The vat for extraction is fitted with rotating horizontal agitators mounted on a vertical shaft, so that their direction of rotation may be reversed, or their position may be altered, by raising or lowering the whole shaft by means of a rack and pinion or other contrivance, operated from a platform above the vat. A sluice-valve is arranged for drawing off the solution from any level in the vat, and a waste-discharge valve is provided at the lowest level for the removal of the extracted ore. The inner surface of the vat has projecting pieces, which should produce eddies during the rotation.

—W. G. M.

Steel, Impts. in the Production of, and in the Refining of Iron Ore. H. H. Lake, London. From H. Schweitzer, New York, and E. Lungwitz, Brooklyn. Eng. Pat. Aug. 18, 1896.

Calcium carbide is introduced into the Bessemer converter, either at first or (preferably) some time after the commencement of the blow. The objects of the addition are:

the removal of phosphorus and sulphur from the metal by by means of lime *in statu nascendi* acting upon the anhydrides of the metalloids, aided by the thorough mixture of the calcium carbide and iron; the production of a higher temperature in the converter by the formation of CaO from CaC_2; and the carburisation of the steel by the carbon of the carbide.—W. G. M.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(*A.*)—ELECTRO-CHEMISTRY.

Analytical Chemistry. The Importance in, of the Ionic Dissociation Theory of Arrhenius. F. W. Küster. Zeits. f. Elektrochem. **3**, (1896), 233—236 and 257—260.

ANALYTICAL chemistry can boast of a large and increasing literature: but considered purely as a science it cannot be rated very highly. Until recently, indeed, we were not in possession of such genuine views and theories as were necessary for its development on broad scientific lines. But recently the position has improved. The theory of solutions and the law of mass-action have enabled us to investigate the conditions of equilibrium in solutions. We have a better knowledge of the nature of salt solutions and of reactions between salts, and it is now possible to develop a theory of analytical reactions. With rare exceptions the analytical chemist has to deal with aqueous solutions of salts (including, of course, acids and alkalis). These differ entirely in their physical behaviour and properties from other solutions. Arrhenius has explained this by the hypothesis that on solution in water, salts are split up more or less completely into electrically charged atoms or groups of atoms. This also explains why such solutions differ from all others in being relatively good conductors of electricity. Arrhenius' theory affords an explanation of many chemical as well as physical phenomena. Consider, for example, the remarkable fact that there are no specific reactions for salts in aqueous solution. The reactions exhibited by common salt are not peculiar to it alone. Part of them are shared by KCl, $BaCl_2$, $FeCl_2$, and other chlorides; others are exhibited by other salts of sodium. Compare with this the fact that solutions of substances like sugar, naphthalene, azo-benzene, &c., which are not salts, *do* exhibit specific reactions. The difference in behaviour becomes intelligible if we assume with Arrhenius that when common salt is dissolved in water the solution contains little or no salt, but only the products of its dissociation—the ions Na and Cl. Suppose that we have in analytical chemistry to deal with 50 cations and 50 anions. These would give us 2,500 simple salts. If these behaved as salts in their solutions they would exhibit 2,500 different reactions. Arrhenius' theory explains why we have actually to deal with only the reactions of 50 cations and 50 anions, or 100 altogether.

Again, consider the reactions of the compounds—

I. $SO_4\left\langle\begin{matrix}Na\\Na\end{matrix}\right.;\ SO_4\left\langle\begin{matrix}K\\K\end{matrix}\right.;\ SO_4=Cu;\ SO_4\left\langle\begin{matrix}NH_4\\K\end{matrix}\right.$

$$SO_4=Fe-SO_4-Fe=SO_4,\ \&c.$$

and—

II. $SO_4\left\langle\begin{matrix}K\\CH_3\end{matrix}\right.;\quad SO_4\left\langle\begin{matrix}Na\\C_2H_5\end{matrix}\right.;\quad SO_4\left\langle\begin{matrix}NH_4\\C_6H_5\end{matrix}\right.$

$SO_4\left\langle\begin{matrix}CH_3\\CH_3\end{matrix}\right.;\quad SO_4\left\langle\begin{matrix}CH_3\\C_2H_5\end{matrix}\right.;\quad SO_4\left\langle\begin{matrix}CH_3\\C_6H_5\end{matrix}\right.$

Those of the first group exhibit certain definite reactions, of which that with barium chloride is specially characteristic. Those of the second group are derived from sulphuric acid in the same manner (by replacement of hydrogen), but they do not exhibit any common reaction. On Arrhenius' theory this difference in behaviour offers no difficulty. When the compounds of the first group suffer electrolytic dissociation they produce different cations; but they all produce the same anion, SO_4'', and hence all exhibit the reactions of this anion. The compounds of the second group behave quite differently when dissolved in water. The last three are non-conductors; in solution they do not split up into ions. The first three behave as conductors; but the constituents which are attracted towards the anode are not the ions SO_4'', but the monovalent groups $CH_3—SO_4'$, $C_2H_5—SO_4'$, and $C_6H_5—SO_4'$. No single one of the six derivatives produces the sulphuric acid anion SO_4''; hence we cannot expect any one of them to exhibit the reactions of this ion. Furthermore, as all six produce either different anions, or none at all, they naturally have no common reaction.

We must refer to the original paper for the explanation according to Arrhenius' theory of other phenomena and reactions, among which the following may be mentioned:—(1.) The precipitation (or non-precipitation) of metals in the presence of certain salts; *e.g.*, Why are silver salts not precipitated by sulphuretted hydrogen when excess of potassium cyanide is present? (2.) The classification of acids and bases as "strong" and "weak." (3.) The influence of the presence of salts in solutions of the corresponding acids. Here the precipitation of iron from solutions of its acetate is discussed, and the effects of adding acetic acid and sodium acetate are explained. (4.) How the delicacy of methyl orange as an indicator for a particular acid is affected by the presence of a neutral salt of this acid.
—D. E. J.

Electrolysis of Salts and Bases in Presence of Ammonia. S. M. Losanitsch and Z. Jovitschitsch. Mitteil. d. königl. Serb. Akad. d. Wiss. (Ber. **29**, 2436—2438.)

WHEN alkali salts are electrolysed in presence of ammonia so that the cation sets free hydrogen, and the anion (by reacting on the ammonia) nitrogen, the amount of nitrogen liberated is relatively too small. The authors have examined the causes of this irregularity, using graphite anodes and platinum cathodes. Electrolysis of NaCl with NH_4OH gave too little nitrogen by 10 per cent. After the electrolysis, hypochlorite was found to be present, and also chloride of nitrogen. The authors believe that the hypochlorite is formed directly, as when chlorine-water reacts on ammonia. When the hypochlorite has attained a certain concentration it forms with the ammonia, chloride of nitrogen; this again reacts upon the ammonia, and nitrogen is set free. The amount of undecomposed hypochlorite depends upon the concentration and temperature, increasing as the temperature falls, and the solution becomes weaker in NH_3. At a certain concentration and temperature, the amount of nitrogen evolved should be strictly equivalent. (This was verified by experiment. Solution, $NaCl:NH_3 = 9:1$. Temperature, 100°.)

When KBr, KI, and KF are electrolysed in presence of ammonia, hypobromite, hypoiodite, and iodide of nitrogen are found to be formed. No hypofluorite or bromide or fluoride of nitrogen could be detected. Hence the non-equivalence in the electrolysis of KF must be due to the fluorine acting upon the electrode. When KOH and K_2SO_4 are electrolysed, part of the oxygen developed is set free as such, and the rest goes to form nitrites and to set free nitrogen. Other examples of a similar action of nascent oxygen are given.—D. E. J.

PATENTS.

Electrolytic Apparatus, Impts. in. [*Rotating Depositing Barrel.*] R. Heathfield and W. S. Rawson, London. Eng. Pat. 25,002, Dec. 31, 1895.

IN lieu of wood, the staves of barrels described in Eng. Pat. 5537, 1894 (this Journal, 1895, 756), are made of Portland cement, strengthened with an iron framework consisting of two parallel bars, and cross bars, between which bolts are passed to fix the staves to the ends of the barrel. The conductors are tubes or angle-bars sunk in the cement, so as to present only a small part of their surface on the inside of the barrel, and their ends are flattened and soldered to metal sockets moulded into the cement, and covered with a rubber sleeve, over which the sleeve of the lead-wire can

be drawn, so as to form a water-tight joint. Slots to admit the electrolyte are provided in the staves, and are covered with wire gauze on the outside, and formed with one side sloping and the other abrupt, so that the articles to be galvanised may not fall on the gauze. The barrel may be divided into compartments by inwardly-projecting rings, and the conductors divided into sections connected by plugs, so that they may be used independently. Where it is undesirable to cover the anode with a louvre, a cut-out is provided in case of a short circuit. This consists of an electro-magnet which, while the normal current is passing, attracts a lever having at its other end two prongs. On a short circuit occurring, these dip into two mercury cups, close the external circuit, and cut out the barrel. By means of a similar lever, the circuit through the barrel is closed automatically when the latter is lowered into the bath.
—G. H. R.

Chloride Solutions for Disinfecting and like Purposes, An Impt. in the Preparation of Electrolysed. E. Hermite, E. J. Paterson, and C. F. Cooper, London. Eng. Pat. 2197, Jan. 30, 1896.

See under VII., *page* 142.

Filaments for Electric Incandescent Lamps, Impts. relating to Treating or Flashing. [*Organic Borates.*] J. H. Douglas-Willan and F. E. W. Bowen, London. Eng. Pat. 3213, Feb. 12, 1896.

The filaments are given a coating of carbon and boric anhydride by flashing them in an atmosphere of certain organic compounds containing both boron and oxygen, such as the borate of the alkalis, to which from 1 to 2 per cent. of ethyl iodide is preferably added, to lower the vaporising point.—G. H. R.

Waterproofing, A New or Improved Process of Electrically Treating Various Materials [*Silk, Cotton, or other Fabrics*] *for the Purpose of.* C. M. White, London. From The Electro-Waterproofing and Dye Fixing Company, New York, U.S.A. Eng. Pat. 8323, April 21, 1896.

The silk, cotton, or other fabric to be waterproofed is moistened with water from the ordinary town supply, and subjected to electrolysis between an oxidisable anode and any suitable cathode, which are made to press on one another in any convenient way. The anodes are preferably composed of aluminium or tin, antimony and bismuth being unsuitable for the purpose. The electrodes may be of any shape, provided they bear accurately on the fabric throughout its surface, and the electrolysis must not be continued long enough to cause the reduction of the oxide first formed on the material, or the goods will not be waterproofed. The time required may vary between 1 and 30 seconds; and to render the process less delicate it is advantageous to cover the negative roller or plate with cotton-cloth, in order to absorb the hydrogen evolved.—G. H. R.

(*B.*)—ELECTRO-METALLURGY.

Gold from Cyanide Solutions, Electrolytic Precipitation of. S. Creasdale. Eng. and Mining J. 1896, **62**, [24], 557—558.

The author, after a large number of experiments conducted on a commercial scale, came to the conclusion that the most advantageous method for the recovery of gold from cyanide solutions was electrolytic precipitation, using amalgamated zinc plates for the positive electrodes and sheet lead for the negative electrodes. Zinc was chosen on account of its efficacy in precipitating gold from cyanide solutions, and its price not placing it beyond a commercial possibility. The strength of current used was 0·04 to 0·05 ampère per square foot of depositing surface. The voltage was not determined, but was very low. The flow of solution through the precipitating tanks was such that each square foot of depositing surface received the gold from ½ lb. of solution in 30 seconds. In three experiments the gold extraction was respectively 95·50, 99·56, and 99·06 per cent., and experience proved that these results could be obtained day after day without difficulty. With "lean" solutions, the flow of solution through the tanks was doubled, and an extraction of 93·18 per cent. was obtained. The zinc plates at the end of the experiments showed little or no wear. The destruction or loss of cyanide in the experiments referred to was as follows:—(1) 8·78 per cent.; (2) 2·60 per cent.; (3) and (4) none. The cyanide solution as it came from the precipitating tanks was remarkably active in dissolving gold, due no doubt to the presence of nascent oxygen, which makes it especially valuable for treating the next lot of ore in the leaching vats. This increased solvent action was fully as great as that produced by the addition of sodium peroxide, &c. to ordinary cyanide solutions, whilst the destruction of cyanide was apparently not so great.—A. S.

Gold Milling, Electricity in. H. M. Chance. Proc. of the Eng. Club of Philadelphia, 1896, **13**, [3], 214—218.

Methods by which it has been proposed to use electricity in the extraction of gold are classified into—

1. *Electro-Magnetic*, in which a magnet is used to separate iron and other magnetic materials from particles of gold. Such methods have never been of practical importance.

2. *Electro-Solvent*, in which the ore, crushed or whole, is immersed in some solvent of gold (chlorine, potassium cyanide), through which an electric current passes. No process based on this idea seems to be actually at work.

3. *Electro-Amalgamating*, in which a current of electricity is passed from some part of the crushing apparatus through the water to the amalgamating plates or mercury pots, with the idea of aiding the amalgamation. This seems to be more or less effectual in keeping the amalgamating surfaces bright and preventing "sickening"; in one instance where such a process was applied to tailings, a considerable saving of gold was effected.

4. *Electro-Precipitating*, in which the gold, having been got into solution by chemical means, is deposited electrolytically. In the Siemens-Halske process, largely used in South Africa, the anodes are cast-iron plates and the cathodes sheet lead, arranged as baffle plates in a trough through which the solution slowly flows. When sufficiently coated the cathodes are melted and the gold cupelled. With 8,000 sq. ft. of cathode surface, 100 tons of solution can be dealt with in 24 hours.

5. *Electro-Inductional*, in which high-frequency alternating currents are employed, with the idea of causing electric attractions between the particles of gold in ores which contain the metal in grains. No application of this on the large scale appears to have yet been made.

6. *Electro-Smelting.*—This does not appear to have been put in practice to any extent, no doubt because it is more expensive than the ordinary smelting processes.

That more successful applications of electricity to gold extraction have not been made, is probably due to the fact that electricians who have worked in this field have not had a sufficiently intimate acquaintance with the conditions of gold-milling practice.—J. T. D.

Uranium, The Electrical Preparation of. H. Moissan. Ann. de Chim. et de Phys., Oct. 1896.

The author obtained uranium by heating in the electric furnace a mixture of the oxide with sugar carbon. The oxide was prepared by heating nitrate of uranium. In 12 mins. a button of uranium weighing 200 to 220 grms. was obtained. The proportion of carbon in the cast metal varied according as the oxide or the carbon predominated in the mixture.

It was found that under the following conditions, if the heating were carefully carried out, the metal could be prepared containing little or no carbon. About 500 grms. of a mixture of 500 parts of oxide of uranium with 40 parts of sugar carbon was placed in a carbon crucible and heated in the electric furnace with a current of 800 ampères at 45 volts. A fused ingot weighing 350 grms. was obtained. If the mixture be heated too long, the metal carbonises very easily, and eventually a crystallised carbide, C_3U_2, is formed. To prevent the action of nitrogen, the experiments are best made in a carbon tube closed at one end.

The author also obtained metallic uranium by electrolysis of the fused double chloride of uranium and sodium. The salt, which is kept fused by the calorific action of the current, was placed in a cylindrical porcelain jar, covered by a plate of glazed porcelain, through which passed the two pure carbon electrodes and a glass tube bent at right angles. Through this latter a current of perfectly dry hydrogen, devoid of nitrogen, was led above the fused salt. After completely cooling, the contents of the jar were taken up by ice-cold water and then directly washed in alcohol, since uranium in a fine state of division decomposes water at the ordinary temperature. By using an iron electrode, alloys of uranium and iron can be obtained. These are white as silver, can be easily filed, and have a very fine grain.

Uranium, when fairly pure, is absolutely white; when yellow, nitrogen is probably present. Uranium can be filed easily and will not scratch glass. It carbonises slightly when heated in carbon dust, and can be tempered. When free from iron it is non-magnetic. Uranium is much more volatile than iron in the electric furnace, and can be distilled, small metallic spheres, free from carbon, being obtained on condensing the vapour.—A. S.

PATENTS.

Gold, Silver, and other Precious Metals, Electro-depositing, and subsequently Stripping the same from the Cathodes. [*Molten Lead Stripping Bath.*] E. Andreoli, London. Eng. Pat. 23,459, Dec. 7, 1895.

The anodes are formed of peroxidised lead, and the cathodes of iron, or any other convenient metal which will stand the temperature of molten lead or other molten metal or alloy which will absorb the gold. The tank is divided by impervious partitions into compartments in which the electrodes are placed. The electrolyte flows between them in a zig-zag course, and passes out at the upper end. When a sufficient deposit has been obtained on the cathodes they are dipped into a bath of molten lead, covered with a layer of oil, where the gold is removed; and they are then wiped, cleaned, and replaced in the electrolyte.—G. H. R.

Metal Sheets or Strips by Electro-Deposition, Impts. in or relating to the Manufacture of. [*Continuous Deposition.*] K. Klič, Lancaster. Eng. Pat. 26,281, Nov. 20, 1896.

The deposition is effected on a roller, which may be plain, embossed, or perforated, and which is nearly submerged in the bath in which it revolves. The roller is smeared with a film of tallow, so that the deposit may not adhere, and curved anodes are employed. When the deposit is sufficiently thick, it is cut transversely at the level of the electrolyte, and stripped back sufficiently to admit of its being attached to a band, which passes and re-passes through the electrolyte, over and under rollers, and between upright anodes, which deposit metal on both sides of the strip which forms the cathode. The other end of the band is attached to a reel, on to which the metal sheet is wound, and as it leaves the bath it is cleansed from the solution by a jet of hot water. The process is continuous, as fresh material is deposited on to the roller as fast as the sheet is wound off, and strips of any desired length can be produced. —G. H. R.

XII.—FATS, OILS, AND SOAP.

Beeswax and Stearine, Bleaching of. S. Ramboe. Chem. Zeit. 1896, **20**, 1004—1005.

Beeswax is generally bleached by exposure to sunlight in the form of thin cakes. The process is very slow, and much space is required. A better process is exposure to air artificially enriched with ozone; but although a quicker process than the first, many weeks, or even months, are required. Bleaching by chemical agents is unsatisfactory, as not only the colour, but other qualities of the wax are destroyed by them, and all attempts to restore these qualities have hitherto been unsuccessful.

With the view of shortening, if possible, the natural process of bleaching, the different necessary conditions were studied, with the following results:—As regards the effect of the presence of water, it was found that dry wax takes nearly twice as long to bleach as when 2 to 5 per cent. of water is present. The presence of much moisture in the air, however, retards the bleaching. When wax was kept at a temperature of 0°—5° C., direct sunlight had no effect for a long time, whilst at 35° the bleaching was very rapid. Bright daylight, as compared with direct sunlight, has very little effect. It is most important to expose as large a surface of wax as possible. Crude wax, with which half its weight of bleached wax has been mixed, takes only about half the usual time to bleach.

A very quick method of bleaching by sunlight is to prepare an emulsion (by means of the De Laval's emulsor) in water at 60°—80° C. This is poured into cold water, to which a little oil of turpentine has been added, when the wax solidifies in minute globules, which, under favourable circumstances, may be bleached in three or four days, when about one-third of previously bleached wax has also been added. When the emulsion apparatus is employed, the wax can be chemically bleached in 10 minutes. Slightly alkaline water (at 80°) is used, and the emulsion treated first with sodium hypochlorite, and, after 10 minutes, with hydrochloric acid until acid. It is then again emulsified to remove the acid. The product is free from ash, and white, but is brittle and odourless.

Bleaching with sunlight, after treatment in the emulsion apparatus, is also recommended for stearine. The bleaching is said to be quick, and the product satisfactory. —N. H. J. M.

Oils, The Iodine Absorption of. H. Mastbaum. Zeits. angew. Chem. 1896, **23**, 719—721.

The author disputes the statement made by Ketel and Amtusch that linseed oil obtained from the seeds by different methods has the same average iodine absorption, no matter how the oil has been extracted. If the seeds are pressed, the expressed oil has a higher iodine value than the oil obtained by extracting the linseed with ordinary solvents. In the latter case the whole of the oil is obtained, whilst in the former the more fluid portion, having a higher iodine absorption, is pressed out. Several results given of experiments with olives bear out this statement, the temperature at which the olives were pressed also having an influence on the iodine value of the oil. It is further well known that this value changes according to the age of the oil. As regards the applicability of the iodine figure for detecting adulteration, its value is lessened, more especially in the case of drying oils, as it is difficult to say whether the deficiency in the amount of iodine absorbed is due to adulteration or to the age of the oil itself.—W. P. S.

Wool-Fat, The Composition of. L. Darmstaedter and J. Lifschütz. Ber. **29**, 2890—2900.

Continuing their researches on the composition of wool-fat (this Journal, 1896, 548), the authors have discovered a new acid in that portion of saponified wool-fat which is soluble in cold alcohol. This acid, $C_{16}H_{32}O_3$, lanopalmitic acid, is obtained by first fractionating with calcium chloride, and then partially precipitating the acids obtained, from the insoluble calcium salts with magnesium acetate in alcoholic solution. The portion of magnesium salt insoluble in hot alcohol is filtered off and washed with boiling alcohol. On decomposing the latter, the acid, freed from traces of other acids by means of methyl alcohol, forms a white powder, melting at 87°—88° C., and solidifying at 83°—85° C. to a lustrous crystalline mass. It has the peculiar property of readily forming an emulsion with water.

When the filtrate from the above magnesium salt is allowed to stand for a few hours, a small quantity of the magnesium salt of carnaübic acid separates out. The filtrate from this, when treated with an excess of magnesium acetate, yields corresponding salts of two fatty acids, myristic acid preponderating. In the filtrate from these a further precipitate is obtained consisting of the magnesium salt of an acid of the oleic series.

With the alcohols obtained from wool-fat the authors have made a number of experiments, and have separated them into various groups by the following methods :—30 grms. of the mixed alcohols were dissolved in 1 litre of absolute alcohol, allowed to stand some hours at 15° C., and filtered. The precipitate consists of at least three alcohols, two saturated and one unsaturated. The filtrate gave a saturated alcohol, $C_{24}H_{50}O$, carnaubyl alcohol. Again, the filtrate from this, when treated with warm water, yielded a crystalline residue on cooling, which was found to be impure cholesterol. Finally, the solution, after filtering off the cholesterol, was evaporated. A viscous, brown residue remained, which, after removing unsaponified fat, yielded a small quantity of cholesterol.—W. P. S.

Glycerin, New Method of Estimating. Bordas and De Raczkowski. Comptes rend. 1896, **123**, [24], 1071.

See under XXIII., *page* 167.

"*Sulphur Oils,*" *so-called; Estimation of Free Sulphur in.* G. Morpurgo. Pharm. Post. 1896, **29**, 501.

See under XXIII., *page* 166.

PATENTS.

Cotton-Seed Oil, Crude, the Separation and Purification of Colouring Matter from: Impts. in. E. S. Wilson, Strood, and E. Stewart, London. Eng. Pat. 24,418, Dec. 20, 1895.

The colouring matter extracted by alkalis or alkaline carbonates (with or without the aid of a soluble sulphite) from crude cotton-seed oil, is purified by fractional precipitation of the solution containing the colouring matter by means of magnesium or calcium chloride or other salts. The fatty acids, resins, and other impurities in the solution are thus separated as insoluble soaps. After removing these soaps, the colouring matter is precipitated with dilute sulphuric or other acid. The flocculent precipitate is filtered off and drained, the resulting product being a paste suitable for use in dyeing, printing, &c.—W. P. S.

Oleaginous and Fatty Substances of all kinds, and Hydrocarbons; Impts. in the Treatment of. [*Purification.*] A. E. Morgans, London. Eng. Pat. 24,714, Dec. 24, 1895.

The oil to be purified is run into a vessel containing a quantity of warm water. Air is caused to bubble up through the liquids, thereby aerating and washing the oil. The heavier impurities become detached and sink, whilst the air carries off the volatile bodies. The water used, may be pure, or rendered acid, alkaline, saline, or saccharine.

In the treatment of edible fats and oils, butter milk or milk may be used in the place of water.—W. P. S.

Soap Tablets, Cakes, or Bars with Indelible Colour; Improved Means and Apparatus connected with and for the Marking of. G. D. MacDougald, Wormit, Fifeshire, and The London and Dundee Syndicate, Ltd., London. Eng. Pat. 3329, Feb. 13, 1896.

This process consists of piercing the tablets, bars, or cakes of soap with needles to the required depth, and then placing them in a strong tank fitted with a hermetically closing lid. The tank when so charged is exhausted of air, and liquid colour run in until the soap is covered, whereby the holes in the soap are filled. After drawing off the excess of colouring liquid, the soap is washed with a powerful spray of water.—W. P. S.

Purifying and Deodorising Butter and other Solid Fats and Oils, Impts. in Processes for. [*Agitation with Cold Water.*] J. G. Hargrave and A. Hargrave, Manchester. Eng. Pat. 22,873, Oct. 15, 1896.—W. P. S.

XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

(*B.*)—RESINS, VARNISHES.

PATENTS.

Varnishes or Compositions, Impts. in Air and [Water-proofing. J. L. Leitch, Paris. Eng. Pat. 2568, Feb. 4, 1896.

This is a composition for coating hats and other objects in order to render them waterproof. It consists of: acetone, 300 parts by weight; sulphuric ether and gum lac, of each 50 parts; nitro-cotton, 200 parts; and 100 parts each of acetic ether, camphor, paper (dissolved in sulphuric acid), and mastic.—W. P. S.

Celluloid [*Uninflammable*], *Impts. in or relating to the Manufacture of.* M. E. Asselot (née Martin), Paris. Eng. Pat. 6389, March 23, 1896.

25 grms. of ordinary celluloid are dissolved in 250 grms. of acetone, and mixed with a solution of 50 grms. of magnesium chloride in 150 grms. of alcohol, until a paste is finally obtained containing, say, 20 grms. of the latter solution to every 100 grms. of the former solution. This paste, after being thoroughly mixed and dried, is the non-inflammable celluloid forming the subject of the invention. The material thus modified, is said to be "absolutely non-inflammable."—R. B. P.

(*C.*)—INDIA-RUBBER, &c.

India-Rubber Industry, The. R. Robine. Rev. Chim. Ind. 1896, 307.

The best qualities of india-rubber are still those collected in the districts surrounding the River Amazon. But with the rapidly increasing demand, African and Asiatic brands entered the market. *Parà rubber* maintains its reputation as the highest quality amongst the different brands. Its loss in washing is from 10 to 16 per cent. *Negrohead* loses from 25 to 35 per cent. *Matto Grosso,* which approaches Parà in purity, loses only about 10 per cent. in washing. Pernambuco, Maranham, and Bahia lose from 30 to 40 per cent. *Céara,* a very good brand, loses about 20 per cent. The losses on purifying the African qualities, are generally considerably higher. The Asiatic brands, which at one time used to be of high quality and very plentiful, have of late years deteriorated in quality and suffered a considerable reduction in quantity owing to the reckless methods adopted for their collection. The purification of the crude india-rubber is effected by first treating it in hot water for from 12 to 24 hours, then cutting it into slices, which are subjected to a repeated passage between friction rollers upon which a supply of water is flowing. The honeycombed sheets thus obtained, are dried in a hot-air stove. The dry sheets are then given solidity and homogeneity by treatment in the masticator or between a horizontal pair of steam-heated friction rollers. Continuous rubber sheets may be obtained by three different methods, and are accordingly described as cut sheets, rolled sheets, and spread sheets respectively. India-rubber tubing is produced by pressing the india-rubber through a nozzle, in the centre of which a fixed core is arranged. The tubes so produced, are laid in flat spirals on chalk beds, and are in this state vulcanised. India-rubber threads of square cross-section are manufactured by cutting india-rubber sheets by means of suitable cutting-machines. Threads of circular cross-section are obtained by pressing india-rubber dough through mouth-pieces of the desired cross-section. Moulded articles are manufactured by pressing india-rubber sheets, or dough, into suitable moulds which are heated to the required temperature. India-rubber toy-balls are produced by cutting india-rubber sheets into spherical segments, which are cemented together with india-rubber solution. Before finally closing the body so formed, a small quantity of ammonium carbonate is introduced into it, and the opening closed with a small disc of india-rubber. The

body thu formed is placed in a mould and gently heated, whereupon the dissociating ammonium carbonate distends the ball. India-rubber belting is obtained by coating stout cotton cloth on both sides with india-rubber solution. Several of these fabrics are then united by pressure, and subsequently the whole is covered over with india-rubber sheets. Insulated electric cables are manufactured by india-rubber tape wound round the strands to be insulated. These tapes are held in position by similar strips of cotton cloth lapped on in the opposite direction. The cables are then vulcanised in a closed vulcaniser. The manufacture of pneumatic tyres is carried out by cutting strips of india-rubber sheet of a length corresponding to the circumference of the wheels, but successively decreasing in width. These strips are then, in the order of their widths, cemented one upon the other, so that the central longitudinal axis of the first, forms also the axis of the whole fabric. The whole is then steam-vulcanised. The ends of the band so obtained are then joined together. For the purpose of vulcanisation, from 7 to 15 per cent. of sulphur are generally used, but only from 1 to 2 per cent. combine with the india-rubber. The different vulcanisation processes employed are:—(1.) Vulcanisation in a bath of molten sulphur. (2.) Steam vulcanisation. (3.) Dry-heat vulcanisation. (4.) Cold vulcanisation. (5.) Vapour vulcanisation. The first of these processes is practically obsolete; the second is largely employed for the vulcanisation of india-rubber goods of various descriptions; the third process is extensively used, especially for the vulcanisation of waterproof cloth. The temperature in these processes is 170° C. The fourth of the above processes is chiefly used for the vulcanisation of waterproof cloth, and of thin india-rubber sheets. The process is carried out by treating the articles to be vulcanised with a solution of sulphur monochloride in carbon disulphide or benzene. In the fifth process, the goods to be vulcanised are exposed to the vapour of sulphur monochloride, either by itself or in conjunction with gaseous nitric acid, or nitric, or nitrous oxide.—C. O. W.

XIV.—TANNING, LEATHER, GLUE, SIZE.

"Suède" or Kid Tanning. Der Gerber, **23**, 29.

The superiority of French "Suède" glove leather, or glove kid finished on the flesh, lies in the expert knowledge the producers possess that the skins from one source are, and that those from another source are not, suitable for their purpose. Of the suitable kinds, American goat-skins and some kinds of African lamb-skins are more particularly noteworthy. The skins should be strong and firm in structure, but without the fibre being thin and rough. Young goat-skins approach very near to the desired conditions, but are mostly too small for the purpose. Sheep-skins are too loose in structure.—C. O. W.

A Cheap Chrome Tannage. H. R. Procter. Leather Trades Circular and Review, Jan. 12, 1897.

The process of making a basic chrome liquor for tanning, as described in Eng. Pat. 7732, 1893, this Journal, 1893, 612, taken out by J. D. Gallagher, on behalf of Martin Dennis, is said to be roundabout and expensive. A simpler and more satisfactory method consists in reducing bichromate of potash at once with a calculated quantity of acid only sufficient to form the required basic salt. Glucose is conveniently employed as a reducing agent, as it becomes completely oxidised to water and CO_2, but cane sugar, starch, oxalic acid, hyposulphite (thiosulphate) of soda, and other bodies may be substituted. The liquor is made as follows:—3 lb. of bichromate of potash is dissolved in hot water and half a gallon of strong hydrochloric acid (32° Tw.) is added, then glucose gradually, heating, if necessary, till the yellow liquid changes to a deep clear blue-green. Sulphuric acid ($2\frac{1}{2}$ lb., 168° Tw.) may be used instead of hydrochloric, but makes a somewhat harder and plumper leather. About $1\frac{1}{4}$ lb. of glucose is required, and the operation should be performed in a good-sized vessel. The solution is made up to 2 galls. For tanning, the goods are placed in a liquor containing, at first, 2 per cent. of this strong solution, but gradually strengthened to 6 or 8 per cent. Light goods are kept in motion by a paddle, heavier ones are best suspended on a frame, to which a gentle to and fro motion is given. Thick hides can be fully tanned in a week. The process is complete when the hide shows a uniform blue colour throughout. The liquors may be spent by green goods, as in bark tannage; 5—15 per cent. of salt (reckoned on the tanning liquor) will make the leather softer and somewhat looser; whether salt is used or not the leather is tough and stands the boiling test. The fully-tanned leather is well-washed with warm water, and treated with a $\frac{1}{2}$—$\frac{3}{4}$ per cent. borax bath as in the two-bath process.—J. T. W.

Tannin Extracts, The Manufacture of. Fuchs and Schiff. Chem. Zeit. **20**, 926.

See under XX., *page* 161.

XV.—MANURES, Etc.

Detection and Estimation of Perchlorates in Chili Nitrate of Soda. B. Sjollema. Chem Zeit. **20**, 1002.

See under XXIII., *page* 163.

Iron and Alumina, Estimation of, in Phosphates and Superphosphates. Von Grüber. Zeits. f. angew. Chem. 1896, **11**, 741.

See under XXIII., *page* 164.

Perchlorates in Chili Nitrate of Soda as a cause of Injury to Rye. B. Sjollema. Chem. Zeit. 1896, **20**, 1002—1004.

The injury to rye after the application of commercial nitrate of soda, occasionally observed in Belgium since 1892, is now shown to be due to the presence of perchloric acid, probably in the form of the potassium salt. The effect of the perchlorate is seen some (2—4) weeks after the nitrate containing it is applied. The crop is sometimes quite destroyed and has to be ploughed in; in other cases in which the injury is less, the plants are stunted and the seed small. A number of direct experiments on the effect of perchlorate on germination and on growth were made. The results showed that perchlorate in small quantities retards growth, whilst with 0·5 grm. of potassium perchlorate in 3 kilos. of soil the plants were soon destroyed. The experiments do not, so far, decide what percentage of perchlorate in nitrate of soda is injurious (with an application of 200 kilos. per hectare), but as little as 0·5 per cent. would probably be slightly prejudicial. The samples of Chili nitrate analysed, contained from 0·08 to 6·79 per cent., but 0·94 was the lowest percentage in the samples of nitrate which proved injurious.

The author suggests that the perchlorates found in nitrate of soda may have been formed from chlorides by bacteria, or possibly by chemical action, analogous to the production of iodates and periodates from iodides (Guyard, Ber. 1874, **7**, 1040).—N. H. J. M.

XVI.—SUGAR, STARCH, GUM, Etc.

Vacuum Apparatus, Temperature of Vapour and Sugar Juice in. H. Claassen. Zeits. Zuckerind. Böhm. **21**, 263—265.

The author confirms the facts brought forward by Curin on this subject (this Journal, 1897, 55), but does not agree with his explanation of the difference in temperature observed between the vapour and syrup. The vapour escaping from the liquid is not, as Curin supposes, saturated, but as Regnault and Magnus have shown, superheated, and the lower temperature of the vapour is attributed to the unavoidable cooling effect of the walls of the vessel. Bubbles of steam forming on the heating surface have the temperature corresponding to the pressure at that point, including the column of liquid, but only at the moment of formation. Any higher temperature of the liquid is quickly equalised by circulation and a thermometer inserted at any level merely shows this average temperature, and not that of a particular layer.—L. J. de W.

Aqueous Digestion [Beetroot Pulp]. H. Pellet. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 489–492. This Journal, 1896, 746 and 819.

In reply to the critique of Antuszewiez (*loc. cit.* 1896, 95), Pellet maintains that if the instructions he gave in 1888 and 1889 are followed, with regard to the fineness of the pulp, the concentration, and the quantity of basic lead acetate added, the method of cold aqueous digestion gives results in close agreement with those obtained by the alcoholic method.
—L. J. de W.

Beetroot Juice, Nitrogenous Constituents of. E. O. von Lippmann. Ber. 1896, **29**, 2645—2651.

A somewhat limited number of the nitrogenous constituents of beetroots and beetroot juice is to be found described in current literature. Among these may be mentioned asparagine, glutamine, together with their related acids, betaïne and choline, also leucine, tyrosine, glutaminic acid, pyroglutaminic acid, citrazinic acid, lecithin, and legumin.

In describing lecithin (Ber. 1887, **20**, 3201) the author incidentally stated that other substances containing phosphorus and closely resembling nuclein might be extracted by alkalis from the tissues of beetroots, after the latter had been exhausted with water, alcohol, and ether. Further, the decomposition products of these substances, namely, the xanthine bodies, ultimately find their way into the molasses.

In prosecuting further investigations on the question, the author submitted to examination certain liquors occurring in de-saccharification processes, instead of molasses. The method adopted was as follows:—After removal of the lime, the liquors were neutralised and repeatedly treated in a somewhat dilute solution with small quantities of basic lead acetate, subsequently they were fractionally precipitated alternately (according to Scheibler's directions) with phosphotungstic acid and mercuric nitrate. The substances were liberated from the precipitates, purified by repeated boiling, washing, and treatment with animal charcoal, and finally purified from phosphotungstic acid and mercury salts by fractional crystallisation and precipitation.

In addition to those mentioned above, the following substances were identified: xanthine, guanine, hypoxanthine, adenine, carnine, argenine, guanidine, allantoïne, vernine, and possibly also vicine. Phosphotungstic acid precipitates the xanthine bodies, also argenine, vernine, and guanidine; but the precipitates are soluble in excess of the precipitant. Mercuric nitrate acts similarly, and also precipitates allantoïn, which is not precipitated by mercuric chloride; the latter reagent however, precipitates xanthine bodies and vicine. Ammoniacal silver nitrate, or cuprous chloride solution, is a convenient precipitant for the xanthine bodies (Balke, J. Pr. Chem. [2], **47**, 537); nascent cuprous oxide, that is to say, Fehling's solution together with a reducing sugar, behaves in an analogous manner (Drechsel, Ber. **25**, 2454; Krüger, Zeits. f. Phys. Chem. **18**, 351). The basic mixture is most readily dealt with by means of mercuric chloride; after the filtrate has been freed from mercury and evaporated, xanthine is the first body to crystallise out, then follow guanine, generally mixed with other substances, finally hypoxanthine and adenine. Xanthine is purified by repeatedly dissolving in ammonia and cautiously adding acetic acid, guanine by precipitating a solution in hydrochloric acid with an excess of ammonia. Hypoxanthine is separated from guanine and adenine, either by Schindler's method (Zeit. Phys. Chem. **23**, 432) of the silver compound, or by the picrates (Bruhns, Ber. **14**, 533; Wulff, *ibid.* **17**, 468; Bruhns, *ibid.* **23**, 225). Hypoxanthine remains in solution and can be precipitated by ammoniacal silver nitrate, whereas, to separate guanine and adenine from the recovered picrates, crystallisation of the hydrochlorides as well as the sparing solubility of the former in warm aqueous ammonia, can be taken advantage of (Schindler, *loc. cit.*). Hypoxanthine is separated from the remaining xanthine by treatment with ammoniacal silver nitrate, and by dissolving the precipitate in the least possible quantity of hot concentrated nitric acid: hypoxanthine nitrate crystallises from the solution, whilst xanthine may be obtained from the filtrate by addition of ammoniacal silver nitrate solution. Adenine and hypoxanthine are separated, either by one of the above given methods or by Krüger's method (*loc. cit.*) by means of copper sulphate and sodium thiosulphate, which in the cold precipitate adenine only.

The author gives analyses and full descriptions of the various compounds.

According to Ritthausen (Ber. **29**, 894 and 2108) vicine is a glucoside, yielding on hydrolysis a reducing sugar.
—J. L. B.

Massecuites, A New Method for determining the Density of. J. Curin. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 492—496.

The density of the normal weight made up to 100 c.c. is taken, and the degree Brix corresponding to this density multiplied by the factor: $\frac{100 \text{ c.c.}}{26 \cdot 048}$ gives the degree Brix of the original massecuite. The polarisation may be made on the same solution, after weighing, by adding a carefully measured quantity of basic acetate of lead and correcting for the alteration in volume. A table is given ranging from 86·34 to 98·13 per cent. of solids in the massecuites.
—L. J. de W.

Ammonia in the Manufacture of Beetroot Sugar. L. Jesser. Bull. de l'Assoc. des Chim. de. Sucr. et de Dist. 1896, **14**, 529—532. Deutsche Zuckerind, Aug. 7, 1896.

Following up the formation of ammonia in the course of the manufacture of beetroot sugar, 100 c.c. of juice were taken from each diffuser and distilled with 1 to 20 c.c. of normal caustic soda. The quantity of ammonia obtained was found to increase from diffuser to diffuser, and with the amount of alkali added, leading to the conclusion that the ammonia is not present as a salt, which would diffuse in the earlier stages of the process, but probably exists combined as a nitrogenous organic substance. After defecating at 70° C. for 12 minutes with $2\frac{1}{2}$ per cent. of dry lime, a sample was taken and filtered rapidly, made neutral to phenolphthalein with acetic acid, and tested as before. Although more ammonia was found, the quantity was less influenced by increasing the alkali. The nitrogenous compounds are changed by defecation into compounds more easily decomposed. Saturation juice behaved similarly, the effect of alkali being even less marked. In the further course of manufacture the juice continually loses ammonia, so that the massecuite contains very little. The easily decomposable ammonia disappears, and that decomposed with difficulty remains, without modification, in the syrup. The change in the nitrogenous compounds takes place chiefly during defecation, and at different stations the longer the duration of defecation and the higher the temperature employed, the poorer will the weak juices be in ammonia, and the more will the evaporators be spared.

The disengagement of ammonia is in intimate relation with the loss of alkalinity from the ammonia set free and the alkali neutralised. But it is not possible to determine exactly the loss of alkalinity from the ammonia expelled. The alkalinity of the products of decomposition of glutamine is greater by one equivalent of alkali as indicated both by phenolphthalein and litmus. With asparagine this reaction occurs only with phenolphthalein. Although the decomposition of notable quantities of glutamine is not to be expected during the manufacture, yet the reaction should be taken into account, for an increase of the alkalinity of the juice is actually observable in course of manufacture. From the practical point of view we may conclude, without other considerations, as to the loss of alkalinity, by the loss of ammonia.—L. J. de W.

Animal Charcoal, The Weinrich Process for the Revivification of. G. M. Newhall. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 537.

The char from the filter is washed and then dried in an apparatus which utilises the waste heat from the decarboniser to which the heated and partially dried char is next passed. The decarboniser is a revolving cylinder, 4 ft. in diameter and 20 ft. long, heated externally. The char is heated in presence of air to a temperature lower than that in char kilns. After cooling, it is ready to be used afresh.

According to Newhall this process has given good results in the National Sugar Refinery.

The carbon of a char containing usually 12 per cent. was reduced to 9 per cent. It was tested in comparison with the char not revivified, the syrup employed showing 200 on the colorimetric scale. After filtration the colours were: from non-revivified char, 80, and from revivified char, 27.

The composition of the char was as follows:—

——	Before Decarbonising.	After.
Carbon	12·320	8·900
Carbonate of lime	3·320	3·260
Iron	0·320	0·210
Sulphate of lime	0·904	0·877
Sulphite of lime	0·628	0·494

—L. J. de W.

Fodder, Manufacture of, from Molasses and desiccated Beet Pulp. L. Wüstenhagen. Bull. de l'Assoc. Chim. de Sucr. et Dist. 1896, **14**, 542—543.

The pulp is sprinkled with 6 to 7 per cent. of molasses slightly warm, and brought directly to the drier by an endless screw or an elevator. By using this proportion a product is obtained which is not sticky. It is readily taken by cattle, and has a higher nutritive value than ordinary dried pulp. (Austrian Pat. 445,235.)—L. J. de W.

Sulphite of Lime, Solubility of, in Sugar Liquors. J. Weisberg. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 485—489.

On sulphiting sugar juice in the works, sulphite of lime is formed, which gradually passes into sulphate of lime at the high temperatures employed, and in the final stages of the manufacture little or no sulphite can be detected. Moreover, certain organic non-sugars of the juice may be reduced, also giving rise to a change of sulphites into the corresponding sulphates. Owing to the rapidity of this change, the difficulty of preparing a sulphite free from sulphate, and the greater solubility of sulphate of lime, it is almost impossible to determine exactly the solubility of sulphite of lime in water and sugar liquors. In Wurtz's dictionary we find: "one part of sulphite [of lime] dissolves in 800 parts of water." According to Battut (Bull. Assoc. Chim. **8**, 226—227) a 10 per cent. solution of sugar dissolves 0·0368, and a 30 per cent. solution, 0·0374 per cent. of sulphite of lime. These figures being calculated from the lime present, are too high, the greater portion having passed into solution as sulphate. Eckleben (v. Lippmann, Chemie d. Zuck. 648) found that an 8 per cent. solution of sugar dissolved 0·015674 per cent. Weisberg added freshly prepared sulphite of lime in excess (0·5 grm. to 250 c.c.) to water and sugar solutions at 18° C., agitated for some time, and filtered. The sulphurous acid was titrated with iodine, and calculated to sulphite of lime. Water dissolved 0·0043 per cent, 10 per cent. solution of sugar, 0·00825 per cent., and 30 per cent. solution, 0·00800 per cent. of sulphite of lime. After standing for 24 hours, or after boiling for two hours under an inverted condenser, the 10 per cent. solution showed 0·0066 per cent. and 0·0025 per cent. of sulphite of lime respectively.—L. J. de W.

Starch Formation, Observations on. T. Bokorny. Chem. Zeit. 1896, **20**, 1005—1006.

Laurent's experiments with etiolated potato sprouts showed abundant deposition of starch in presence of cane sugar or dextrose. The author obtained the same results with potatoes, but with *Spirogyra maxima* there was no production of starch from cane-sugar solution, from dextrose, or from xylose. *Spirogyræ* even became free from starch when kept dark for some days in 1 per cent. dextrose (with and without addition of calcium nitrate). Under the influence of light *Spirogyræ* readily produced starch from cane sugar, grape sugar, and glycerol, &c., provided that oxygen is present. In an atmosphere of hydrogen not a trace of starch was formed.—N. H. J. M.

Starch, Hydrolysis of, by Acids; An Analytical Investigation of. G. W. Rolfe and G. Defren. J. Amer. Chem. Soc. 1896, **18**, 869.

See under XXIII., *page* 167.

Sugar Solutions, On the Clarification of, for Analysis. K. C. Neumann. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 524.

See under XXIII., *page* 167.

Cotton Wool, Absence of Pentose Derivatives in. H. Suringar and B. Tollens. Zeits. f. angew. Chem. 1897, 4.

See under XXIV., *page* 168.

XVII.—BREWING, WINES, SPIRITS, Etc.

Hops, The Sulphuring of. J. Behrens. Woch. für Brau. **13**, 917—923.

The sulphuring of hops has no influence whatever on their hygroscopicity, sulphured and unsulphured dry hops taking up an equal quantity of moisture from the air under the same conditions. Very exhaustive experiments on the action of sulphuring on the micro-organisms of hops, carried out with various growths of hops, by inoculation into solutions of ammonium succinate and mineral ash, and also into hop extract, and by plate cultures, showed conclusively that its disinfecting power was very imperfect and unreliable, the cultures of sulphured hops showing bacterial and mould growth in every experiment, even though the exposure to sulphur dioxide, and the amount of this gas absorbed by the hops, considerably exceeded what would obtain in commercial practice. A plate-culture determination of the total number of micro-organisms existing on a sample of hops before and after very excessive sulphuring, gave the following result:—Unsulphured: Total organisms per grm. of hops. 13,637,600, including 422,800 mould spores. Sulphured: 8,056,300 organisms, including 169,200 moulds. The only one of the results attributed to the action of sulphur which has any foundation, is the improvement in colour, which is a doubtful advantage, as only brown and second-rate hops would be affected by it.

Hops absorb sulphurous acid, and the more the longer they are exposed to it. Only a part of the absorbed gas remains unaltered, another part is oxidised to sulphuric acid, and a third enters into combination with alkaline organic compounds existent in the hops.

Endeavours were made to find a substitute for sulphurous acid. Exposure to formalin did not effect disinfection, but treatment with chloroform vapour completely sterilised the hops, when applied in sufficient quantity. It produced at the same time a depreciation in colour, but did not appear to exert any prejudicial effect on the fermentation of the wort which was hopped with the hops so treated.—J. G. W.

The Action of Diastase on Starch. A. R. Ling and J. L. Baker. Proc. Chem. Soc. 1897, [173], 3—4.

The authors show that maltose, when heated with Fehling's solution, under the conditions prescribed by Wein, reduces 1·079 grms. of copper per grm. of sugar. The table of Wein, therefore, gives results which are 4·5 per cent. too low, a result also arrived at by Brown, Morris, and Millar.

They have examined in detail the products of the limited action of diastase on starch at 70°, and have separated maltose and the following unfermentable substances, which were purified to such an extent as to free them from all extraneous matter.

Maltodextrin α, $C_{36}H_{62}O_{31}$, identical with Brown and Morris's maltodextrin, but having the properties $[\alpha]_D = 180$, $R = 32·81$.

Maltodextrin β, $C_{24}H_{42}O_{21}$, identical with Prior's "achroodextrin III," and having the properties $[\alpha]_D = 171·6$ and $R = 43$.

A substance, $C_{12}H_{22}O_{11}$, isomeric with maltose, and obtained from the unfermentable residue of that particular fraction previously called isomaltose by Lintner. It had the constants $[\alpha]_D = 156$ and $R = 62·5$, and may consist of the simple "dextrin," $C_{12}H_{20}O_{10} + H_2O$, the existence

of which, the authors' previous work foreshadowed. Inasmuch as it gave a small amount of crystalline osazone, it perhaps contained maltose.

When the three substances above named are treated with an excess of diastase at 60° for a few hours, the approximate reducing powers of the products are R = 90, 91·5, 91 respectively.

There are now ample data by which to conclude that starch, when hydrolysed by diastase, is converted into a series of maltodextrins of gradually decreasing molecular weight and optical rotatory power, and of increasing reducing power. These appear to have the optical and reducing properties of mixtures of the original starch and maltose.

Beetroot Juice, Difficulties in the Fermentation of. G. Aracheguesne. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 508—516.

Juice from beetroots gathered during the rainy period following the long drought of 1895 refused to ferment. Hydrofluoric acid and formalin produced no improvement, but it appeared that the cause might be the antiseptic action of volatile acids, as acetic or formic acids, or of a fixed acid as oxalic acid, liberated by sulphuric acid. Barbet suggested the existence of a special diastase in unripe beetroots having a saccharogenic action, the effect of which counterbalanced the saccharophagic action of the sucrase or invertin of the yeast.

It was found that juice boiled for a few minutes, after acidifying, fermented normally, as did also juice obtained from the same beets after being stored in silos. The addition of lime to remove oxalic acid had to be abandoned on account of the butyric fermentation which followed the reduction of the acidity.—L. J. de W.

Dextrose, Levulose, and Invert Sugar, The Solution Density and Cupric-Reducing Power of. H. T. Brown, G. H. Morris, and J. H. Millar. Proc. Chem. Soc. 1897, [173], 4—5.

See under XXIII., *page* 166.

Antiseptics, Use of, in Spirit Production and in Yeast Production. F. Rothenbach. Zeits. f. Spiritusind. 1896, **19**, 361.

The author draws the following conclusions from his experiments:—

1. Formaldehyde and hydrofluoric acid appear to be the only specific antiseptics against bacteria (spalt-pilze), but other substances are nevertheless more or less suitable for use.

2. The best results were obtained when hydrochloric acid was used during the growth of the yeast.

3. The morphological and physiological characteristics of the yeast species are modified in different ways by the different antiseptics.

4. With careful choice of conditions, a long accustoming of the yeast to the antiseptic is not necessary, a few cultures generally giving satisfactory results.

5. When fairly good potatoes are used, the most harmful bacteria generally come from the stock-yeast rather than from the green malt. This stock-yeast should then either be changed or be purified by means of formalin.

6. In practice, the best yield of alcohol is obtained with yeast which is free from bacteria.

7. Of the prophylactic or purifying media, formalin is preferable, as hydrofluoric acid is more weakening to the yeast cells.

8. The high yield of alcohol obtained where formalin is employed is probably due to the smallness of the quantity of acid present, and the consequently stronger and more continuous saccharification of the mash.

9. In practice, yeast prepared with hydrochloric acid and formalin should yield at least as good results as the lactic acid yeast, especially in distilleries where high acid production occurs.

10. Whether the hydrochloric or the lactic acid method is being employed, the use of formalin is advisable to keep down the development of bacteria. This addition may be made to the yeast, and in the fermenting vessels.

—L. T. T.

Dried Yeast, Duration of Life of. H. Will. Zeits. ges. Brauw. 1896, **19**, 453.

The author finds that in some cases the duration of life of dried yeast is very great, in some of his experiments even exceeding nine years. As a rule, the wild yeasts are stronger in this respect than the ordinary cultivated forms. Saccharomyces apiculatus and the allied species appear especially durable. The best results are obtained when the yeast is mixed with an indifferent substance which is neither hygroscopic nor dehydrating in properties, and rapidly dried at a temperature which is slowly raised to, but does not exceed, 40 C. The best material to use is sawdust and next charcoal. Gypsum or infusorial earth does not give such good results.—L. T. T.

On the Influence of Strong Light on the Cell Formation of Saccharomyces Cerevisiæ. W. Lohmann. Wochenschr. für Brauerei, 1896, 1342.

The author has investigated the effect of a strong electric light and of sunlight on a single yeast cell growing in wort gelatin, and kept under constant conditions of heat and moisture on a microscopic slide.

It was found that, if the temperature be not too low, both lights hindered the budding of the cell, and appeared to injure it if the exposure to the light was prolonged. If the temperature was low the effect was not so marked.

The effect of strong light on *Mycoderma Cerevisiæ*, *Torula*, and on *Saccharomyces Pastorianus I.*, was also determined, and the latter was found to have a much greater power of resistance to the light than the others.

—A. L. S.

Diastase, Chemical Nature of. T. B. Osborne and G. F. Campbell. J. Amer. Chem. Soc. 1896, **18**, 536—542.

In a previous paper, Osborne (this Journal, 1896, 44) described some attempts to isolate diastase. In the present communication the former results are confirmed, although no more highly active diastase than that before described, has been prepared. The authors now separate the diastase by treating the finely ground malt with 5 per cent. sodium chloride solution, and saturating the filtered extract with ammonium sulphate. The precipitate was dissolved in brine, and the filtered solution saturated with ammonium sulphate, the subsequent method of treatment being practically the same as that previously described *loc. cit.* The result of this experiment shows that little diastase was precipitated by bringing the alcohol content of the malt extract to 50 per cent. by weight, whilst nearly all the diastase was thrown down when the proportion of alcohol in the malt extract was made 60 per cent. Experiments were made to isolate diastase from the malt extract of commerce, which had been concentrated at a low temperature *in vacuo*. The authors, however, failed to obtain a product with a higher diastatic capacity than 300.

Several experiments were then made with the object of determining the influence of conditions, such as the age of the diastase solution, the presence of certain substances—especially sodium chloride, disodium hydrogen phosphate, tri-potassium phosphate, orthophosphoric acid, acetic acid, and citric acid. In some cases the results were definite; thus, citric acid in the minutest quantities was always found to depress or destroy diastatic action; but in the majority of the above substances no uniform results were obtainable from which reliable conclusions could be drawn in regard to the circumstances that ensure a high degree of diastatic capacity.

From their experience in testing various preparations, the authors state that the purer the diastase the more sensitive is it to external conditions, and that the method of testing the purity of the ferment by its maltose-producing power becomes of uncertain value, and perhaps fails to furnish a safe criterion of the purity of the enzyme. That the proteid is not the only factor involved in the amylolytic action of diastase is indicated by the great influence on its activity that often accompanies the addition of various substances to its solution. It is therefore not improbable that in attempting to purify diastase, some substance which favours or is essential to its action is removed, and that the enzyme itself is being dealt with,

which is feeble in its action through the absence or deficiency of some accessory substance. Thus the addition of sodium chloride in many cases increases the diastatic action. Experience tends to show that albumin is an essential factor in diastatic action. In all the preparations made, none from which albumin was absent showed amylolytic power, and those containing the most albumin were the most active. It was always possible to roughly judge of the diastatic power of a preparation by heating a portion of its solution to 65° C. and observing the amount of coagulum formed.

The fact that active diastase was only obtained from solutions, the alcohol content of which was between 50 and 60 per cent., may be regarded as fair evidence that the enzyme is not carried down mechanically with the proteid.
—J. L. B.

Wine from Wine-Press Residues. Spaeth and Thiel. Zeits. angew. Chem. 1896, **23**, 721—727.

After pressing grapes and obtaining a juice which yields a good wine, the residue of the fruit is further treated, in order to procure a secondary fluid which, when fermented, gives a wine of poorer quality. The methods used for this purpose are, the addition of sugar and water to the residues, the addition of very dilute alcohol, or allowing the skins, &c. to ferment. After either treatment the juice is expressed. The poor wine thus obtained is sometimes used for adulterating other wines, and the authors give the results of analyses made by themselves of wines, both good and poor, to see whether this adulteration can be detected. They arrived at the conclusion that the small percentage of phosphoric acid in the above wine is the most important factor for detecting its presence. A high percentage of tannin also points to the presence of wine obtained by the before-mentioned processes, although the fining of the wine with white of egg or gelatin tends to reduce the amount of this constituent. Some white wines, however, contain an abnormal quantity of tannin.—W. P. S.

Wines, Algerian: Lactic Acid contained in. J. A. Muller. Bull. Soc. Chim. 1896, **15**, [23], 1210—1213.

The author gives full analyses of 14 samples of red wines from different districts of Algeria. The chief point to which he draws attention is the abnormal quantity of lactic acid in some of these wines. In seven of the samples, the lactic acid exceeded 2 grms. per litre, and four of these contained from 3·5 to 4·5 grms. According to Gayon and Dubourg, glucose and levulose, under the influence of a special ferment, are broken up into mannite and lactic and acetic acids, the proportions of the two former being as 72·0 to 10·1. In the wines analysed by the author, the ratio of lactic acid to mannite is, however, in nearly every case far in excess of the above, and cannot therefore result from a mannitic fermentation of grape sugar. Although the samples rich in lactic acid appeared to contain the characteristic disease filaments described by Pasteur, they cannot be properly regarded as "*tournés*," as they contained the normal proportions of tartar and glycerin. It is, however, probable that, before attacking the glycerin and the tartar, the ferment decomposes some of the grape sugar still present, with production of lactic acid.—A. K. M.

Wines, Loss of Colour in ("*Casse*"). Laborde. Comptes rend. 1896, **123**, [24], 1074—1075.

The loss of colour constituting the disease known as "la casse," is due to a fungus, *Botrytis cinerea*, which, being one of the family of oxidising diastases, appears to exert an oxidising action on the colouring matter when in contact with air. This fungus can be destroyed by heat, a temperature of 70° C. being sufficient to destroy the oxidising diastatic growth in the wine. This temperature, however, is a much lower one than would suffice for the destruction of the organism in cultures of the same.
—J. T. D.

Colouring of Wines: possibility of mistaking Coal-tar Colours for Caramel. A. J. da Cruz Magalhaës. Comptes rend. **123**, [21], 896—897.

Caramel is frequently employed to give a fictitious appearance of age to white wines. The author finds that certain colour reactions relied upon for the identification of caramel are also afforded by coal-tar colours. Attention is also drawn to the fact that the reactions given by caramel prepared from saccharose are not identical with those of caramel prepared from dextrose.—V. C.

Alcohol, Denaturing. G. Arachequesne. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 516—524.

A majority of the members of a Commission appointed to study questions relating to denatured alcohol and denaturing agents, are of the opinion that the process of denaturing at present in force in France, based on the employment of methyl alcohol containing acetone, affords the greatest security to the Treasury. The author contends that the method is too costly, and that the alcohol can be purified, and at a considerable profit. He also points out that as denaturing is forbidden in the distilleries, a middleman is necessary, and the chances of fraud are increased.

To illustrate the processes of purification, he gives the following as the composition of a denatured alcohol, worth 46·86 francs per 115 litres:—100 litres of 90° alcohol, 15 litres of crude methyl alcohol ("methylene"), 1 grm. of malachite green, and 500 grms. of petroleum spirit ("heavy benzin e"). This mixture would have the following proximate composition:—

——	Litres.	Boiling Point.
		° C.
Anhydrous ethyl alcohol	90·00	78
" methyl "	9·00	66
Water { 10 per cent. of 100 litres	10·00	100
Water { " " 15 "	1·50	
Acetone, 25 per cent. of 15 "	3·75	56·4
Pyroligneous impurities, 5 per cent. of 15 litres	0·75	80 to 95
Petroleum spirit, 500 grms.	..	80 to 100
Malachite green, 1 grm.	..	..

The acetone and methyl alcohol might be separated almost completely by a single rectification, and the colour also, but the process would fail in the presence of the pyroligneous impurities and the petroleum spirit. On adding, however, a little chloroform and a suitable salt solution, so as to reduce the alcoholic strength to 10°, and agitating, the liquid separates into three layers: below, the chloroform and methylic impurities; next, the saline layer of weak alcohol; and at the top an oily layer of spirit. These are separated by decantation and the alcohol recovered by distillation. It contained only 0·5 per cent. of methyl alcohol, and not a trace of acetone. The value of the products from 115 litres is estimated at 296·05 francs, and deducting 90 francs, the present cost of the alcohol, leaves a profit of 206 francs. To the objection that chloroform is costly, he replies that it may be produced by the action of chloride of lime and lime on acetone, taking care to keep the solution cool and dilute. It will thus be possible to destroy every trace of acetone without attacking the ethyl alcohol, which is acted on only at a high temperature.

The problem of purifying alcohol becomes more and more difficult in proportion as aldehyde and fusel oil are present from the nauseous first and last runnings, especially the latter; and it is a grave mistake to have forbidden more than 1 per cent. of essential oils in alcohols for denaturing. The author suggests that all alcohols in the works regarded as unfit for consumption should be denatured when the manufacturer declares they cannot be improved by further rectifications, chemical or physical.
—L. J. de W.

Alcoholic Fermentation without Yeast Cells. E. Buchner. Ber. **30**, 117—124. (Preliminary Communication.)

One thousand grms. of purified pressed yeast is mixed with an equal weight of quartz sand, and 250 grms. of kieselguhr, and well rubbed together until the mass is moist and plastic. The paste is then well mixed with 100 grms. of water, placed in a filter cloth, and subjected to from 400 to 500 atmospheres pressure. About 300 c.c. of extract are obtained.

The pressed cake is again mixed with 100 grms. of water and again pressed, and about 150 c.c. more of extract are obtained. About 500 c.c. of extract can be obtained from 1,000 grms. of yeast, which contain about 300 c.c. of cell-contents. In order to remove a slight turbidity it is shaken up with about 4 grms. of kieselguhr, and filtered through filter paper.

The extract thus obtained forms a clear but opalescent yellow solution, smelling of yeast. Its specific gravity was in one case found to be 1·0416. On boiling, a copious coagulation takes place, and the whole becomes solid. The coagulation begins at 35°—40°, and is preceded by an evolution of carbon dioxide, with which the solution is saturated. This solution contained over 10 per cent. of dry substance. In a previously prepared solution there was 6·7 per cent. of dry substance, 1·15 per cent. of ash, 3·7 per cent. of albuminoid.

The remarkable property of this yeast extract is that it is capable of causing alcoholic fermentation of sugar solutions. This was found to take place with solutions of cane sugar, dextrose, levulose, and maltose; but not with mannitol or lactose solutions, these two latter not being fermentable by beer yeast.

Mixtures of the active extract and sugar kept in an ice cupboard for several days became turbid, but no yeast cells could be detected by microscopical examination, and the turbidity appeared to be due to flocks of albumin. If the mixture be saturated with chloroform the fermentation still goes on, although this appears to cause an earlier slight precipitation of albuminoid. Neither does filtration through a sterilised Berkfeldt kieselguhr filter destroy the fermenting power.

A parchment paper dialyser full of the yeast extract was hung in a 37 per cent. solution of cane sugar; after some hours the surface of the paper was covered with numerous small bubbles of gas, and also there was a lively evolution of gas in the interior, caused by the sugar solution diffusing in.

The activity of the yeast extract does not last long; it had vanished after being kept five days in an ice cupboard, but lasted for two weeks when mixed with cane-sugar solution.

Heated to 40°—50° the extract soon loses its fermenting power. If the extract be precipitated by strong alcohol and the precipitate dried over sulphuric acid, it is found to have lost its fermenting power.

The author proposes the name *Zymase* for this ferment.

—A. L. S.

Brandy, Composition of. X. Roques. J. Pharm. Chim. 1897, **5**, 55.

The author having analysed a number of samples of Charente brandy which he believes may be considered pure, has tabulated the results as follows:—

	Cognac Types. 1893.	1892.	1888.	1883.	1873.	Cognac Types. 1893.	1892.	1888.	1883.	1873.
Alcohol content at 15°	68°	68°	60°	57°	52°	65°	63°	61°	60°	59°
Dry extract per litre	4·20	0·48	3·64	2·20	2·04	0·44	0·64	1·12	1·12	1·80
Non-volatile acids (calculated as acetic acid per litre)	..	..	..	..	..	..	..	..	..	..
Per hectolitre of alcohol of 100°: Acids	8·91	8·94	32·25	36·91	57·94	48·00	68·57	78·68	92·00	138·30
Per hectolitre of alcohol of 100°: Aldehydes	7·88	7·37	21·65	26·80	38·07	15·69	18·46	24·78	28·28	40·05
Per hectolitre of alcohol of 100°: Ethers	127·19	107·00	115·35	100·35	137·44	182·15	198·34	174·39	159·66	120·20
Per hectolitre of alcohol of 100°: Furfurol	1·31	1·01	1·23	1·16	1·57	2·30	2·71	2·65	2·51	2·53
Per hectolitre of alcohol of 100°: Higher alcohols	198·83	162·85	162·05	205·54	285·07	180·00	174·34	229·50	271·41	304·59
Total (per hectolitre of 100° alcohol), or impurity coefficient.	344·15	287·17	332·53	370·76	520·09	428·14	462·42	510·00	553·86	605·67
Total of ethers + higher alcohols	362·02	269·85	277·40	305·89	422·51	362·15	372·68	403·89	431·07	424·79
Ratio $\frac{\text{Higher alcohols}}{\text{Ethers.}}$	1·5	1·5	1·4	2·0	2·1	0·98	0·9	1·3	1·7	2·5
Coefficient of oxidation	4·9	5·6	16·2	17·2	18·4	14·8	18·8	20·2	21·6	29·4

	Champagne. 1895.	Borderlais. 1895.	Petite Champagne. 1895.	Fin Bois. 1893.	Champagne. 1875.	Borderlais. 1858.
Alcohol content at 15°	69·3°	66·9°	67·9°	65·3°	52·4°	50·85°
Dry extract per litre	0·16	0·05	0·25	0·16	2·08	2·56
Non-volatile acids (calculated as acetic acid per litre)	0·048	0·024	0·024	0·024	0·252	0·348
Per hectolitre of alcohol of 100°: Acids	36·36	30·49	33·59	40·43	141·98	265·31
Per hectolitre of alcohol of 100°: Aldehydes	9·26	13·08	14·81	9·75	35·86	49·99
Per hectolitre of alcohol of 100°: Ethers	160·13	116·26	121·39	130·55	154·50	190·36
Per hectolitre of alcohol of 100°: Furfurol	2·10	1·73	2·74	1·76	1·59	1·26
Per hectolitre of alcohol of 100°: Higher alcohols	124·72	143·23	123·72	165·31	140·42	186·94
Total (per hectolitre of 100° alcohol), or impurity coefficient.	332·57	304·79	296·25	347·80	474·35	633·86
Total of ethers + higher alcohols	284·85	259·49	245·11	295·86	294·92	377·30
Ratio $\frac{\text{Higher alcohols}}{\text{ethers.}}$	0·8	1·2	1·0	1·3	0·9	1·0
Coefficient of oxidation	10·7	14·2	16·3	14·1	37·5	40·2

The following observations are made:—

1. There is considerable difference between the maximum and minimum impurity coefficients; this difference is less if the sum of higher alcohols and ethers be taken, as recommended by Lusson, the proportion of acid being very variable.

2. This total of the higher alcohols and ethers is one of the least variable values as a basis of comparison for brandies; the number, however, increases considerably with the age of the sample. In ageing there is produced a concentration of these two impurities.

3. The ratio $\frac{\text{Higher alcohols}}{\text{Ethers.}}$ is in most cases nearly unity but increases with age.

4. The coefficient of oxidation, *i.e.*, the ratio of aldehydes + acids to total impurities, furnishes to some extent a measure of the age of a brandy, but the coefficient varies very considerably in brandies of the same age but of different sources. The last portions of the distillate being very acid, the acid content will be small or large according to the extent to which distillation is continued in the manufacture.

5. The numbers which express the total coefficient of impurities and the sum of higher alcohols + alcohols give the greatest information as to the nature of a brandy. The former is about 400 and the latter 300.—A. C. W.

Maltose and Soluble Starch, On the Specific Rotation of. H. T. Brown, G. H. Morris, and J. H. Millar. Proc. Chem. Soc. 1896, [172], 242—243.

See under XXIII., *page* 166.

Yeast, Consumption of [Organic] Acids by. J. Schukow. Centralbl. Bakteriol. 1896, **2**, 601.

See under XXIV., *page* 169.

Starch Hydrolysis by Diastase, On the Experimental Methods employed in the Examination of the Products of. H. T. Brown, G. H. Morris, and J. H. Millar. Proc. Chem. Soc. 1896, [172], 241.

See under XXIII., *page* 165.

Starch Hydrolysis by Diastase, On the Relation of the Specific Rotatory and Cupric Reducing Powers of the Products of. H. T. Brown, G. H. Morris, and J. H. Millar. Proc. Chem. Soc. 1896, [172], 243—244.

See under XXIII., *page* 166.

Invert Sugar, The Determination of. A. Leys. J. Pharm. Chim. 1896, **4**, 488.

See under XXIII., *page* 167.

XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

(*A.*)—FOODS.

Coal-Tar Dyestuffs [for Colouring Edible Substances, &c.]; The Behaviour of, towards the Process of Digestion. H. A. Weber. J. Amer. Chem. Soc. 1896, **18**, 1092—1096.

The effect on digestive ferments was studied of four coal-tar colouring matters known to be employed for the purpose of colouring confectionery, *viz.*, Oroline Yellow ("Fast Yellow"), Saffoline (Acridine Red), Magenta, and Methyl Orange. The experiments were made with pepsin and pancreatin, blood fibrin being digested with the ferment and hydrochloric acid, and parallel tests made with and without addition of the colouring matter. It was found that Acridine Red, Magenta, and Methyl Orange do not interfere with the action of pepsin, but considerably retard that of the pancreatic ferment, whilst in the case of Fast Yellow the exact reverse takes place. Since all these colours retard the action of one or other of the digestive ferments, they are evidently unsuited for the purpose of colouring articles of food and drink.—R. B. B.

Albumins, Putrefaction of. O. Emmerling. Ber. **29**, 2721.

See under VI., *page* 140.

(*B.*)—SANITATION; WATER PURIFICATION.

Arsenical Wallpapers, Cause of the Poisonous Action of. O. Emmerling. Ber. **29**, 2728.

The author tests the accuracy of the statement that arsenic hydride is evolved by the action of micro-organisms on arsenical wallpapers.

Bouillon cultures of bacillus prodigiosus, violaceus, subtilis, anthracis, fluorescens liquefaciens, proteus vulgaris, several sarcinæ, and micrococci, were treated with 0·5 per cent. of arsenious acid and a slow current of air passed through the flask and then through several tubes containing silver nitrate solution. No arsine could be found. Moulds were next tried (Penicillium glaucum, mucor mucedo, and corymbifer, Aspergillus glaucus and fumigatus). These grew on bread paste containing 0·2 per cent. of arsenious acid, and again not a trace of arsine was detected.

Ordinary yeast also ferments arsenical grape sugar solution, without producing arsenic hydride.

The experiments were varied by exposing strips of filter-paper painted with a mixture of starch paste and Schweinfurt green to the air. Numerous colonies of micro-organisms of all kinds developed, but no arsine was detected. It appears thus that the poisonous effects of arsenical wallpapers must be ascribed to dust.—T. E.

Waterpipes, Galvanised Wrought Iron [Uninjurious]. Illustr. Zeit. für Blechind. **25**, [48], 1568.

According to reports from a number of American water companies, the objections raised on sanitary grounds against the use of galvanised wrought-iron pipes have no foundation; and the researches of Ruoff have shown that the amount of zinc dissolved by water during an exposure of 8 months is so minute (0·0024 grm. per litre) that it may be regarded as not injurious to health.—C. S.

Sulphuric Acid, Cleaning Transport Tank Waggons. [Avoiding Risks.] R. Nörrenberg. Chem. Ind. 1896, **19**, 553.

See under VII., *page* 141.

Phosphoric Acid in Potable Waters, Determination of. C. Lepierre. Bull. Soc. Chim. 1896, **15**, [23], 1213.

See under XXIII., *page* 163.

Iron Salts, Manufacture of, from Pyrites. A. and P. Buisine. Rev. Chim. Indust. 1896, 297.

See under VII., *page* 141.

PATENTS.

Purification, Softening, or Hardening of Water for Domestic, Manufacturing, or other Purposes; Apparatus and Process for the. J. H. Williams, Waterford. Eng. Pat. 821, Jan. 13, 1896.

The apparatus comprises a series of tanks or compartments (generally three) arranged at different levels, the water being delivered from each tank to the next lower of the series through a floating hinged tube adapted to retain the sediment, and the lowest tank being provided with a filter, aërating pipe, and suitable inlet and outlet taps and stop cocks where required. In the uppermost or "depositing" tank the water is treated with calcium carbonate, calcium sulphate, alum, potassium or calcium permanganate, or other suitable precipitant; or it may be subjected to the action of an electric current, or a stream of oxygen, in any or all of the tanks. In the intermediate or "neutralising" tank, the water is further treated with softening or precipitating agents, such as borax, sodium or potassium carbonate, &c. In the lowest or "storage" tank, the water is (when necessary) aërated and filtered.—R. A.

Sewage Precipitation Tanks, Impts. in. H. Walker, Nottingham. Eng. Pat. 9602, May 6, 1896.

The tank is formed with a sloping bottom, on to which the sewage is delivered through a suitable inlet pipe. The precipitated matter or sludge gradually slides down the sloping bottom, and collects at the deeper side of the tank. The sludge is discharged through a vertical pipe having a horizontal branch outlet, the outlet being fitted to the vertical pipe below the level of the sewage in the tank. The discharge pipes are formed with suitable plug-closed apertures, through which a rod can be inserted for cleaning or clearing purposes. The overflow channel is constructed round the top of the tank.—R. A.

XIX.—PAPER, PASTEBOARD, Etc.

Kaolin, Calcium Carbonate in. [Injurious Effects, and Remedy.] Papier-Zeit. 1896, **21**, 3138.

Difficulty in sizing some papers has been traced to the kaolin, used for weighting, containing calcium carbonate. Experiments carried out in consequence, showed that when the calcium carbonate in the kaolin exceeded 0·3 per cent., more or less difficulty in the sizing ensued. Of course, the actual limit depended considerably on the kind and thickness of paper and the amount of kaolin used for weighting, &c. The difficulty can be overcome by increasing the quantity of size or by converting the carbonate in the kaolin into sulphate. Where circumstances render the rejection of kaolin found to contain calcium carbonate inconvenient, it is advisable

to convert the carbonate into sulphate. This is best effected by adding to the pulp of kaolin in the vats sulphuric acid of 66° B., to the extent of 1·1 per cent. of the weight of the kaolin for every 1·0 per cent. of calcium carbonate found to be present in the kaolin. The reaction takes place rapidly, the addition of the acid causing increase of temperature of the liquid, and so facilitating the escape of the carbonic anhydride liberated. The mixture should be left for about 10 minutes for the carbonic anhydride to escape, as otherwise the evolution of the bubbles of this gas in the pulp vessels causes inconvenience. As 100 parts of calcium carbonate form 172 parts of gypsum (already sometimes used for weighting under the name of "annaline"), the weight of the kaolin is increased by the treatment, and it may sometimes be found cheaper to treat a local low-priced kaolin containing calcium carbonate than reject it in favour of a higher-priced kaolin from a distance.
—L. T. T.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

Eu-Quinine. C. v. Noorden. Separatabdr. aus Centralbl. inn. Med. 1896, No. 48; Chem. Zeit. **20**, [104], 315.

Zimmer and Co., of Frankfort, have produced a derivative of quinine which, it is alleged, has the characteristic medicinal properties of quinine itself, whilst entirely lacking the bitter taste, &c. characteristic of the drug. It is, moreover, said to possess certain other properties peculiar to quinine, only in a less degree. The new substance, which is termed euquinine, is the ethyl carbonic acid ester of quinine, and has the formula $CO(OC_2H_5)OC_{20}H_{23}N_2O$. It is produced by the action of ethyl chlorocarbonate on quinine. It forms delicate white needles, melting at 95° C., slightly soluble in water, but dissolving readily in alcohol, ether, and chloroform. The solution shows a basic reaction with litmus paper, and forms, with acids, salts which crystallise well. The solution in sulphuric or nitric acid exhibits a strong blue fluorescence, as quinine does. Precipitates are formed with the usual alkaloid reagents; and it gives the characteristic green colour with ammonia after the addition of chlorine water, but not the Herapathite reaction. Of the salts, the hydrochloride is easily soluble in water, the sulphate with difficulty, and the tannate with very great difficulty.—W. G. M.

Codeïne and Morphine, A Solvent [Anisol] which permits of the Separation of. L. Fouquet. J. Pharm. Chim. 1897, **5**, 49.

Morphine was found by Hugounenq to be soluble in hot anisol, but insoluble in cold; codeïne dissolves in the cold liquid. The following are the quantities of the two alkaloids dissolved by 100 parts of this solvent by weight:—

Temperature.	Morphine.	Codeïne.
° C.		
9	Insoluble.	7·80
16	..	15·28
100	0·95	161·00
250	4·80	..

The anisol used was very pure. It boiled at 152° C., and had a sp. gr. of 0·991. Morphine may be separated from codeïne by trituration with anisol at 15° C. The solubility of codeïne in anisol is increased after crystallisation from that solvent.—A. C. W.

Essential Oils. Duyk. J. Pharm. Chim. 1896, **4**, 491.

A continuation of articles abstracted in this Journal, 1896, 739 and 919.

Bergamot Oil.—This essence, obtained by expression from the peel of the fruit of *citrus bergamia*, is usually more or less green in colour; its density is 0·833 at 15° C., its specific rotation a_D = + 9° to + 12·5° at 15° C.; it is soluble in twice its volume of 80 per cent. alcohol. Oils of orange and lemon are easily detected by the polarimeter after decolorisation with animal charcoal, oils of lemon and turpentine by their reaction with magenta decolorised by sulphurous acid (see below). The best method for the detection of adulteration is the determination of linalyl esters, of which the oil contains 25—40 per cent. The oil also contains free linalol, limonene, dipentene, a camphor (bergaptene), and compounds of the coumaric series. (Compare Schimmel and Co.: this Journal, 1896, 925.)

Essence of Néroli is obtained by the steam distillation of the flowers of *citrus bigaradia*, 1 kilo. of which affords 0·5 to 1 grm. of the oil. This essence is almost entirely composed of limonene and its isomers; these hold in solution the stearoptene auradine, to which the odour is in great part due. *Néroline* is the methyl ester of β-naphthol; in alcoholic solution it has the perfume of essence of néroli. The essence is often mixed with essence of petit grain, orange, bergamot, &c. The pure oil has the density 0·881—0·887 at 15° C.; mixed with an equal volume of alcohol a violet fluorescence is to be observed at the surface of the liquid. Essences of néroli, orange, cedrat, bergamot, and petit grain give no coloration with magenta decolorised by sulphurous acid; essence of lemon affords a strong bluish-violet and essence of turpentine a rose coloration. To detect adulterations of the former oils by the latter, triturate a few drops with pure calcium phosphate, treat with distilled water, and filter in a tube; if no coloration be given by the magenta reagent, the oil contains no essence of turpentine or lemon. It is necessary in these experiments to well cork the tube to exclude oxidising action of the air.

To detect essence of petit grain in néroli, add carbon bisulphide in small quantities; the pure oil becomes turbid in contact with the solvent and then gives a clear solution as the quantity of solvent increases. On the contrary, essence of petit grain gives a clear solution with a small quantity, but with a large quantity of carbon bisulphide an opaque whitish liquid.

Essence of Cajeput is contained in all parts of *melaleuca*, a tree growing in Indian countries. It has a camphor-like odour, a burning taste, and the density 0·889—0·918 at 15° C. It contains cineol, $C_{10}H_{18}O$, a hydrocarbon not yet studied, and traces of esters. To obtain cineol (eucalyptol) from essence of cajeput or eucalyptus, treat with a very concentrated solution of phosphoric acid, subject the compact mass to strong pressure, wash with ether, add warm water, decant, wash, and dry the supernatant cineol.

Eucalyptus Oil.—The essential oil of *E. globulus* is the most highly prized. This species, growing especially in Algeria, contains 1—3 per cent. of the oil, which consists essentially of cineol (50 to 70 per cent.), and contains also dipentene and a pinene of boiling point 158°, which forms a crystalline dextro-rotatory hydrochloride. The essential oil of the Australian variety, *E. amygdalina*, contains little cineol but much eucalyptene, $C_{10}H_{16}$, which does not form a solid hydrochloride. To distinguish the two essences, wet the surface of a tube with the oil and expose to the action of bromine vapour; the essential oil of E. *globulus* produces crystals, but other essential oils give nothing similar. Essences of eucalyptus and cajeput have a varying action on polarised light; eucalyptol, which is sometimes substituted for them, is inactive. Cineol and the essential oils in which it is found give a violent reaction with iodine; this property permits of the detection of turpentine.

Mint Oils.—There are many varieties known in commerce, derived from the following plants:—*M. piperita* (peppermint), *M. arvensis piperasceus* (Japanese mint), *M. viridis*, *M. crispa*, *M. pulegium*. In general they are characterised by the presence of menthol in the free state, and as esters of acetic, butyric, and isovaleric acids. In addition, the ketone menthone, the hydrocarbons menthene, pinene, phellandrene, cadinene, acids, &c., are found.

The English (Mitcham) peppermint oil is the most esteemed and the most often adulterated. The density varies from 0·840 to 0·975; it is completely soluble in rectified spirit, incompletely in carbon bisulphide; 70 per cent. alcohol dissolves one-fifth of its volume of essence of best quality, and leaves almost undissolved the American oil or essence of turpentine. Glacial acetic acid enables a recent essence to be distinguished: five drops of essence of

peppermint and 1 c.c. of acid give a green coloration, which appears red by reflected light; old essences do not produce this phenomenon.

The Zeiss butyrorefractometer gives valuable indications as to the purity of peppermint oils. In daylight the Mitcham essence gives a narrow band hardly coloured blue at the edges, occupying the divisions 50—53°; the essential oils of *M. crispa* and *pulegium*, on the contrary, give broad bands in which blue and violet predominate. With sodium light the following indices were found:—

Mitcham oil	52
„ (old)	57
French I.	57·58
Saxon	54
Japanese (menthol extracted)	75·5
French II.	53
„ III.	52·25
American	59·7
„ (old)	62
M. crispa	94·5
M. pulegium	88·5

There is good reason to suspect any essence the index of which on this scale does not fall between 50° and 60° at 25° C., and the line of demarcation of which is not sharp.

Mint oils are levo-rotatory, for English oil $\alpha_{[D]} = -34° 29'$; French, $-14° 3'$; Japanese, $-105°$ to $-106°$; they begin to boil at 203°—209° under ordinary pressure. Deflagration is produced by the addition of powdered iodine; if this is not the case, the presence of essence of *M. crispa*, or oil of turpentine, is to be suspected.

Strongly cooled, the stearoptene menthol is separated. In the Mitcham oil this reaches 53 per cent., in the Japanese 72 per cent.; the greater part of the stearoptene is usually extracted from the latter oil. In a suspected oil the menthol should be determined: first saponify the esters by boiling with normal alcoholic soda solution; titrate the excess of alkali. Boil with acetic anhydride and determine the esters formed as before. Treat the oily residue with sodium. Menthone is reduced to menthol, and can then be determined. Essence of camphor is detected by the polarimeter, its rotatory power + 34° being opposite in direction to that of oil of peppermint.—A. C. W.

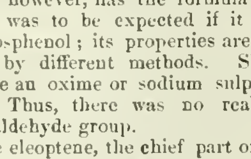
Essential Oil of Buchu Leaves, Composition of. Kondakoff. J. Prakt. Chem. 1896, **54**, 433.

The stearoptene of this oil, diosphenol, $C_{10}H_{16}O_2$, has been shown by Flückiger and Shimoyana to be an aldehyde-phenol; it gives a green coloration with ferric chloride; sodium liberates hydrogen from it. It dissolves in alkalis, and forms both alkyl and acid esters. According to its aldehyde character it reduces Fehling's solution and ammoniacal silver nitrate, and is converted by alcoholic potash, potash, and potassium permanganate into so-called diolic acid. This acid, however, has the formula $C_{10}H_{18}O_3$, and not $C_{10}H_{16}O_3$, as was to be expected if it is obtained by oxidation of diosphenol; its properties are not the same when prepared by different methods. Shimoyana was unable to prepare an oxime or sodium sulphite compound of diosphenol. Thus, there was no real proof of the presence of the aldehyde group.

As regards the eleoptene, the chief part of which boils at 205°—210° (Flückiger), nothing definite was known. The author has prepared the oxime and hydrazone of diosphenol and proved it to be a phenol-aldehyde. In the eleoptene he finds two substances—a ketone similar to menthone, and a reduced aromatic hydrocarbon.—A. C. W.

Pyramidone [Dimethylamido-antipyrine]. W. Filehne. Pharm. Zeit. 1896, 812.

This is the name given to phenyl dimethyl dimethylamido-pyrazolone, or dimethylamido-antipyrine—



Pyramidone is a yellowish crystalline powder, soluble in 10 parts of water; the solution gives a violet-blue coloration with ferric chloride, soon turning violet and disappearing; with a nitrite and sulphuric acid a fugitive violet is produced (antipyrine gives a very stable green). Pyramidone has a similar physiological action to antipyrine, but smaller doses are required.—A. C. W.

Malarine. Pharm. Zeit. 1896, 865.

This is the condensation product of acetophenone and *p*-phenetidine, $C_2H_5O.C_6H_4.N : C(CH_3)C_6H_5$. Malarine crystallises in yellow plates melting at 88° C., is very soluble in warm alcohol, petroleum spirit, benzene, and chloroform; little soluble in cold alcohol and ether; insoluble in water. Warm hydrochloric acid resolves it into its constituents. Malarine has a peculiar odour, somewhat like that of nitrobenzene. It forms a citrate, soluble in 800 parts of cold water, and much more soluble in hot, which may be used as an antipyretic in doses of 0·5 grm. The solution of the citrate (1 in 800) gives a purple-red with ferric chloride; with bromine water, chromic acid, or iodine, a solution of 1 in 5,000 gives a light violet, changing into a deep purple-violet coloration.—A. C. W.

Mydrol [Iodomethylphenyl Pyrazolone]. Pharm. Centralh. 1896, **37**, 718.

Iodomethylphenyl pyrazolone or mydrol is a white odourless powder with a bitter taste, soluble in cold water and alcohol, but insoluble in ether. Mydrol diminishes the heart's action and dilates the pupil, and is not toxic.
—A. C. W.

Caffeïne, Compound of, with Mercuric Chloride. Dennhardt. Ber. pharm. Ges. 1896, **6**, 284.

A compound, $C_8H_{10}N_4O_2.HgCl_2$, is precipitated when a solution of mercuric chloride is added to a solution of caffeïne. It forms needles melting at 245° C. In consequence of the formation of this compound, caffeïne cannot be extracted with ether from acid solutions containing mercuric chloride, but when excess of caustic soda is added, the double salt is decomposed, and ether then extracts the caffeïne.—L. T. T.

Antipyrine, Compound of, with Mercuric Chloride. Hirsch. Ber. pharm. Ges. 1896, **6**, 285.

When equal quantities of these two compounds are mixed in aqueous solution, a compound, $C_{11}H_{12}N_2O.HgCl_2$, is precipitated. Recrystallised from ether, this substance yields microscopic plates. No compound of antipyrine with mercurous chloride could be obtained.—L. T. T.

Kola Nuts. K. Dieterich. Apoth. Zeit. 1896, **11**, 810.

Roasted kola nuts contain much less active principle than the dried nuts. A fluid extract from roasted nuts showed a specific gravity of 0·976, a dry residue of 11 per cent., and 1·04 per cent. of ash. A similar extract from dried nuts showed sp. gr. 0·984, dry residue 13·5 per cent., and ash 1·36 per cent.—L. T. T.

Drugs, Examination of some New. L. Spiegel. Chem. Zeit. 1896, **20**, 970—971.

1. *Yohimbehe Bark.* — This bark comes from the Cameroons in the form of hard chocolate-brown pieces from ½ to 1 cm. in thickness. Extracts prepared in different ways gave alkaloid reactions; and physiological experiments indicated the presence of a strongly poisonous substance. A closer examination showed that, in addition to a Bordeaux-red colouring matter, formed from the corresponding leuco compound by standing in alkaline solution exposed to the air, at least two alkaloids are present, only one of which, yohimbine, could be isolated in a pure condition. Pure yohimbine crystallises from alcohol in white needles melting at 231° C.; it is readily soluble in ethyl-, methyl-, and amyl-alcohol, ether, acetone, &c., but is insoluble in water. Analysis gave numbers agreeing with the formula $C_{23}H_{32}N_2O_4$, or $C_{21}H_{28}N_2O_3 + \frac{1}{2}H_2O$. This alkaloid forms a colourless solution with concentrated sulphuric acid; but if a crystal of potassium bichromate be added, a blue-violet-edged streak is produced, gradually changing to dirty green. Concentrated nitric acid forms a colourless solution, rapidly

becoming intensely yellow; this colour is not altered by heating, and on the addition of sodium hydroxide becomes orange-red. Characteristic reactions are likewise produced with Erdmann's reagent, phosphotungstic acid, phosphomolybdic acid, &c. A hydrochloride, $C_{24}H_{28}N_2O_3 \cdot HCl$, melting at 28° C., a sulphate, and picrate were also prepared. Physiological experiments showed that this alkaloid is extremely energetic.

The second alkaloid, *Yohimbenine*, is easily soluble in 50 per cent. alcohol, and melts at 105°—106° C.

2. *Njalla-bean.*—The juice from these beans has an acrid and bitter taste, and from it was isolated a white substance named Njallin, which decomposed at 160° C. The substance was very hygroscopic. It dissolves readily in hot water, glacial acetic acid, acetone, &c., is very slightly soluble in chloroform, and insoluble in benzene. In sodium carbonate it dissolves to a slightly yellow physiologically-active solution; on exposure to air it becomes red, and loses this activity. This is the case also when it is dissolved in sodium pyrophosphate solution, but in a solution of borax it remains unaltered for a longer period. The alcoholic solution gives a dirty brownish-violet coloration; with ferric chloride, the acetic acid solution showed this reaction sooner.

3. *New-Guinea Nuts (Illipe MacClayana).*—These nuts form the seed of a large fleshy fruit. In appearance they resemble para-nuts, but are about twice as large, and have a smooth, brown, hard shell. The latter contains one, or perhaps several, alkaloids in small quantities. The author found that ether extracted from the dried kernel 8½ per cent. of an oily fat, from which a white crystalline substance separated on long standing. After removal of this fat, the extraction was continued with hot alcohol, and a body was obtained which, when purified, was very hygroscopic, and scarcely soluble in the ordinary solvents. It melted at 165° C., and appears to have the formula $C_{17}H_{32}O_{10}$. The author names this body *Macleyin.*

Macleyin is coloured, first, orange-red, and finally rose-red, by concentrated sulphuric acid; a crystal, of potassium bichromate causes the colour to become violet. Concentrated nitric acid dissolves it in the warm without coloration, whilst fuming nitric acid produces an acid insoluble in water, but separating in a crystalline condition from alcohol. It melted at 200° C. Macleyin also forms a crystalline benzoyl and acetyl derivative, the latter melting at 124° C. Physiological examination showed the properties of macleyin to be very strongly marked.—J. L. B.

Tannin Extracts, The Manufacture of. Fuchs and Schiff Chem. Zeit. **20**, [93], 926.

The first liquors obtained in the manufacture of tannin extracts contain, besides tannic acid, gummy and resinous matters in suspension, some tannin soluble with difficulty, which separates out in the cold liquor, and further, certain colouring matters. To eliminate these undesirable constituents, 1,000 litres of the liquor, at a temperature of 50° C., were mixed with 0·94 kilo. of dissolved albumin, and the temperature was then raised to 60° C. By this treatment all the above-named impurities were precipitated, but it was also found that at the same time upwards of 12 per cent. of the total tannic acid contained in the liquor was precipitated by the albumin. For this reason this method must be described as irrational, and the clarification of the tannin liquors by mechanical means, appears preferable.
—C. O. W.

Alkaloids, Characterisation of, by their Micro-crystalline Precipitate. Vadam. J. Pharm. Chim. 1896, **4**, 485.

See under XXIII., *page* 165.

Quinine Salts; Potassium Chromate, the most Practical Test for the Purity of. J. E. de Vrij. A pamphlet.

See under XXIII., *page* 165.

Tartar Estimation, A Modification of Goldenberg's Method for. M. Zecchin. Staz. Sper. Ag. Ital. **28**, 788; and Analyst, 1896, **21**, 333.

See under XXIII., *page* 168.

Sandalwood Oil. A. J. Hendrix. J. Pharm. Chim. 1896, **4**, 499.

See under XXIII., *page* 168.

Acetone, Volumetric Determination of. E. R. Squibb. J. Amer. Chem. Soc. 1896, **18**, 1068.

See under XXIII., *page* 168.

Soluble Oxidising Ferments and Drugs. E. Bourquelot. J. Pharm. Chim. 1896, **4**, 481.

See under XXIV., *page* 169.

XXI.—PHOTOGRAPHY.

Photography in Colours. Sir H. Trueman Wood. J. Soc. of Arts, 1897, **45**, 158—159.

A process suggested by Dr. Adrian Dansac, and developed by M. Villedieu Chassagne. Four solutions are used, the nature of which the inventor has not disclosed. A negative is taken on a gelatin plate prepared by treatment with one of the solutions, developed and fixed as usual, and from this a transparency or paper print is taken on glass or paper prepared with the same solution. Neither negative nor positive shows any trace of colour. The positive is then washed over successively with the three other solutions, blue, green, and red respectively, and takes up the appropriate colours in the appropriate parts. The process was demonstrated by M. Chassagne in King's College Laboratory to Sir H. Trueman Wood, Prof. J. M. Thomson, Mr. Herbert Jackson, and Capt. Abney, when, it is said, a bunch of flowers and other test objects were successfully photographed in their natural colours.—J. T. D.

PATENT.

Sensitised Paper suitable for Photographic Purposes, Impts. in the Manufacture of. W. Friese-Greene, London. Eng. Pat. 24,440, Dec. 20, 1895.

Instead of coating paper with sensitising material, the latter is added to the paper pulp before it has been formed into paper; or, preferably, by applying the sensitising material to the surface of the "paper-web" just after it had been formed, and whilst it is still moist.—R. B. P.

XXII.—EXPLOSIVES, MATCHES, Etc.

PATENTS.

Explosives [for Guns, &c.], and in Apparatus therefor; Impts. in the Manufacture of. Hudson Maxim, London. Eng. Pat. 16,862, Sept. 9, 1895.

In order to obtain low pressures, which shall be sustained during the travel of the projectile along the chase of the gun, a special form is given to the explosive charge; this forms the subject of the invention.

The explosive material is first made into a sheet of suitable size and thickness, with a number of holes or indentations upon one surface, but which do not pass right through the sheet.

The sheet is then bent into a hollow cylinder, with the indentations on the inner side.

The charge is ignited on the inside, and the outer wall of the cylinder is pressed against the wall of the powder chamber, so that combustion can only take place over the inner surface of the cylinder.

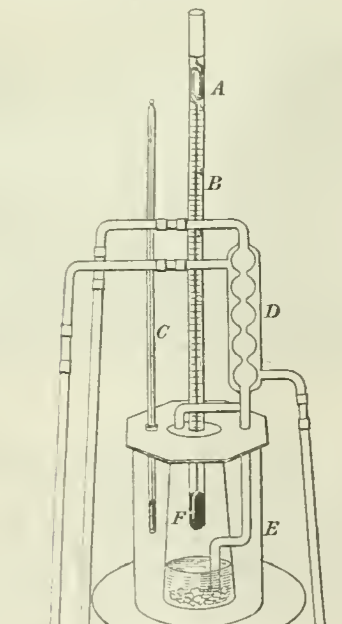
As combustion proceeds, the area exposed to its action constantly increases, whereby the pressure in the gun is maintained as the projectile is driven along the chase.
—R. B. P.

XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

Ebullioscope, A Modified Form of the. H. W. Wiley. J. Amer. Chem. Soc. 1896, **18**, 1063—1067.

The chief difficulties attending the use of the ebullioscope for the determination of alcohol in wine and beer have been (1) that the reading of the thermometer is influenced, not only by the temperature of the vapour emitted, but also by the initial boiling point, which is affected by the dissolved matters contained in the liquid; (2) that it is difficult to ensure the continuous evolution of a vapour containing a definite percentage of alcohol; (3) that when the condensed vapours, as is usually the case, are returned through the apparatus and come into contact with the uncondensed vapours, the reading of the thermometer is affected; and (4) that the variation of atmospheric pressure renders necessary a frequent checking of the instrument with pure water. These defects the author proposes to remedy by means of the apparatus here described.



The flask F is closed by an india-rubber stopper, through which pass the thermometer B and a tube leading to the condenser D. The vapours given off are condensed in D and returned to the flask, entering the latter below the surface of the liquid. The glass cylinder E protects the apparatus from the influence of external temperature, and this cylinder rests upon a disc of asbestos. The flask covers a hole in the centre of the asbestos disc, which is a little smaller than the bottom of the flask. The cylinder is covered at the top with a disc of rubber cloth. The thermometer C indicates the temperature of the air between F and E, and the reading of B should always be made at a given temperature, as indicated by C. The long tube leading from the top of D is open at its lower end, in order to secure atmospheric pressure in F, and to prevent diffusion of the alcohol vapours through D. The thermometer B is set, in the first place, by putting the bulb in water containing 16 grms. of common salt in 100 c.c. When the water is fully boiling, the excess of mercury is removed from the column into the receptacle A; and then, on placing in ordinary boiling water, the column of mercury will be found a little above the 5° mark, by A.

The liquid to be tested should not contain more than 5·5 per cent. of alcohol, and stronger liquids are diluted before testing. The temperature of C is brought to about 90° C. in ten minutes, and after boiling for a few minutes, B indicates a constant temperature, and readings are taken at intervals of about 30 seconds for two minutes. The reading of this thermometer is arbitrary, but the degrees indicated are Centigrade. When the thermometer is graduated so that it indicates about 5° on the scale in the vapour of boiling water, it will be found that the presence of 1 per cent. by volume of alcohol causes a depression of about 0·8° in the boiling point. The reading of the thermometer is facilitated by covering the bulb with a test tube containing water.

To avoid the correction for barometric pressure, it is merely necessary to use at the same time a similar apparatus containing boiling water, and to read the thermometers in the two flasks at the same instant.—R. B. B.

Temperature Determinations. [*Le Chatelier Pyrometer*]. Hecht. Centralbl. f. Glas Ind. 1896, **11**, 311 and 321.

See under VIII., *page* 143.

PATENT.

Fire-damp or other Noxious Gas or Gases in the Atmosphere of Mines and other Places; Method of and Apparatus for Detecting and Indicating the Presence of. E. L. Mayer, Billiter Square, London, and G. Bush, Gracechurch Street, London. Eng. Pat. 18,188, Sept. 28, 1895.

The presence of fire-damp or other noxious gases in mines or other places is detected and indicated by causing such gas or gases to generate, by means of a gas battery, charges of static electricity that are caused to control means or apparatus whereby a visual or audible signal, or both, are brought into operation. The gas battery consists of a hermetically closed box filled with pure air, through the walls of which are passed a number of porous non-conducting blocks, as fire-clay, &c., impregnated with an electrolyte, as sulphuric acid, and connected up in series. The box is exposed to the noxious gas, and the outer ends of the blocks, which generally project, being also exposed, set up a charge of static electricity in the conductors connected therewith. The specification contains descriptions and drawings of a large number of alternative forms of apparatus which may be employed to make use of this electricity to control the various signals. The following distinct forms are illustrated and described:—(1) A condenser, (2) an ordinary quadrant electrometer, (3) a Kelvin quadrant electrometer, (4) a Bohnenberger electroscope, (5) a condenser embodying the principle of a Bohnenberger electroscope, and (6) an ordinary gold-leaf electroscope, which is preferably used in connection with two condenser plates. The signals operated may be audible ones, or electric lamps may be brought into use, or put out, or the light rendered much less. This last is especially useful in mines. The light of a lamp may act upon a selenium cell to produce a signal; and a special selenium cell is described for this purpose, consisting of two metallic gratings connected together by selenium, and so arranged that a considerable surface of selenium can be exposed to the rays of light.—R. S.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Analytical Chemistry, The Importance in, of the Ionic Dissociation Theory of Arrhenius. F. W. Küster. Zeits. f. Elektrochem. **3**, (1896), 233—236 and 257—260.

See under XI. A., *page* 148.

Formaldehyde as a Reducing Agent in Analysis. B. Grützner. Arch. Pharm. 1896, **234**, 634.

Chloric Acid.—On adding silver nitrate and formaldehyde to a solution of potassium chlorate and acidifying with nitric acid, the whole of the chlorine is converted into silver chloride, thus:—

$HClO_3 + 3CH_2O + AgNO_3 = 3CH_2O_2 + AgCl + HNO_3$.

The reaction is hastened by warming. In this way chloric acid may be conveniently determined, either gravimetrically or volumetrically. If the chlorate contain chlorides, these may first be estimated by titration with $\frac{1}{10}$ N silver solution, and the total chlorine is determined after the addition of formaldehyde and nitric acid.

Bromic Acid may be determined in the same way, but the mixed solution must be heated for 2—2½ hours on a water-bath to complete the reaction.

Iodic Acid is not reduced; periodic acid and perchloric acids are only reduced in very small proportion (the former to iodic acid). The reaction of formaldehyde with potassium chlorate may be used for the estimation of the former by adding to the aldehyde some chlorate, together with a few c.c. of nitric acid and silver nitrate solution, and weighing the resulting silver chloride.—W. G. M.

Sulphates, Decomposition of, in Analysis, by Ammonium Chloride. M. Chikashigé. J. Coll. of Science, Imp. University, Japan, **7**, iv., 251—252.

In the separation of barium from magnesium and alkali salts, the small quantity that is left in solution by ammonium carbonate is precipitated by the addition of 3 or 4 drops of dilute sulphuric acid. The author contradicts the statement of Fresenius in a footnote to § 153 A. of the seventh edition of his "Quantitative Analysis," that gentle ignition for the purpose of driving off ammonium salts will also effect the removal of any small quantity of sulphuric acid which may remain after precipitating the barium, sulphates of the alkalis being converted into chlorides on ignition with a large proportion of ammonium chloride. It was proved by experiment that very little, if any, of the sulphuric acid is driven off by ignition at a temperature barely approaching red heat. (See this Journal, 1895, 629.)—A. S.

Limestones, Estimation of Iron and Alumina in. Désiré de Paepe. Chem. Zeit. 1896, **20**, 1004.

The very convenient method of separating iron from alumina by means of ammonium sulphide, in presence of ammonium tartrate, is prejudiced by the presence of much lime. The present method is proposed as a rapid and convenient one for attaining the object in view, in spite of the condition named.

The limestone (5 to 10 grms.) is treated in a covered beaker with strong hydrochloric acid and boiled for five minutes. The diluted and filtered solution is treated with a few drops of nitric acid and heated to boiling. When cold, an excess of ammonia is added and the whole boiled until the odour of ammonia becomes slight. It is then filtered, washed, and made up to 500 c.c. In 50 c.c., lime and magnesia are determined.

The ammonia precipitate is dissolved in hydrochloric acid, the solution diluted to 125 c.c. Alumina and iron are determined in 50 c.c., whilst iron alone is determined in another 50 c.c., as follows:—10 per cent. of tartaric acid solution (5 or 10 c.c.), then an excess of ammonia, and finally, a slight excess of ammonium sulphide are added. The whole is heated moderately for 2—3 hours, filtered, and washed with boiling distilled water containing a trace of ammonium sulphide. This should be done quickly, and the funnel covered so as to exclude air as much as possible. The precipitate is redissolved in hydrochloric acid, treated with a few drops of nitric acid, and heated to boiling. The iron is again precipitated with ammonia, filtered, washed, &c., and weighed.—N. H. J. M.

Alkalis and Alkaline Carbonates, Volumetric Estimation of, with Phenolphthaleïn and Methyl-orange as Indicators. F. W. Küster. Zeits. anorg. Chem. 1896, **13**, 127.

In the titration of mixed solutions of alkalis and alkaline carbonates, the only satisfactory method, it is said, is that recommended by Winkler, *viz.*, the barium chloride method, with direct titration of the solution and precipitate, with methyl-orange as indicator.

Methyl-orange (contrary to previous statements) is decidedly coloured by carbonic anhydride. For accurate work it is essential to have for comparison of "normal tint," a solution of the indicator of like strength and saturated with carbonic anhydride. Phenolphthaleïn is coloured by alkaline carbonates in weak solutions, and is therefore not reliable as an indicator.—L. T. T.

Phosphoric Acid in Potable Waters, Determination of. C. Lepierre. Bull. Soc. Chim. 1896, **15**, [23], 1213—1217.

The maximum limit of phosphoric anhydride in potable waters was fixed at 0·5 mgrm. by the congress of Brussels, this number probably indicating sewage contamination. The author has devised a colorimetric method based upon the yellow colour of a solution of ammonium phosphomolybdate in nitric acid. This colour is found to be exactly proportional to the amount of phosphoric acid present (at least up to 30 mgrms. P_2O_5 to the litre) when the temperature is uniform. A series of standard tubes is prepared containing known quantities of phosphoric acid in the form of ammonium phosphomolybdate. In testing a water, a litre is evaporated with the addition of nitric acid, the residue heated sufficiently to render the silica insoluble, redissolved in nitric acid, tested with molybdate, and the colour compared with the standard tubes. Silica, if not removed, interferes with the reaction.—A. K. M.

Boric Acid, Volumetric Determination of. G. Jörgensen. Zeits. f. angew. Chem. 1897, 5. (See also this Journal, 1896, 742—743.)

The method is based on the facts that in aqueous solutions boric acid behaves like an excessively weak acid, whilst in presence of mannite or glycerin it is capable of neutralising caustic soda. The author finds that, in aqueous solution, phenolphthaleïn is reddened when about 1 molecule of NaOH is added to $6B(OH)_3$; in presence of excess of glycerin the end point corresponds fairly accurately to the formation of $NaBO_2$.

The method is expected to be of service in the determination of the small quantities of boric acid which are sometimes added to foods as a preservative. As an example of its application, three separate quantities of milk of 100 c.c. each were treated with 0·39, 0·1, and 0·0 grm. of boric acid respectively; after adding a little Na_2CO_3 these were evaporated to dryness and incinerated; the residue was taken up with dilute sulphuric acid, filtered, and titrated with caustic soda, using phenolphthaleïn as indicator. About 20 c.c. of glycerin were then added (causing the red coloration to disappear), and caustic soda run in until it appeared again. From the amount of caustic soda used in the second titration, the quantity of boric acid is calculated, 35 grms. of B_2O_3 corresponding to 850 c.c. of normal alkali. It is, however, preferable to titrate a solution containing a known quantity of boric acid side by side with that under examination, treating both solutions in exactly the same way; this eliminates some uncertainty which exists owing to the end point being deficient in sharpness; it also makes it unnecessary to know the strength of the caustic soda solution.

In the samples of milk above mentioned the author found 0·388, 0·107, and 0·013 grm. of boric acid.—T. E.

Perchlorates in Chili Nitrate of Soda; Detection and Estimation of. B. Sjollema. Chem. Zeit. **20**, 1002.

Detection of Perchlorates in Chili Nitrate of Soda.—20 grms. of nitrate of soda are dissolved in water (20 c.c.), cooled, and gradually treated with strong sulphuric acid (15 c.c.). The nitric acid is then reduced with hydrogen sulphide and the sulphur removed by filtration. On adding a solution of rubidium chloride to the filtrate, a crystalline precipitate is formed, if sufficient perchlorate be present. Potassium acetate may be employed, but the reaction is less sensitive. The crystals are washed with a little water and dilute alcohol, and ignited with a little sodium nitrate or carbonate; the product is dissolved in dilute nitric acid and tested for chloride.

Another method is to distil the solution after addition of sulphuric acid, and to treat the last portions of the distillate with rubidium chloride, either directly or after precipitation of the sulphuric acid.

Estimation of Perchlorates in Nitrate of Soda.—The substance (10 grms.) is ignited in a platinum crucible, or better, in a conical platinum flask and the chlorine estimated by titration, using Volhard's method. From the results so obtained, the amount of chlorides in the sample before ignition is deducted. Owing to the irregular distribution of perchlorates in Chili nitrate of soda, it is desirable to dissolve a large amount (100 grms.) of the substance in water (500 c.c.); in 50 c.c. of the solution, the chlorides are determined directly, whilst a second 50 c.c. are evaporated to dryness and ignited.—N. H. J. M.

Bismuth and Lead, The Separation of. A. L. Benkert and E. F. Smith. J. Amer. Chem. Soc. 1896, **18**, 1055—1056.

Bismuth may be completely separated from lead by precipitation of the former as basic formate. This is effected as follows:—A nitric acid solution of the mixed nitrates is almost neutralised with sodium carbonate, and a considerable quantity of sodium formate is then added, together with a few drops of aqueous formic acid. The mixture is heated to boiling and boiled for five minutes; the precipitate is then allowed to subside, but filtered while still hot. The precipitate of basic formate is washed with hot water, dissolved in dilute nitric acid, and precipitated with ammonium carbonate. The ignited bismuth trioxide contains a small quantity of lead, and to effect complete separation it is dissolved in nitric acid, almost neutralised with sodium carbonate, and precipitated as before. On the second ignition no lead is found in the residual bismuth trioxide. —R. B. B.

Vanadium and Arsenic, The Separation of. C. Field and E. F. Smith. J. Amer. Chem. Soc. 1896, **18**, 1051—1052.

When arsenates and vanadates are heated in an atmosphere of hydrochloric acid gas, both acids are volatilised. If, however, the dry sulphides of arsenic and vanadium are treated in this manner, arsenic trisulphide is completely expelled, whilst brown vanadium sulphide is not altered at temperatures below 250° C.

These facts may be utilised in the analysis of *vanadinite*. The finely divided mineral is placed in a porcelain boat and heated in a combustion tube in a current of hydrochloric acid gas. Vanadic and arsenic oxides are expelled; lead phosphate and chloride remain behind. The oxides are made alkaline and digested with ammonium sulphide, and the sulphides are set free by a dilute acid. These sulphides are washed and dried, separated by hydrochloric acid gas as above, converted into oxides, and weighed.—R. B. B.

Arsenic, Volumetric Estimation of. E. Szarvasy. Ber. **29**, [18], 2900—2902.

The arsenic is precipitated from solution as sulphide in the usual way, and the liquid filtered through an asbestos filter consisting of a combustion tube, 80—100 cm. long, having a plug of asbestos of suitable quality about 15 cm. from its upper end. The tube is held vertically above a beaker, and the liquid is best poured directly on to the asbestos by means of a long funnel. Having washed the precipitate with water, and then with alcohol and ether to expedite the drying, a stream of dry air or carbon dioxide is passed through the gently inclined tube, which may be warmed either in a water bath or by means of a flame, cautiously applied. It is necessary to dry the tube thoroughly, to prevent sulphur dioxide from being subsequently retained. A good plug of glass-wool is now placed in the tube, about 10 cm. from the other end; a stream of oxygen is passed through the tube, which is then kept heated to gentle redness immediately behind the asbestos plug; and by means of another flame the sulphide is heated, care being taken that it is made to burn away gradually, and not all at once. Arsenic trioxide sublimes and condenses beyond the glowing ring; if the sublimate is tinged yellow, the unburnt sulphide causing this is driven back to the asbestos plug, the stream of oxygen being meanwhile stopped, and, on restarting the stream of gas, the sulphide can be completely burnt to oxide. The sulphur dioxide is next driven out of the tube by a stream of air, and the cold tube held obliquely, glass-wool end uppermost, whilst sodium hydrate solution (about 30 to 40 c.c. of 10 per cent. solution) is poured down it to dissolve out the arsenic trioxide. Having washed it down with 400—500 c.c. of water, the solution is acidified slightly with HCl, saturated with excess of sodium bicarbonate, and titrated iodometrically, the iodine solution being standardised against sublimed arsenic trioxide. The test analyses quoted are very satisfactory.—H. B.

Copper; Electrolytic Analysis for Technical Purposes: Estimation of Arsenic, Antimony, Sulphur, and Foreign Metals. A. Hollard. Comptes rend. 1896, **123**, [24], 1063—1065.

The solution from which the copper has been removed, is evaporated almost to dryness, taken up with a little hydrochloric acid, made up to about 200 c.c., and heated to to 70°—75°; hydrogen sulphide is then passed through till quite cold, and the solution filtered after 24 hours.

Precipitate, (arsenic, antimony, traces of lead, and, it may be, of copper) is well washed, treated with ammonium sulphide, and filtered; the filtrate is evaporated, oxidised by hydrochloric acid and potassium chlorate, the arsenic precipitated as magnesium ammonium arsenate, and the antimony in the filtrate from this converted into sulphide, dissolved in alkaline sodium sulphide, and deposited by electrolysis.

Filtrate, (iron, nickel, and cobalt) is boiled down, treated with nitric acid to peroxidise the iron, evaporated till fumes of sulphuric acid appear, and redissolved in water.

Slight excess of ammonia is now added to precipitate the iron (which is redissolved and reprecipitated to separate traces of nickel or cobalt), which can be determined volumetrically. To the filtrate, ammonia and ammonium sulphate are added, and the nickel and cobalt are deposited electrolytically.

To determine silver, the deposited copper (if rich in silver), or a new sample of 10 grms. to 50 grms., is dissolved in nitric acid, the silver precipitated as chloride, the chloride redissolved and reprecipitated, then dissolved in potassium cyanide, and submitted to electrolysis.

To determine lead, 10 grms. are dissolved in a large excess of nitric acid, and the lead deposited by electrolysis at the anode as PbO_2.

Sulphur is determined by dissolving a sample in aqua regia and proceeding in the ordinary way.—J. T. D.

Manganese and Tungstic Acid, The Separation of. W. T. Taggart and E. F. Smith. J. Amer. Chem. Soc. 1896, **18**, 1053—1054.

The two methods tried by the authors have both proved insufficient to effect a quantitative separation of manganese from tungstic acid. By digestion with yellow ammonium sulphide in presence of ammonium chloride, and conversion of the manganous sulphide into oxide, the weight of the latter found, is too great, tungstic acid apparently adhering to the metallic oxide. In the second method the mixture was digested with a strong solution of potassium carbonate, and, in this case, ignition of the manganese carbonate to oxide gave too low a percentage of manganese. The authors suggest fusion with an alkaline carbonate as a possible solution of the difficulty.—R. B. B.

Iron and Alumina, Estimation of, in Phosphates and Superphosphates. Von Grüber. Zeits. f. angew. Chem. 1896, **11**, 741—742.

The solution for estimating iron and alumina separately, is prepared as follows:—The crude phosphate or superphosphate (5 grms.) is heated with water (150 c.c.) and strong hydrochloric acid (20 c.c.), in a covered dish on a water-bath, until the phosphates are dissolved. It is then evaporated to dryness, the residue redissolved by heating with acid and water, and, when cold, diluted to 500 c.c. It is then filtered and aliquot portions used for estimating alumina and iron.

Estimation of Alumina.—50 c.c. of the solution (= 0·5 grm. of substance) are heated in a 200-c.c. flask and treated with 20 per cent. caustic soda until a slight precipitate is formed; more soda (30 c.c.) is then added, the whole heated to boiling, and kept for 10 minutes in a warm place, being frequently shaken. The solution is diluted to 200 c.c. and filtered through a dry filter. 100 c.c. of the filtrate are slightly acidified, heated to boiling, treated with an excess of ammonia, and boiled. As soon as the precipitate (aluminium phosphate) has settled, it is filtered, well washed, dried, and ignited. The weight multiplied by 41·8 × 2 gives the percentage of alumina.

Estimation of Iron.—100 c.c. of the solution, contained in a 250-c.c. flask, are treated with pure zinc and the necessary amount of dilute sulphuric acid. After about an hour, the solution is cooled, the flask filled to the mark, 50 c.c. of the filtered solution run into a beaker containing 50 c.c. of 20 per cent. sulphuric acid and 200 c.c. of water, and titrated with permanganate. Another 50 c.c. of the solution are added, and again titrated. This is repeated a third and, if necessary, a fourth time; but the third titration generally agrees with the second.

Very good results are obtained by the methods, which are, it is said, simpler and more rapid than Glaser's.

—N. H. J. M.

ORGANIC CHEMISTRY.—QUALITATIVE.

Analytical Chemistry, The Importance in, of the Ionic Dissociation Theory of Arrhenius. F. W. Küster. Zeits. f. Elektrochem. **3**, (1896), 233—236 and 257—260.

See under XI. A., *page* 148.

Quinine Salts, Potassium Chromate, a Test for the Purity of. J. E. de Vrij. A pamphlet.

One grm. of the sample is heated with 10 c.c. of water until just dissolved. When testing the commercial sulphate, the solution should be faintly alkaline to red litmus paper. Six cubic centimetres of a 5 per cent. solution of perfectly neutral potassium chromate are now added, and the separation of normal quinine chromate, which sets in immediately, is completed by cooling to 15° C. The liquid is now filtered through glass-wool, the filtrate being passed through the filter a second time. After having made quite sure that further addition of the chromate solution gives no more precipitate, the liquid is introduced into a test-tube of about 50 c.c. capacity and mixed with 9—10 drops of a 5 per cent. solution of caustic soda. After allowing it to stand over night, the tube is examined, and if the sample of quinine salt be really pure, not the least trace of precipitate should be visible. When, however, the sample contains an admixture of cinchonidine, quinidine, cinchonine, or hydroquinine, a more or less copious precipitate is obtained, which in careful hands may even be made the basis of a quantitative estimation of the inferior alkaloids, by the well-known method of comparing with standard samples.

The quinine chromate can be easily converted into the pure alkaloid by dissolving it in 100 parts of boiling water and pouring the liquid into soda solution.—L. de K.

Formaldehyde. Detection of Small Quantities of. L. Kentmann. Pharm. Gen. Anz. 1896, **8**, 356. Chem. Zeit. **20**, [104], 313.

0·1 grm. of morphine hydrochloride is dissolved in 1 c.c. of strong sulphuric acid in a test-tube, and an equal volume of the solution to be examined is added to it without mixing; in the presence of formaldehyde the aqueous solution will become clear red-violet in colour after the lapse of a few minutes. The reaction is sensitive to 1 : 5,000—6,000. The author recommends this test as a reaction for the identification of the German Pharmacopœia III.—W. G. M.

Alkaloids, Characterisation of, by their Micro-Crystalline Precipitates. Vadam. J. Pharm. Chim. 1896, **4**, 485.

The microscopic examination of alkaloids has the advantages that any method of extraction may be used, and that, once the alkaloid residue is obtained, a single drop of dilute solution is sufficient for each reaction; thus some quantity of the residue may be preserved for other conclusive tests.

The residue of alkaloid having been obtained and weighed, is dissolved in 2 per cent. hydrochloric acid to give a solution containing about 0·5 per cent. of alkaloid, one drop is placed in the hollow of each of a dozen slides, the drop is mixed with one drop of the reagent, and a cover-slip placed over. After half an hour the slides are examined under a low power. The following reagents are used:—1. Mayer's reagent: 13·54 grms. mercuric chloride, 49·8 grms. potassium iodide, made up to 1 litre. 2. Bouchardat's reagent: 10 grms. iodine, 20 grms. potassium iodide, 500 grms. water. 3. Saturated solution of picric acid. 4. Gold chloride, 5 per cent. solution. 5. Platinum chloride, 5 per cent. solution. 6. Schultze's reagent: solution of sodium phospho-antimoniate. 7. Scheibler's reagent: solution of sodium phosphotungstate. 8. Lepage's reagent: 10 per cent. cadmium iodide solution. 9, 10, and 11. Saturated solution of potassium bichromate, ferrocyanide, and ferricyanide. 12. Sodium nitroprusside, 10 per cent. solution.

In each case there may be a crystalline precipitate, an amorphous precipitate, or no precipitate at all. By means of these reactions in any one case all the alkaloids will have been eliminated except some two or three; the particular one present may then be recognised by the crystalline forms observed.—A. C. W.

Pyramidone. [*Dimethylamido-Antipyrine.*] W. Filehne. Pharm. Zeit. 1896, 812.

See under XX., *page* 160.

ORGANIC CHEMISTRY.—QUANTITATIVE.

Starch-Hydrolysis by Diastase, On the Experimental Methods employed in the Examination of the Products of. H. T. Brown, G. H. Morris, and J. H. Millar. Proc. Chem. Soc. 1896, [172], 241.

The paper is divided into the following sections:—(1) The determination of solids from solution-density; (2) determination of specific rotatory power; (3) the relation of $[\alpha]_j$ to $[\alpha]_D$; (4) determination of cupric reducing power; (5) limits of accuracy of the methods.

The author states that this account is a preface to a series of papers dealing with the question of starch-hydrolysis, and is a critical review of the experimental methods which have been employed by different observers who have approached this subject. An attempt has also been made to remove the misunderstanding which still exists as to the relations of the different systems of notation.

The determination of the total solids from the density of the solution by the employment of the "divisor" method admits of great accuracy if the solution-densities of the pure substance have been previously determined.

For mixed starch hydrolytic products, the divisor for equal concentrations increases with the specific rotatory power, and in such a regular manner that when the value of R is known, the divisor at any given concentration can be calculated. From the relation which this divisor bears to the divisor of the apparent maltose present in the mixed hydrolytic products, it is deducible that the divisor for the amylin constituent is constant for equal concentrations, even in starch products of very different grades of hydrolysis.

In the section on specific rotatory power, the methods of exact determination are discussed. As the dispersive power of cane sugar is sensibly different from that of dextrose and starch hydrolytic products obtained by diastase, the factors for the conversion of $[\alpha]_j$ into $[\alpha]_D$ are not identical in these cases. Much confusion of these relations has also been introduced by the unrecognised fact that $[\alpha]_j$ has been referred to two distinct rays in the yellow of different refrangibility.

The cupric reduction of maltose and of the products of starch-transformation is constant only when the conditions of experiment are identical.

Dextrose, Levulose, and Invert Sugar, The Solution Density and Cupric-Reducing Power of. H. T. Brown, G. H. Morris, and J. H. Millar. Proc. Chem. Soc. 1897, [173], 4—5.

The authors have extended the methods described in their previous paper (Proc. Chem. Soc. 1896, **12**, 241) to the examination of the solution density and cupric-reduction of dextrose, levulose, and invert sugar. They find that the solution densities of the two former differ considerably with the same concentration of the solution, but that the volume occupied in solution by a unit of weight of each is less at lower than at higher concentrations, consequently the divisor to be applied to the specific gravity decreases with the concentration. The solution density of invert sugar was calculated from those of dextrose and levulose, and the results so obtained were confirmed at various points by direct experiments.

They also find that the cupric-reducing powers of the three sugars, when determined under their standard conditions, are, for dextrose, $\kappa = 117$ to 105; for levulose, $\kappa = 107 \cdot 5$ to 101; and for invert sugar, $\kappa = 111$ to 103. The higher numbers are obtained when a small amount of cuprous oxide is precipitated, and the lower when reduction is carried nearly to the maximum. When the experimental numbers are expressed in the form of a curve, it is found that at the one end, taking the cupric reduction of dextrose at 100, levulose is represented by 91·3, and invert sugar by 94·2; at the other end of the curve, the ratio is 100, 91·6, and 97·5; whilst at an intermediate point, which corresponds to the amount of cuprous oxide usually reduced, the relation is, dextrose, 100; levulose 92·3; and invert sugar, 96·15.

Maltose and of Soluble Starch, On the Specific Rotation of. H. T. Brown, G. H. Morris, and J. H. Millar. Proc. Chem. Soc. 1896, [172], 242.

The authors' determinations of the specific rotatory power of maltose at a temperature of 15·5° do not confirm the statement of Meissl that the values of $[\alpha]_D$ vary with the concentrations between 2 and 20 per cent., but confirm the general statement of Ost that between these limits the specific rotatory power is constant. At higher concentrations than 20 per cent., the specific rotatory power diminishes slightly.

The actual results point to a value of $[\alpha]_D = 137 \cdot 93°$, which is sensibly greater than Ost's value of 137·46° at 15·5°.

This discrepancy is due to the fact that Ost employed weighed quantities of hydrated maltose which had been dried in a desiccator over sulphuric acid. The authors find that even after six weeks' drying in this manner, hydrated maltose contains 0·46 per cent. more water than corresponds to $C_{12}H_{22}O_{11} . H_2O$. If Ost's numbers are corrected for this they give values, up to 20 per cent. concentrations, of $[\alpha]_D = 138 \cdot 12°$ at 15·5°, a result almost exactly identical with that of the authors.

The specific rotatory power of soluble starch for concentrations of 2·5 to 4·5 per cent. is, at 15·5, $[\alpha]_D = 202 \cdot 0°$.

Starch-Hydrolysis by Diastase, On the Relation of the Specific Rotatory and Cupric Reducing Powers of the Products of. H. T. Brown, G. H. Morris, and J. H. Millar. Proc. Chem. Soc. 1896, [172], 243.

When starch is transformed by diastase, a certain relation is always found to subsist between the cupric reduction and specific rotatory power of the hydrolytic products. This relation can be expressed in such a manner as to be entirely independent of any view we may hold as to the true nature of the transformation products, and it is of so exact a nature that if one property is known the other can be predicted with certainty. This is true not only for the mixed hydrolytic products, but for any fractionated portion of them.

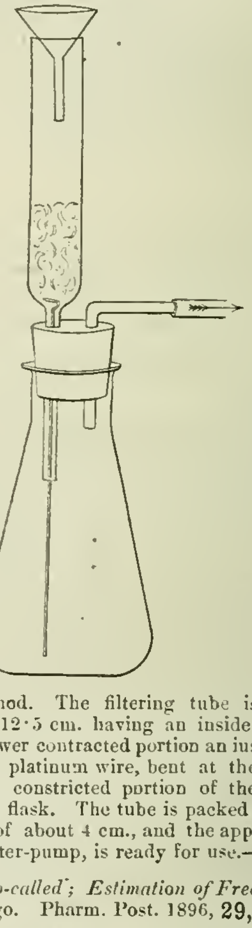
The authors regard this fact as lying at the root of the whole question of starch-hydrolysis, and, as it is still not admitted by most continental workers, they bring forward a large amount of fresh evidence which they regard as absolutely conclusive.

The properties of soluble starch being R = 0, $[\alpha]_D = 202°$, and of maltose, R = 100 and $[\alpha]_D = 138 \cdot 0$, then the relation of specific rotation and cupric reduction for any mixture or fractionation of the starch-hydrolytic products will be expressed by $[\alpha]_D = 202 - 0 \cdot 64$ R.

The authors have examined the published results of C. J. Lintner and of Ost, both of whom have denied the existence of any relation between $[\alpha]_D$ and R, and find that, when rightly interpreted, they, for the most part, strictly conform to the law of relation expressed above.

Alkaloidal Extracts, The Separation of. C. Platt. J. Amer. Chem. Soc. 1896, **18**, 1104.

The author has found the apparatus here illustrated of great value in effecting the rapid separation of the emulsions often met with in the analysis of alkaloids; as, for example, in the petroleum spirit and benzene extractions of



Dragendorff's method. The filtering tube is 19 cm. in length, the upper 12·5 cm. having an inside diameter of 14 mm., and the lower contracted portion an inside diameter of 3 mm. A stout platinum wire, bent at the upper end, passes through the constricted portion of the tube to the bottom of the 8-oz. flask. The tube is packed with washed cotton to a depth of about 4 cm., and the apparatus, when connected with a filter-pump, is ready for use.—R. B. B.

"Sulphur Oils," so-called; Estimation of Free Sulphur in. G. Morpurgo. Pharm. Post. 1896, **29**, 501.

Oils commercially extracted by means of carbon bisulphide often contain traces of sulphur resulting from the impurity of the bisulphide. To estimate such sulphur, a weight of 25 grms. of the oil is saponified with caustic soda, then mixed with 10 c.c. of a 10 per cent. solution of lead acetate, and acidified with a few drops of acetic acid. After thoroughly shaking the mixture, the supernatant liquor is withdrawn from the precipitate of free fatty acids, lead sulphide, and lead soap; the precipitate is then covered with 50 c.c. of 95 per cent. alcohol, and strongly acidified with acetic acid, and the mixture is warmed on the water-bath until the lead sulphide is deposited. The fatty acids set free, are removed by means of alcohol, and the residue is thoroughly washed, first with warm but very dilute acetic acid, and then with a 5 per cent. solution of ammonia, after which it is collected on a filter, washed with water, dried, and weighed as PbS.

—W. G. M.

Glycerin, New Method of Estimating. Bordes and de Raczkowski. Comptes rend. 1896, **123**, [24], 1071—1072.

The method is based on one of Nicloux for estimating small quantities of alcohol. When potassium bichromate is caused to act upon glycerin in presence of sulphuric acid, formic acid and carbonic acid are formed. A solution of potassium bichromate of 48 grms. per litre is added to 5 c.c. of the sample, mixed with 2 c.c. of sulphuric acid. The end of the reaction is marked by the change of tint (to green), when a drop of unchanged bichromate mixes with the titrated solution. Each c.c. of the bichrome solution corresponds to 1 grm. of glycerin per litre. The method is only available for solutions containing under 2 grms. of glycerin per litre; stronger solutions must be diluted. The solutions are so adjusted that 0·1 c.c., more or less, of bichromate give either a yellowish-green or bluish-green tint.—J. T. D.

Pentosans, Proportion of, in Cotton Wool. H. Suringar. Chem. Zeit. Rep. 1896, 290.

The percentage of pentosans in cotton wool is determined by the furfuraldehyde resulting from a phloroglucinol precipitate obtained from wadding. The proportion is very small, and when, therefore, large quantities of gummy substances are found in purified wadding, they must have resulted from the hydrolysis of the cellulose itself.

—W. G. M.

Invert Sugar, The Determination of. A. Leys. J. Pharm. Chim. 1896, **4**, 488.

In the volumetric determination of invert sugar by means of Fehling's solution, the cuprous oxide often remains yellow, and the liquid does not clear, so that the exact final point is difficult of observation. The author finds that this is due to the acidity of the solution. It may be avoided by making alkaline with concentrated potash, before adding the Fehling's solution. In the case of wines it is not possible to make the liquid alkaline, because of the resulting precipitation of phosphates; the feeble acidity usually met with in wines is, however, no obstacle; therefore the following process is recommended. Solutions of hydrochloric acid and potash are prepared exactly equivalent to one another, 50 c.c. of the wine are inverted by 2·5 c.c. of acid, and, after cooling, 2·5 c.c. of potash are added, and the liquid made up to 55 c.c.—A. C. W.

Sugar Solutions, On the Clarification of, for Analysis. K. C. Neumann. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 524—526. (From Oest. ung. Zeits.)

The author examines the effect on polarisations, of the addition of alum. His results show that an immoderate excess of alum affects the polarisation, and has an injurious action on the colour of sugar solutions; but that 1 to 2 c.c. of a saturated solution of alum added with 1 to 2 c.c. of basic acetate of lead, as adopted by the Austro-Hungarian chemists in 1890, has absolutely no injurious effect, but gives an absolutely clear solution.—L. J. de W.

Starch, Hydrolysis of, by Acids; An Analytical Investigation of. G. W. Rolfe and G. Defren. J. Amer. Chem. Soc. 1896, **18**, 869—900.

The authors have carried out investigations to determine whether there is any simple constant relation between the optical rotation and the cupric reducing power of acid hydrolysed starch products; also whether there are any laws that affect the three simple bodies, assumed to be formed, and determined by the usual methods of analysis.

One hundred grms. of a commercial starch of good quality was mixed with a litre of water containing the acid, placed in an autoclave, and when the contents had acquired constant temperature, portions of 50 to 75 c.c. were withdrawn at different stages of the conversion, and shaken with a few grms. of marble dust. Two drops of decinormal sodium hydroxide were then added, and the mixture cooled and filtered. This method of neutralisation gave clear filtrates; low converted products, however, required to be treated with alumina before filtering. The specific gravity of the sample was estimated by a Westphal balance corrected to a temperature of 15·5° C.; the factor 3·86 being employed to calculate the total solids; the specific rotatory power was determined, also the cupric reducing power, by O'Sullivan's method. The authors have studied the action of acids on starch under varying conditions, and their results show that the cupric reducing power of the total product bears a constant relation to the specific rotatory power, so that the one being known, the other may be calculated. From the curves plotted by taking the values of maltose, dextrin, and dextrose, they have constructed a table, calculated on the factor 3·86, giving the values within one-tenth per cent. of maltose, dextrose, and dextrin, for successive stages of acid hydrolysis represented by each degree of rotation between 195° and 53·5°. From the examination of some commercial glucoses it is concluded that: (1) in any homogeneous acid converted starch product, irrespective of the conditions of hydrolysis, the specific rotatory power always represents the same chemical composition; (2) that only three simple carbohydrates, possible in molecular aggregates (not considering the small amount of reversion products), exist in the solution of a starch product hydrolysed by acids.

A table is appended, from which the specific rotatory power may be quickly calculated; it is recommended as being useful for the control of the acid conversion. Curves are added showing the influence of temperature, of $\frac{1}{50}$N sulphurous-, $\frac{1}{2}$N acetic-, $\frac{1}{25}$N oxalic-, $\frac{1}{50}$N sulphuric-, and hydrochloric acid; of the concentration or amount of acid used; and of the relative curves due to temperature.

—J. L. B.

Wine, Determination of Lactic and Succinic Acids in. J. A. Muller. Bull. Soc. Chim. 1896, **15**, [23], 1203—1206.

In the determination of lactic and succinic acids in wine, these two acids may be separated by titration with barium hydrate and treatment of the resulting barium salts with alcohol, in which the barium lactate alone dissolves. From the barium found in the solution and in the residue, the amounts of each of the two acids may be calculated. The results found, however, do not agree with the quantities originally present in the wine; and, as the result of three experiments, the mean ratio of lactic acid present to that found is as 1·5:1, whilst, in the case of succinic acid, the mean ratio is 1·2:1. With regard to the lactic acid, the difference is attributed mainly to the volatilisation of the acid, which is found to take place (see following abstract) during the desiccation *in vacuo* of the residue from the evaporation of the wine.—A. K. M.

Lactic Acid and its Anhydrides, Volatilisation of, at the Ordinary Temperature, and the Volatilisation of Lactic Acid by Steam. J. A. Muller. Bull. Soc. Chim. 1896, **15**, [23], 1206—1210.

When a solution of lactic acid is evaporated over sulphuric acid and the residue exposed for some days in a vacuum, an appreciable quantity is lost by volatilisation. Thus, in one experiment, 0·1280 grm. of lactic acid lost 0·0112 grm. in four days, and, in a second experiment, the loss was 0·0217 grm. in 11 days (the amount of acid in each case being determined by hot titration). In another series of experiments an ethereal solution of lactic acid was employed; the volume of solution taken contained 0·0941 grm. (determined by hot titration with the help of barium hydrated solution), and, after 18 days' exposure in a vacuum, this lost 0·0162 grm. Lactic anhydride, on the other hand, is almost non-volatile under the same conditions, 0·1511 grm. losing only 0·0028 grm. in 25 days. Lactide is more volatile than the latter. When a solution of lactic acid is distilled, the amount of acid carried over with the aqueous vapour increases with the strength of the solution, but here again the amount volatilised is only small; thus 200 c.c. of solution, containing 9·348 grms. of acid, lost only 0·1583 grm. with 190 c.c. of distillate.

Although lactic acid is thus shown to be only slightly volatile under the conditions described, its volatility is

sufficient to account for the discrepancies occurring in wine analyses made by the method mentioned in the preceding abstract.—A. K. M.

Acetone, Volumetric Determination of. E. R. Squibb. J. Amer. Chem. Soc. 1896, **18**, 1068—1079.

After fully describing the method of Robineau and Rollin (Monit. Scient. 1893, 272; this Journal, 1893, 870), the author suggests certain modifications whereby the process may be rendered simpler and more rapid. The following solutions are prepared:—(1) Standard acetone solution containing 1 grm. acetone in 100 c.c.; this solution should be kept in a dark glass bottle. (2) Potassium iodide solution prepared by dissolving 250 grms. of the salt in one litre of water, and adding 850 c.c. of sodium hydrate solution, 257 grms. per litre. (3) Sodium hypochlorite, a suitable strength being that of the "liquor sodæ chloratæ" of the U.S. Pharmacopœia, which contains 2·6 per cent. of available chlorine. To each litre is added 25 c.c. of sodium hydrate solution, 257 grms. per litre. (4) Starch solution, 0·125 grm. of starch is mixed with 5 c.c. of cold water, and then added to 20 c.c. of boiling water and boiled; when cold, 2 grms. of sodium bicarbonate are added and dissolved with stirring.

Titration.—10 c.c. of the standard acetone solution and 20 c.c. of the alkaline iodide solution are added together and well stirred. The hypochlorite is added from a burette, iodoform being precipitated, and the end of the reaction is indicated by a blue colour being given with starch; the exact details of working are as given in the paper of Robineau and Rollin (reference above). The hypochlorite equivalent of 0·1 grm. of acetone is thus found, and from this the percentage of acetone in a solution of unknown strength can be rapidly estimated; for this purpose from 0·1 to 0·4 c.c. of the solution should be taken, according to its approximate strength. This is weighed, diluted, and titrated as above. Since the hypochlorite solution diminishes in strength on keeping, it should be frequently standardised by means of the standard acetone solution.

The author confirms the statement of Robineau and Rollin, that the presence of ethyl alcohol does not affect the titration of acetone by this method.—R. B. B.

Sandal-wood Oil. A. J. Hendrix. J. Pharm. Chim. 1896, **4**, 499.

A good sample, according to Cripps, should be colourless or slightly yellow, have a thick consistence, a strongly aromatic odour, a piquant, spicy taste, and a neutral or slightly acid reaction. Schimmel and Umney agree that the density should not be less than 0·975 at 15°, and that the oil should dissolve in 5 parts of alcohol of 70 per cent. by volume; this test detects adulteration with 5 per cent. of the following essences: cedar-wood, West Indian sandal-wood oil, santal-macassar, turpentine, copaiba, and castor oil. Pure sandal-wood oil which is old, or has been badly preserved, may not, however, give a clear solution. The author proposes the following test, suitable for use with small quantities:—Weigh into a 10-c.c. flask 2 grms. of a solution of 3 parts of crystallised phenol in 1 part of alcohol, add 0·5 grm. of the oil, and mix perfectly. Add 0·5 grm. of concentrated hydrochloric acid without shaking. At the intersection of the liquids there is formed with pure sandal-wood oil a yellow coloration, changing in a few minutes to a bright red. With essence of balsam of copaiba, the upper liquid becomes mauve-coloured after a few minutes. With oil of cedar-wood, the upper liquid becomes cloudy, and a brownish coloration is developed at the intersection. East Indian sandal-wood oil is levo-rotatory, − 16° to − 20° (Schimmel and Umney), whilst oil of cedar is much more levo-rotatory, and sandal-wood oils of the West Indies and Australia are dextro-rotatory.

Parry recommends determination of the alcohol, santalol, by boiling with acetic anhydride with a little sodium acetate for 1½ hours, washing the acetate, drying, and saponifying. A good oil contains at least 90 per cent. of santalol; cedar oil contains only 15 per cent., and balsam of copaiba 7 per cent. of alcohol.—A. C. W.

Tartar Estimation, A Modification of Goldenberg's Method for. M. Zecchin. Statz. Sper. Ag. Ital. **28**, 788; and Analyst, 1896, **21**, 333.

Weigh out 3·75 grms. of the finely powdered substance, and dissolve in hydrochloric acid on the water-bath; after 10—15 mins. make up to 100 c.c. and filter off 50 c.c. Add solid potassium carbonate in very slight excess, boil for some minutes, and make up to 100 c.c. Filter off 20 c.c., add 5 c.c. of glacial acetic acid and 100 c.c. of a mixture of equal parts of alcohol and ether, allowing the solution to stand in a closed flask for four or five hours. Filter, and wash the precipitate with the ether-alcohol mixture till neutral, dissolve in boiling water, and titrate with $\frac{1}{20}$ N alkali, using phenolphthaleïn as indicator. Each c.c. indicates 2 per cent. of tartaric acid. (See Analyst, **21**, 76.)—A. S.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Chrysoïdine, Behaviour of, towards Cholera Bacilli. A. Blachstein. Münchener Med. Wochenschr. 1896, **43**, 1067.

Chrysoïdine quantitatively precipitates cholera bacilli from solutions in which they are held in suspension, the precipitate being largely flocculent in character; it thus behaves like "*cholera-immun-serum*," and may be regarded as an artificial cholera-agglutinant. Other azo-dyestuffs, even those so nearly related to chrysoïdine as its sulphonic acid or Vesuvine, do not possess this property; and the reaction appears to be specific for the cholera bacilli, for none of the other similar vibriones examined gave a like result. It is, however, a good disinfectant for the whole group, occupying in this respect a position half-way between mercuric chloride and phenol. It is not in the least poisonous, a property which is in favour of its finding practical use. Tried as a remedy for cholera, it has given negative results, but it may yet prove to have some prophylactic value. It could certainly be used for the disinfection of large volumes of water, or of the mouth, œsophagus, or stomach. In the dissolved form, it was found not to penetrate to the intestinal canal, but to be separated by the kidneys.—W. G. M.

Cotton-Wool, Absence of Pentose Derivatives in. H. Suringar and B. Tollens. Zeits. f. angew. Chem. 1897, **4**.

To test the statement by Link and Voswinkel (Pharm. Centr. 1893, 253), that wood gum (a derivative of pentose) is formed in the hydrolysis of cotton, pure cotton wool was heated to the temperature of boiling water, with 4 per cent. sulphuric acid, for eight hours. After removal of the acid, by means of calcium carbonate, a small quantity of a syrup was obtained from the liquid. The reactions of this syrup showed that in all probability it consisted mainly of dextrose. Purified cotton-wool, therefore, contains practically no wood gum, the small quantity of sugar obtained, belonging to the glucose series, and being probably formed by the hydrolysis of the cellulose itself.—T. E.

Antimonio-tungstates. L. A. Hallopeau. Comptes rend. 1896, **123**, [24], 1065—1068.

Hydrated antimonic acid can combine with tungstic acid to form antimonio-tungstic compounds analogous to phospho-tungstic and arsenio-tungstic compounds. Details of methods of preparation are given.—J. T. D.

Metallic Sulphides [Sulphides of As, Sb, and Sn], The Action of Acid Vapours on. J. Kelley, jun., and E. F. Smith. J. Amer. Chem. Soc. 1896, **18**, 1096—1098.

Arsenic and antimony trisulphides, and also stannic sulphide, are completely volatilised when exposed to the action of hydrochloric or hydrobromic acid gas. The action commences in the cold with formation of a liquid that passes out of the containing vessel upon the application of a very gentle heat.

If stannous sulphide be subjected to the action of hydrochloric acid gas in the cold, it is completely converted into the chloride without volatilisation; but it is not possible to separate stannous from stannic sulphide by this means, since the heat required to completely volatilise the latter

would also cause partial volatilisation of the stannous chloride.—R. R. B.

Platinum, The Action of Phosphorus on. A. Granger. Comptes rend. 1896, **123**, 1284.

It is well known that platinum crucibles in which substances capable of liberating phosphorus are heated, are liable to be pierced by the formation of a fusible phosphide; but, nevertheless, it has been found difficult to obtain the phosphides of definite composition. Two porcelain boats, one containing platinum and the other red phosphorus, were gently heated in a glass tube through which carbon dioxide was passed, the temperature being slowly raised until most, but not all, of the phosphorus was distilled. The phosphide thus obtained was dark grey, and friable, but showed no definite crystals. With platinum wire or foil, the attack does not begin below a red heat; and at a still higher temperature phosphides poor in phosphorus are obtained, approaching in constitution to Pt_2P, whilst at a "red white" heat the phosphide retains only 4 per cent. of phosphorus. With spongy platinum the action begins below a red heat, and a biphosphide is formed. Contrary to Schrötter's statement, the author did not find the compound to be completely soluble in aqua regia, the dissolved portion exactly corresponding to Pt_3P_5.—E. S.

Yeast, Consumption of [Organic] Acids by. J. Schukow. Centralbl. Bakteriol. 1896, **2**, 601.

The experiments were conducted in flasks, which were weighed from time to time, in order to trace the progress of fermentation; and at stated intervals measured samples were withdrawn for titration by $\frac{1}{10}$ N caustic soda solution. Various nutrient solutions, mixed with different organic acids, were thus inoculated with pure cultures of yeast. Of the four acids named, citric acid was the most readily consumed, then malic acid, much less tartaric, and only to a slight extent succinic acid. Under like circumstances, different cultures of yeast give different quantitative results with each acid. Usually the consumption is greater when the yeast is well provided with nitrogenous food.—W. G. M.

Soluble Oxidising Ferments and Drugs. E. Bourquelot. J. Pharm. Chim. 1896, **4**, 481.

Various aromatic phenols and amines may be distinguished by means of oxidising ferments (from mushrooms) (see Bourquelot, this Journal, 1896, 683, 748; Bertrand, 1896, 561). Guaiacol carbonate is not directly oxidised by the ferment, but if the solution be warmed with a few drops of dilute caustic soda, and then slightly acidified by acetic acid, the yellow coloration characteristic of guaiacol is produced on addition of the ferment. Of the natural alkaloids, morphine is oxidised by the ferment in dilute alcoholic solution, giving a yellowish-white precipitate. In one experiment, using 0·3 grm. of alkaloid, a diminution of 19 c.c. in the volume of air contained in the vessel was noticed.

A weak alcoholic solution of the gum resin of asafœtida gives at first a rose coloration, then a yellow precipitate; galbanum and gum ammoniac, brownish precipitates; podophyllum resin in dilute alcohol, a browish-yellow coloration; the creosote of pharmacy, since it contains both cresol and guaiacol, a reddish-yellow precipitate; wood-tar water, a yellowish precipitate; coal-tar water, a brown, almost black, coloration.

Many vegetable substances, when fresh, contain oxidising ferments which are destroyed on drying; the dry leaves of chicory, the dry roots of dandelion, chicory, marsh-mallow, &c., will, however, contain a ferment which can be extracted by trituration with sand and water. Gum-arabic and the gum of the apricot tree also contain an oxidising ferment. The kola nut probably contains a tannin united to caffeïne and glucose (kolanine); this compound is decomposed by the oxidising ferment in the kola, the tannin forming a coloured compound. It is only after this oxidation has taken place that caffeïne can be extracted by chloroform. The oxidation takes place slowly in the absence of the ferment, rapidly in its presence. This explains the variations in determinations of caffeïne (see this Journal, 1896, 670).—A. C. W.

Educational.

Chemical Research. Circular issued by City and Guilds of London Institute.

Higher research in chemistry in its relation to manufactures is to be further encouraged by the foundation of one or more Fellowships, tenable at the City and Guilds of London Institute. The Leathersellers' Company is providing the necessary funds, a grant of 150*l*. a year being offered for the purpose. The amount of the grant attached to each Fellowship will be determined by the Executive Committee of the Institute, regard being had as far as practicable to the nature of the research, the time required to complete it, and the merits of the candidate, subject in all things to the approval of the Company. The Fellowships are confined to natural-born British subjects, who have completed a full three years' course of instruction in the Chemical Department of the Central Technical College, or who are otherwise duly qualified in the methods of chemical research in its relation to manufactures. They will be tenable for part of a year or a whole year, and may be renewed for a second or third year, all researches by Fellows being carried out at the Central Technical College. Applications for Fellowships must be addressed to the Honorary Secretary of the Institute, Gresham College, E.C.

The Teaching of Chemistry. The Times, Feb. 5, 1897.

About two years ago *The Times* published a letter by Prof. Armstrong, in which attention was directed to the very unsatisfactory character of the instruction in chemistry that was being given, under the auspices of the Technical Education Board of the London County Council, to science teachers in schools. The great danger they incurred in setting such an example was dwelt on, and the more desirable direction for the Board to follow was pointed out. The letter had some effect, for after a time a sub-committee was appointed to consider the teaching of chemistry in schools and evening classes. This committee consisted of six members of the Board, besides Dr. Russell, F.R.S. (chairman), Dr. Ludwig Mond, F.R.S., and Prof. Ramsay, F.R.S.—three chemists well known not only in the scientific world—and Mr. C. M. Stuart, head master of St. Dunstan's College, Catford. As Sir Philip Magnus and Prof. James Stuart, both members of the Board, were on the committee, and Dr. Garnett, the Secretary to the Board, and also Dr. Kimmins acted as its advisers, there was no lack of educational experience; indeed, the committee was an exceptionally strong one. A report signed by the chairman and secretary of the Technical Education Board, embodying the recommendations of this sub-committee, has just been issued. This report, it may safely be said, is destined to mark an era in the "technical" education movement in this country, both because of the example its issue sets to county councils generally, and more particularly on account of the recommendations which are put forward in it. Of all the work hitherto done by the Board, probably none is of more fundamental importance, as it will give help in a direction in which help has long been sorely needed and of which there was little prospect or hope, and, coming from such a source, the report is a peculiarly valuable contribution to the inquiry which is now being actively prosecuted into methods of education with the object of ascertaining which are the most likely to promote technical progress.

Extracts from the evidence taken by the committee are given in the report. That of Dr. Mond is of the highest value. He gave the opinion that the teaching of technological chemistry in schools or similar institutions was of no value whatever either for chemical industries or for the pupils. He advised that the Board should confine its energies to the teaching of scientific chemistry as an educational discipline, and that special knowledge in chemistry could only be imparted in institutions of university rank, where the student was bound to devote years to thorough study under the guidance of eminent teachers.

Dr. Messel, another German, who is an English manufacture and also a highly-trained scientific chemist, gave similar evidence. According to this witness, chemical industry on the Continent is ahead of that in this country, because science is accorded a consulting and deliberating

voice abroad, and because there are mostly found at the head of affairs people who possess a thorough scientific education. Only a complete training is of value for industrial purposes. To keep abreast of other nations English industries require the services of thoroughly trained men; it must be realised, this witness said, that what creates and maintains the industry of a country is neither capital nor labour, but brains.

Owing to their intimate knowledge of the conditions which prevail in German as well as in English works, the opinions of these two witnesses are invaluable. But Mr. Howard and others expressed similar views. Much evidence was taken by the Committee as to the character of the training most suitable for different classes of students, and it is on this subject that they make most important recommendations.

They begin their report with the statement:—"We are of opinion that the teaching of chemistry in schools should be solely of an educational nature, and should have no reference to practical (*i.e.*, technical) application." Nothing could be clearer, more emphatic, or more in accordance with the opinion of all who have real knowledge of the subject. Among the general conclusions arrived at, the following deserve special notice, as they enunciate educational doctrines of startling boldness and breadth in comparison with those now prevailing:—

That as a branch of elementary education chemistry, when properly taught, is a most effective means of cultivating power of observation and accuracy of thought, and consequently it should form a part of school work on account of its educational value and not because the facts it deals with may be of commercial importance. Further, we are of opinion that even this elementary teaching of chemistry should be largely carried out by means of practical work, the students being induced to form deductions from their own work and to solve problems by actual experiment.

That in carrying out the chemical teaching special attention should at the same time be paid to the writing, spelling, and clearness of expression in all notes and description of experiments, so that the teaching of these subjects may be to a considerable extent interwoven with the chemical teaching.

We are also of opinion that the Board may do great good by enabling scholars who have shown special aptitude for the study of elementary science, to continue such studies. We are, however, strongly of opinion that such a selection cannot be satisfactorily made by any ordinary system of examination, but could be made by the head master or some other officer of the school, who has personal knowledge of the work which has been done during the whole of the scholar's school career. We are much impressed by the evils arising from the too frequent examination of young students.

Passing from the teaching of chemistry simply as an element of education to the teaching of it with the special object of fitting a student for taking an important position in a chemical manufactory, we are convinced that the course of study must extend over several years and be of university standard; that the chemist in a manufactory, if he is to be efficient and able not only to cope with occasional and accidental difficulties, but also able to improve and extend the manufacture, must be a man who has not only had a thorough training in scientific chemistry but has also had experience in original research. Training of this character should be, and in fact is, given at certain colleges in this country, and should be attainable by those who can show that they have special aptitude and inclination for this kind of work. We desire to emphasize most strongly this need of the highest chemical instruction for the technological chemist, and we believe that the employment of men thus educated in the chemical works of this country would prove of great advantage, not only to the manufacturer, but also to the country in general.

If the conclusions of the committee are in any way accepted and carried out within the jurisdiction of the Council, there is little doubt that nothing short of a revolution will be effected, and that science will at last become of some use as an element in our system of education. The Technical Education Board have now a great opportunity, but no light task, and if they are wise their first step will be to appoint an educational council to act as their advisers from among those who are acknowledged leaders in such work in London. Teachers must be trained—for there are very few available at present; and examinations must, as far as possible, be put an end to. It is too terrible that 81·6 per cent.—so the report informs us—of the students attending evening classes at a number of the chief polytechnics and institutes in the London district should be preparing for the examinations of the Science and Art Department, knowing as we do the worthlessness of these as tests of ability to do anything rational or useful. It is to be hoped that the Board will take action to encourage the following of an example such as that set by the Finsbury Technical College, which alone of all the institutions where evening classes are held pays no heed to examinations. And, having now formally entered on the crusade against examinations, the Board are bound to look at home and see that those they impose are carried out as efficiently as possible, and so as to do the *minimum* of harm; for they cannot do good, necessary as they may be.

Among the recommendations made under the head of instruction of university standard during the day, there is one which is of extreme importance at the present juncture in view of the possible establishment of a teaching university in London—*viz.*, the following:—We cannot refrain from observing that the higher technical instruction in chemistry in London would probably be rendered more efficient and accessible if the work of the several institutions in this subject were better co-ordinated.

The Board are to be highly congratulated on the statesmanlike attitude assumed throughout by their sub-committee, and it is to be hoped that they will spare no effort to carry out the recommendations of the report which they have so wisely accepted.

Trade Report.

OFFICIAL NOTICE.

Carbide of Calcium.

In consequence of the growing importance of carbide of calcium and the fact that the mere contact of moisture with this material causes a dangerous evolution of the highly inflammable gas known as acetylene, the Home Secretary has caused inquiries to be made into the subject, with the result that an Order in Council has to-day been made under the 14th section of the Petroleum Act, 1871, bringing carbide of calcium within the operation of that Act.

Accordingly, from the date on which such Order comes into force, *viz.*, 1st April 1897, it will be unlawful to keep carbide of calcium except in virtue of a license to be obtained from the local authority under the Petroleum Act.

Any local authority to whom application may be made for a license to keep carbide of calcium can, if it so desires, obtain, on application to the Home Office, a memorandum showing the character of the risks to be guarded against, and containing suggestions as to the nature of the precautions likely to be most effectual for securing safety.

Whitehall,
26th February 1897.

PROPOSED LEGISLATION.

Steam Engines and Boilers (Persons in Charge).

A Bill to grant Certificates to Persons in Charge of Steam Engines and Boilers.

Be it enacted by the Queen's most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal, and Commons, in this present Parliament assembled, and by the authority of the same, as follows:—

1. This Act may be cited as the Steam Engines and Boilers Act, 1897.

2. This Act shall come into operation on the 1st day of January 1898, which date is in this Act referred to as the commencement of this Act.

3. In this Act—

The term "boiler" means any closed vessel used for generating steam, or for heating water, or for heating

other liquids, or into which steam is admitted for heating, steaming, boiling, or other similar purposes;

The term "engine" means any engine used in the production or motive power, and worked by steam from a boiler;

The term "horse-power" means nominal horse-power.

4. This Act does not apply to any boiler or engine used exclusively for domestic, agricultural, or farming purposes, or to any boiler or engine used in the service of Her Majesty, or to any boiler or engine used by a railway company, or to any boiler or engine used on board a steamship having a certificate from the Board of Trade, or to any road traction engine or steam roller.

5. After the commencement of this Act it shall not be lawful for any person to take the charge or control of, or to employ another person to take the charge or control of, any boiler or engine to which this Act applies, unless the person so taking charge or control is qualified as required by this Act.

6. The qualifications required by this Act are as follows:—

(i.) A person taking charge or control of any boiler or engine to which this Act applies, of five horse-power or upwards, or of any engine to which this Act applies, used for winding workmen or minerals up or down the shaft of a mine, must hold a first-class certificate or a special certificate of service under this Act.

(ii.) A person taking charge or control of any other boiler or engine to which this Act applies must be the holder either of such a certificate as aforesaid, or of a second-class certificate under this Act.

Provided that in the case of any works or mine where there are more than one boiler or engine to which this Act applies, it shall be sufficient if they are under the constant supervision of one person possessing the qualification required in respect of them by this Act: so, however, that no one person shall undertake the supervision of a greater number of boilers or engines, or of boilers or engines situate at a greater distance from one another, than may be prescribed by any regulations of the Board of Trade for the time being in force, and the Board may make such regulations accordingly.

7. Any person who takes charge or control (save in case of a sudden emergency) of a boiler or engine to which this Act applies, without possessing the qualification required by this Act, shall be liable to a fine not exceeding for the first offence 40*s.*, and for the second or any subsequent offence 10*l.*

Any person who employs another person to take charge or control (save in case of a sudden emergency) of a boiler or engine to which this Act applies, without possessing the qualification required by this Act, shall be liable to a fine not exceeding for the first offence 10*l.*, and for the second or any subsequent offence 50*l.*

8. A person who has passed an examination held under this Act shall be entitled to receive a first-class certificate from a Secretary of State.

A person who within three months before or after the commencement of this Act produces to a Secretary of State satisfactory evidence that for not less than 12 months before the commencement of this Act he has within the previous five years had the practical charge or control of a boiler or engine of five horse-power or upwards shall be entitled to receive from a Secretary of State a special certificate of service, which for the purposes of this Act shall be equivalent to a first-class certificate.

A person who produces to a Secretary of State testimonials from at least two persons, which satisfy the Secretary of State that he is, in point of character, knowledge, physical ability, and experience, fit to receive a second-class certificate under this Act, shall, on so satisfying the Secretary of State, be entitled to receive a second-class certificate.

9. An applicant for a certificate under this Act shall pay to a Secretary of State such fee as may be from time to time prescribed by a Secretary of State, not exceeding, in the case of a first-class certificate, the sum of 5*s.*, and in the case of any other certificate, 2*s.* 6*d.*

10.—(1.) All certificates shall be made in duplicate, one part to be delivered to the person entitled to the certificate and one to be preserved.

(2.) Such last-mentioned part of the certificate shall be preserved, and a record of certificates and of the suspending, cancelling, or altering of the certificates, and of any other matter affecting them, shall be kept in such manner as a Secretary of State directs.

(3.) Any such certificate and any record under this section shall be admissible in evidence.

11. If the holder of a certificate under this Act proves to the satisfaction of a Secretary of State that he has, without fault on his part, lost or been deprived of a certificate already granted to him, the Secretary of State shall, and in any other case may, on payment of such fee (if any) as he directs, cause a copy of the certificate to which by the record kept in pursuance of this Act he appears to be entitled, to be certified by the person directed to keep the record, and to be delivered to him; and a copy purporting to be so certified shall have all the effect of the original.

12. If any person—

(*a*) forges or fraudulently alters, or assists in forging or fraudulently altering, or procures to be forged or fraudulently altered, any certificate, or an official copy of any such certificate; or

(*b*) makes, assists in making, or procures to be made, any false representation for the purpose of procuring, either for himself or for any other person, a certificate; or

(*c*) fraudulently uses a certificate or copy of a certificate which has been forged, altered, cancelled, or suspended, or to which he is not entitled; or

(*d*) fraudulently lends his certificate, or allows it to be used by any other person;

that person shall in respect of each offence be guilty of a misdemeanor.

13. A Secretary of State, on good cause shown, may cancel or suspend any certificate granted under this Act, and may at any time, on appeal being made to him by the person whose certificate has been suspended, and on production of evidence in support of his appeal, restore any cancelled or suspended certificate, with or without a statement to that effect appended thereto.

14. A first or second class certificate granted to an engineer under the Merchant Shipping Act, 1894, shall for the purposes of this Act be deemed to be equivalent to a first-class certificate granted under this Act.

15. In the year commencing with the 1st day of January 1898, and in every succeeding year, a Secretary of State shall cause to be held, as often as he deems necessary, examinations for the purpose of testing the qualifications of candidates for first-class certificates under this Act.

The Secretary of State shall have the entire management and control of all such examinations, and shall have power from time to time to make regulations with respect to all or any of the following matters; (that is to say,)

(*a.*) With respect to the subjects for and the mode of conducting the examination of candidates; and

(*b.*) With respect to the times and places of examinations, and the notices of examinations; and

(*c.*) With respect to the evidence to be given by applicants of physical ability, experience, and character; and

(*d.*) With respect to the appointment and removal of examiners, and with respect to the remuneration by fees or otherwise of the examiners so appointed; and

(*e.*) With respect to any other matter or thing as to which a Secretary of State thinks it expedient to make regulations for the purpose of carrying this section into execution.

Any regulation made under the authority of this section may be altered or revoked by a subsequent regulation.

16. Any offence against this Act may be prosecuted, and any fine in respect thereof may be recovered, in manner provided by the Summary Jurisdiction Acts.

17. The expenses of a Secretary of State in carrying out the provisions of this Act shall be defrayed out of moneys provided by Parliament.

EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

The Anglo-Sicilian Sulphur Company.

Board of Trade J., Feb., 206.

A despatch from Her Majesty's Ambassador at Madrid encloses extracts from the "Italie," which states that the Anglo-Sicilian Sulphur Company has been successful in reviving the declining sulphur industry of Sicily.

The amount of sulphur exported during the months of October and November last from mines which have given in their adhesion to the Society was 60,820,046 kilos.—or, with the other mines, a total of 77,318,795 kilos.

The movement for the month of October 1896 was 43,000 tons, as compared with 22,300 tons for October 1895. The demand from abroad, especially from France and the United States, has been very remarkable.

GENERAL TRADE NOTES.

Phosphates in the United States.

Eng. and Mining J., Jan. 30, 1897, 109.

The Southern phosphate miners did not have a prosperous year in 1896, and there was a large decrease in production. The figures as collected by the *American Fertiliser* show a movement for the year as follows, in tons:—

	1895.	1896.		
	Total.	Foreign.	Domestic.	Total.
South Carolina..	515,734	82,260	192,800	275,060
Florida	530,356	395,171	103,894	499,065
Tennessee	45,329	..	42,911	42,911
North Carolina .	7,500	..	7,418	7,418
Total ...	1,098,519	477,431	347,023	824,454

The total decrease from 1895 was therefore 274,065 tons, or 25 per cent. The South Carolina mines showed the greatest decrease—about 46 per cent.—the reduction in Florida being comparatively slight. The new mines in Tennessee did not add anything to the total for the year, though their shipments will probably be felt in 1897.

A good reason for the decrease in shipments is found in the low prices obtained. Phosphate rock, 58 to 60 per cent., sold in Charleston from 3·25 dols. per ton down to 3 dols., while river rock sold down to 2·85 dols., the closing prices being the lowest of the year. The foreign demand continued to decrease, owing to the competition of the Algerian phosphates and the growing use of basic slag as a source of phosphoric acid; while there was hardly any increase in the domestic demand, to make up for the diminished exports. At the prices noted there can be very little, if any, profit in mining phosphate.

Chromic Iron, with Reference to its Occurrence in Canada.

W. Glenn. From 17*th Annual Report of the United States Geological Survey,* 1895–6.

Chromite, nominally $FeO.Cr_2O_3$ with 67·89 per cent. Cr_2O_3, usually has some of each constituent metal replaced by Al_2O_3, MgO, CaO, and SiO_2, so that the greater proportion of chrome ore in the market contains 40—46 per cent. Cr_2O_3; the richest shipment in the experience of the author having averaged 54 per cent. Cr_2O_3. The following are typical analyses of chrome ore in bulk:—

	1.	2.	3.
	Per Cent.	Per Cent.	Per Cent.
SiO_2	7·00	5·22	6·44
Cr_2O_3	39·15	51·03	53·07
FeO	27·12	13·06	15·27
MgO........................	16·11	16·32	16·08
CaO	3·41	2·61	1·20
Al_2O_3	7·00	12·16	8·01

Of these, No. 1 is regarded as a very poor ore, and as containing unusually little Al_2O_3, No. 2 is a rich, and No. 3, a very rich ore. The Canadian ores are hard and tough, with abundance of stones of large size, which bear transport and handling well; so that in this respect they are in marked contrast to the Asiatic ores, which are friable and contain many small lumps and crushed material, and especially so to one Turkish ore, that is yellowish-brown in colour, and so friable that it is generally delivered in the form of fine gravel or sand. Chromite occurs in serpentine, and the only commercially valuable chrome ore known to exist in Canada is found in the great serpentine field in the Coleraine district, 2,837 tons of the ore were shipped from the township of Coleraine in 1895, of which 2,342 tons were consigned to the United States, 441 tons to Great Britain, and 54 tons to Nova Scotia. Besides the above, 1,200 tons were lying at the mines or on the railway. Most of the ore shipped was of the highest quality and was required for the chemical works of Baltimore and Philadelphia. The ore employed in metallurgy may be of inferior grade, and most of the ore sent to England was of the third quality.—W. G. M.

Iron Ore in Germany, Production of.

J. Soc. of Arts, 1897, **45**, 157—158.

The total output of iron-ore in the German Customs Union in 1894 was 12,392,065 tons, of this Germany produced 8,433,784 tons, and Luxembourg 3,958,281. About the same amount was smelted, for imports and exports figure nearly alike—2,000,000 tons. From Spain came about 700,000 tons, from Sweden nearly 600,000, and the rest from Algeria, Elba, and Greece—all through the Dutch ports, while about 130,000 tons came from Holland and Belgium.

The production in Upper Silesia has fallen in two years from 723,000 tons to 618,000, and will not last many years, and in Saxony and Würtemberg it has practically ceased. In Bavaria and Thuringia a few mines yield about 220,000 tons; in Hanover the aggregate output is about 1,000,000 tons; in Westphalia 200,000 tons; in the Siegerland 1,500,000 tons; and in the Dill and Lahn districts 1,000,000 tons. The Lorraine and Luxembourg districts are much more important. In Luxembourg the output has risen from 911,000 tons in 1870 to 2,960,000 tons in 1894, while in Lorraine the figures are:—1872, 677,000; 1880, 996,000; 1894, 3,922,000 tons. The Luxembourg ore, at the present rates of consumption, will last 135 years for the home consumption and 56 years for export, while the Lorraine beds are estimated to last 800 years.

The German freights, in spite of competition between rail and water, are much higher than those of France and Belgium: 1·96 pfennige per ton of ore per kilo., against about 1·2 pfennige.—J. T. D.

Spirit for Technical Purposes, Activity of the German Distillers' Association for the Promotion of the Employment of.

Delbrück. Zeits. f. Spiritusind. 1896, **19**, 409—410.

The printing of a statement made by Posadowsky in the Reichstag having given rise to some misunderstanding with regard to the activity of the German Distillers' Association, the writer gives the following particulars:—The Association has actively assisted in developing the employment of spirit for technical purposes, the amount of duty-free spirit used having increased from 38·7 million litres in 1887–88 to 71·9 millions in 1894–95. Application was made to the Secretary of the Imperial Treasury for funds to establish an experimental institution for investigation in the following subjects:—(1) Incandescent spirit lamp; (2) Apparatus for heating with spirit; (3) Spirit motors; (4) Manufacture of alcohol and vinegar; and (5) Alcoholic preparations, lacks, and varnishes. The cost of fitting up such an establishment was estimated at 15,000 marks, and the yearly expenditure at 14,000 marks. The amount granted by the Secretary of the Treasury was 10,000 marks, and with the help of this sum the Association (*a*) published a collective account of all the technical literature bearing upon the incandescent spirit lamp, (*b*) offered prizes for

the construction of an incandescent spirit lamp with an illuminating power equal to that of the ordinary petroleum lamps. Twenty lamps were tested, and the best of these gave the following results :—

—	Intensity of Light, in Candles.	Cost of Spirit per Hour, in Pfennige.
Incandescent spirit lamp ...	22·5 12·0	1·5 1·2
		Cost of Petroleum, per Hour.
Large petroleum lamp	24·0	2·0
Small petroleum lamp	11·0	0·9

The question is therefore solved for the well-to-do public. (c) The Association has brought about a reduction in the price of burning spirit by getting the licence removed, the cost of spirit being reduced in many places to 25 pfennige (about 3*d.*) or less per litre. The employment of spirit for heating and cooking purposes is also being developed, and the engineer of the Association is likewise engaged on the motor question. An experimental station for the alcohol and vinegar industry was to be opened on Jan. 1, 1897.

—A. K. M.

BOARD OF TRADE RETURNS.

Summary of Imports.

Articles.	Month ending 31st January.	
	1896.	1897.
	£	£
Metals	1,681,443	1,578,012
Chemicals and dyestuffs	816,472	572,947
Oils	823,163	602,376
Raw materials for non-textile industries	3,189,712	3,239,851
Total value of all imports	38,473,856	39,975,668

Summary of Exports.

Articles.	Month ending 31st January.	
	1896.	1897.
	£	£
Metals (other than machinery)	2,558,834	2,535,069
Chemicals and medicines	761,859	727,912
Miscellaneous articles	2,813,085	2,516,777
Total value of all exports.....	21,146,491	19,762,378

Imports of Oils for Month ending 31st January.

Articles.		Quantities.		Values.	
		1896.	1897.	1896.	1897.
				£	£
Cocoa-nut	Cwt.	20,061	26,527	21,540	29,669
Olive	Tuns	1,646	1,010	54,182	33,339
Palm	Cwt.	103,736	60,063	108,978	62,953
Petroleum	Gall.	17,176,225	13,240,569	363,655	259,035
Seed	Tons	3,438	2,489	69,646	56,092
Train, &c.	Tuns	927	318	16,450	5,688
Turpentine	Cwt.	69,362	40,114	79,096	38,146
Other articles ..	Value £	..	..	109,618	117,454
Total value of oils...		..	..	823,163	602,376

Imports of Chemicals and Dyestuffs for Month ending 31st January.

Articles.		Quantities.		Values.	
		1896.	1897.	1896.	1897.
				£	£
Alkali	Cwt.	8,977	25,237	4,843	9,819
Bark (tanners', &c.)	„	16,640	17,201	5,993	5,869
Brimstone	„	35,857	28,192	6,577	6,492
Chemicals	Value £	..	..	116,523	97,674
Cochineal	Cwt.	533	227	3,515	1,400
Cutch and gambier	Tons	2,645	2,057	57,150	37,098
Dyes:—					
Alizarin	Value £	..	..	20,417	11,735
Anilin and other	„	..	..	41,920	40,134
Indigo	Cwt.	19,151	11,438	336,752	211,495
Nitrate of potash	„	19,564	28,666	19,137	22,260
Valonia	Tons	4,442	2,453	49,868	26,254
Other articles...	Value £	..	..	153,777	102,417
Total value of chemicals		..	..	816,472	572,947

Imports of Metals for Month ending 31st January.

Articles.		Quantities.		Values.	
		1896.	1897.	1896.	1897.
Copper:—				£	£
Ore	Tons	4,402	2,150	30,072	19,548
Regulus	„	9,420	5,130	264,206	148,525
Unwrought	„	4,805	5,148	206,516	257,460
Iron:—					
Ore	„	469,657	463,810	331,353	346,824
Bolt, bar, &c. ...	„	4,934	3,564	41,139	27,057
Steel, unwrought..	„	1,136	4,115	9,293	24,862
Lead, pig and sheet	„	11,158	14,034	121,986	161,254
Pyrites	„	54,487	45,956	90,565	75,705
Quicksilver	Lb.	8,910	18,275	1,113	1,925
Silver ore	Value £	..	..	132,761	106,184
Tin	Cwt.	64,562	46,510	190,168	137,217
Zinc	Tons	6,208	4,980	92,280	82,950
Other articles ...	Value £	..	..	160,991	188,501
Total value of metals		..	..	1,681,443	1,578,012

Imports of Raw Material for Non-Textile Industries for Month ending 31st January.

Articles.		Quantities.		Values.	
		1896.	1897.	1896.	1897.
				£	£
Bark, Peruvian ..	Cwt.	3,684	1,837	6,531	2,253
Bristles	Lb.	429,783	330,955	52,090	45,873
Caoutchouc	Cwt.	39,904	44,616	480,811	561,539
Gum:—					
Arabic	„	5,228	4,410	14,522	9,537
Lac, &c.	„	14,971	16,927	77,881	90,790
Gutta-percha	„	3,756	641	31,413	5,856
Hides, raw:—					
Dry	„	28,305	34,614	70,770	75,593
Wet	„	58,199	54,345	124,057	109,660
Ivory	„	794	811	40,062	34,464
Manure:—					
Guano	Tons	870	1,510	3,680	10,755
Bones	„	10,713	7,816	43,737	32,533
Nitrate of soda...	„	7,864	7,784	60,148	62,142
Phosphate of lime	„	21,634	20,313	44,895	32,644
Paraffin	Cwt.	67,783	62,679	71,854	60,399
Linen rags	Tons	1,748	2,848	17,876	26,857
Esparto	„	14,481	19,271	63,230	78,763
Pulp of wood	„	21,170	38,035	115,757	179,663
Rosin	Cwt.	211,820	250,780	50,001	65,097
Tallow and stearin	„	206,994	189,386	234,301	188,927
Tar	Barrels	901	555	392	237
Wood:—					
Hewn	Loads	126,475	147,880	248,871	271,673
Sawn	„	147,747	138,597	339,532	349,486
Staves	„	10,190	7,951	67,178	59,444
Mahogany	Tons	3,774	3,248	31,795	28,650
Other articles....	Value £	..	..	899,428	857,288
Total value		..	..	3,189,712	3,239,851

Besides the above, drugs to the value of 85,514*l*. were imported as against 60,454*l*. in January 1896.

Exports of Metals (other than Machinery) for Month ending 31st January.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Brass............ Cwt.	10,031	9,909	41,431	42,664
Copper:—				
Unwrought.... „	39,222	25,675	92,136	66,248
Wrought...... „	41,560	20,205	111,476	63,089
Mixed metal... „	23,366	10,011	51,021	25,314
Hardware...... Value £	..	..	181,519	181,325
Implements..... „	..	..	117.095	118,058
Iron and steel... Tons	227,607	246,996	1,716,072	1,804,496
Lead............ „	3,307	3,338	44,568	42,918
Plated wares ... Value £	..	..	30,380	29,733
Telegraph wires . „	..	..	57.224	62,120
Tin .... Cwt.	10,255	6,869	33,293	21,498
Zinc............ „	19,272	15,181	12 347	11,363
Other articles .. Value £	..	..	70,272	66,252
Total value	..	..	2,558,834	2,535,069

Exports of Drugs and Chemicals for Month ending 31st January.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Alkali........... Cwt.	69,467	327,804	111,193	82,489
Bleaching materials „	116,479	94,103	42,194	30,565
Chemical manures Tons	29,685	32,846	201,025	185,514
Medicines....... Value £	..	..	102,590	83,648
Other articles... „	..	..	304,857	345,696
Total value	..	..	761,859	727,912

Exports of Miscellaneous Articles for Month ending 31st January.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Gunpowder...... Lb.	649,800	1,060,900	16,064	23,273
Military stores.. Value £	..	..	158,692	64,997
Candles.......... Lb.	2,539,900	1,873,200	40,443	26,622
Caoutchouc..... Value £	..	..	116,711	97,235
Cement.......... Tons	20,612	27,645	33,678	44,485
Products of coal Value £	..	..	177,795	157,185
Earthenware ... „	..	..	158,660	111,483
Stoneware...... „	..	..	12,798	13,552
Glass:—				
Plate........... Sq. Ft.	158,613	147,651	8,508	9,391
Flint........... Cwt.	9,417	6,723	20,630	16,776
Bottles......... „	66,809	65,792	31,265	30,855
Other kinds.... „	23,675	18,087	19,247	14,092
Leather:—				
Unwrought .... „	10,880	10,371	105,660	97,457
Wrought ..... Value £	..	..	29,401	28,816
Seed oil.......... Tons	4,007	4,451	79,755	78,478
Floorcloth ..... Sq. Yds.	2,566,800	1,886,000	99,403	80,550
Painters' materials Val. £	..	..	132,390	119,737
Paper .... Cwt.	95,786	73,170	145,114	119,224
Rags............. Tons	4,269	4,706	25,667	22,506
Soap............. Cwt.	71,440	60,762	70,005	59,505
Total value	..	..	2,813,085	2,516,777

Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—PLANT, APPARATUS, AND MACHINERY.

Applications.

1459. J. Cleghorn and T. Wilkinson. Improvements in apparatus for crushing rock-salt, coke, or other substances. Jan. 19.

1494. S. Chatwood and S. R. Chatwood. Improvements in apparatus for superheating steam. Jan. 19.

1580. J. Thomas and L. P. Thomas. Improvements relating to presses suitable for the treatment of oil-cake and other materials. Complete Specification. Jan. 20.

1663. R. Wright. Improvements in apparatus for compressing air and other gases. Jan. 21.

1843. A. J. Boult.—From H. H. Dikema. Improved method of mixing or separating solid, liquid, or gaseous bodies in solution with or from other bodies, and apparatus therefor. Complete Specification. Jan. 23.

2618. L. Gunn. An improved refrigerating and cooling appliance. Feb. 1.

2876. J. B. Alliott. Improved method and apparatus for evaporating liquids and condensing. Feb. 3.

2877. J. B. Alliott. Improved methods of and apparatus for cooling and evaporating. Feb. 3.

2900. H. Hirzel. An improved column for condensing, absorbing, heating, distilling, and otherwise treating gases and liquids. Complete Specification. Feb. 3.

2927. E. T. Bousfield. Improvements in apparatus for heating or cooling liquids. Feb. 3.

2931. R. G. Brooke. Improvements in apparatus for purifying fluids. Feb. 3.

3027. Sir C. S. Forbes, Bart. Improvements in condensers. Feb. 4.

3219. G. Zschocke. Improvements in hurdles or grids for gas purifiers and cooling, drying, and graduating apparatus. Feb. 6.

3328. D. Halpin, J. B. Alliott, and L. Sterne. Improvements in cooling liquids and apparatus therefor. Feb. 8.

3482. J. B. Alliott. Improved evaporating process and apparatus applicable for concentrating, cooling, and other purposes. Feb. 9.

3483. J. B. Alliott. Improved methods of and apparatus for cooling and evaporating. Feb. 9.

3592. J. E. Campbell. *See* Class VII.

Complete Specifications Accepted.*

1896.

1300. C. Polony. *See* Class II.

2150. R. Cunliffe. Apparatus for drying brewers' refuse and other materials or substances requiring similar treatment. Feb. 3.

3365. H. B. McKenna. Treatment of hydrocarbons for the production of crystalline substances, and apparatus for treating and manufacturing same. Feb. 17.

4208. W. Griffiths and V. C. Read. Apparatus for treating feed water to prevent incrustation in boilers, applicable for treating or purifying water for other purposes. Feb. 17.

5431. S. M. Lillie. Evaporating apparatus. Jan. 27.

6210. S. T. Wellmann and G. H. Wellmann. Apparatus for charging metallurgical and other furnaces. Feb. 17.

9127. M. Douane. Apparatus for producing cold by means of volatile liquids. Feb. 17.

15,488. F. H. Haviland, A. Holloway, J. B. Collier, and W. H. Murch. New or improved furnace. Feb. 10.

20,087. J. von Grubinski. Apparatus for purifying and heating feed water for steam generators. Jan. 27.

20,716. A. Muller, jun. Drying kiln with sinking shelves. Feb. 17.

28,411. S. Straker. Apparatus for evaporating water containing salts and calcareous matter, and in heating furnaces for removing the incrustation from the coils of such evaporating apparatus. Feb. 3.

28,763. J. Y. Johnson.—From The Electric Rectifying and Refining Co. *See* Class XVII.

1897.

852. T. B. Lightfoot.—From the Gesellschaft für Linde's Eismaschinen. Apparatus for the production of ice and for cooling liquids by the expansion of compressed air. Feb. 17.

II.—FUEL, GAS, and LIGHT.

Applications.

1254. R. J. Moss. An improved apparatus for generating and purifying acetylene gas. Jan. 18.

1370. F. G. Bartlett. An improvement relating to incandescent gas-lighting. Jan 19.

1398. W. R. Addicks. Improvements in the manufacture of carburetted water-gas and in apparatus therefor. Complete Specification. Jan. 19.

1440. A. J. Boult.—From H. F. Fuller. Improvements in or relating to apparatus for the generation of gas. Complete Specification. Jan. 19.

1490. G. Baldwin. Improvements in apparatus for the manufacture of acetylene gas. Jan. 19.

1549. A Sterza. Apparatus for preparing or rendering acetylene gases fit for heating purposes. Complete Specification. Jan. 20.

1653. G. de R. de Sales. Process for the agglomeration of carbides. Complete Specification. Jan. 21.

1776. C. Pond and R. S. Richards. Improvements in the manufacture of calcium carbide and apparatus therefor. Jan. 22.

1777. L. Denayrouze. An improvement in burners for incandescent gas lights. Jan. 22.

1784. W. P. Thompson.—From A. Gobron. Improved portable apparatus for generating and burning acetylene gas. Jan. 22.

1879. A. Trosier. Apparatus for generating acetylene gas and regulating its production. Jan. 23.

1923. S. J. von Romocki and H. Ascher. Improvements in incandescent gas burners. Jan. 25.

1929. T. Thorp and T. G. Marsh. Improvements in and relating to apparatus for the production of acetylene from calcium carbide. Jan. 25.

1935. W. H. Chambers. Improvements in the manufacture of coke. Jan. 25.

1952. J. C. Mewburn.—From A. M. Scott. Improvements in apparatus for generating gas by the reaction of solid substances with water or other liquid, especially applicable to the generation of acetylene gas by contact of water with metallic carbides. Jan. 25.

2032. T. Lyons, J. Lyons, and J. Pearson. Improvements in acetylene gas store generators. Jan. 26.

2050. C. Kaestner. Burner for use with acetylene gas. Jan. 26.

2065. A. J. Boult.—From C. Benoit and A. Graviere. Improvements in or relating to apparatus for the production of acetylene gas. Jan. 26.

2071. J. Bowing. Improvements in the process of and apparatus used for coking. Jan. 26.

2113. J. Spurge. Improvements in gas-cleansing apparatus. Jan. 26.

2129. W. L. Wise.—From A. Piatti and Co. Improvements in the manufacture of gas. Jan. 26.

2176. W. J. Engledue. Improvements in or relating to the generation of heat from fuel. Jan. 27.

2194. P. W. von Gehlen. An apparatus for producing and using an incandescent light produced from benzene gas. Complete Specification. Jan. 27.

2260. F. S. Thorn and C. Hoddle. A new and improved acetylene jet for optical lantern use. Jan. 28.

2261. F. S. Thorn and C. Hoddle. A new and improved receptacle for safely storing calcium carbide. Jan. 28.

2262. F. S. Thorn and C. Hoddle. A new and improved lantern-view dissolver for acetylene gas. Jan. 28.

2284. R. F. Carter. Improved apparatus for the production and storage of acetylene gas. Complete Specification. Jan. 28.

2292. G. de R. de Sales. Apparatus for generating and storing acetylene. Complete Specification. Jan. 28.

2337. G. W. B. Crees. Improved means for and method of producing acetylene gas. Jan. 28.

2428. H. R. Bean and H. Ringwood. An improved generator condenser and holder for acetylene and other gases. Jan. 29.

2511. T. Holmes, G. S. Holmes, and R. W. Stewart. An improved process for the production of inflammable gas. Jan. 30.

2551. J. Gilligan. Improvements in mantles for incandescent lighting. Jan. 30.

2554. E. G. Villejean and H. J. J. Frossard. Apparatus for the automatic generation of acetylene gas. Jan. 30.

2574. J. Akroyd and B. Akroyd. (Excelsior) acetylene gas generator. Feb. 1.

2738. P. B. Watson. Apparatus for producing atmospheric gas from liquid hydrocarbons. Feb. 2.

2756. E. A. Javal and G. Cargill. Improvements in apparatus for the production of acetylene gas. Feb. 2.

2863. W. C. Tyler. Improvement relating to apparatus for the production of oxygen from the atmospheric air. Feb. 3.

2894. H. Maxim. An improved method of and apparatus for the production of calcium carbide, applicable also to other purposes. Feb. 3.

2945. T. Thorburn. An improved generator for the manufacture of acetylene gas. Feb. 4.

2976. W. H. Dennis. Acetylene gas lamps and generators. Complete Specification. Feb. 4.

2984. H. Walker. An improved process and apparatus for the production and purification of acetylene gas. Feb. 4.

3013. W. P. Thompson.—From P. P. H. Mace and L. L. H. Gerard. Improvements in apparatus for the production of acetylene gas. Feb. 4.

3029. W. C. Tyler. Improvement in apparatus for the production of oxygen from the atmospheric air. Feb. 4.

3060. J. B. Fenby. Improvements in apparatus for producing acetylene. Feb. 5.

3129. A. Freitag. Improvements relating to the burning of pulverulent fuel and to apparatus therefor. Feb. 5.

3504. J. Wilson. Improvements in and in the manufacture of burners for incandescent gas lighting. Feb. 10.

3539. A. Rose. Improvements in and connected with burners for incandescent gas lights. Feb. 10.

3667. H. A. Kent. Inverted regenerated incandescent gas burners or lights. Feb. 11.

3823. J. P. van der Ploeg. Improvement of the heating system applicable to most different furnaces, ovens, kilns, and other producers of heat by combustion of any combustible. Feb. 12.

3891. J. W. Leadbeater. An improved mode of admixing and preparing petroleum and other matter in compound form with a view to its final use as a combustible. Feb. 13.

3892. J. W. Leadbeater. Improvements in the manufacture of briquettes or blocks of fuel from waste products or otherwise enriched by petroleum compounds. Feb. 13.

3898. F. Richard. Improvements in the manufacture of calcium and other carbides. Feb. 13.

3975. E. L. Browne. An acetylene gas generator. Feb. 13.

3981. A. V. Cristiani and O. Scholzig. Improvements in and connected with apparatus for generating, purifying, cooling, storing, and directly supplying acetylene gas. Feb. 13.

Complete Specifications Accepted.

1895.

24,088. W. Synnock and G. Gosling. An apparatus for the production and automatic distribution of acetylene gas. Jan. 27.

1896.

1300. C. Polony. A process and furnace installation for continuous process of production of a non-exploding mechanical admixture of oxygen and hydrogen. Jan. 27.

4548. F. J. Stedman. Apparatus for the production of oxygen gas. Jan. 27.

5686. S. Hersey and Kirkham, Hulett, and Chandler, Ltd. Apparatus for washing, scrubbing, and purifying gas. Feb. 10.

6857. G. Hilgenstock. Coke ovens. Feb. 17.

7463. L. Denayrouze. Incandescent gas lighting. Feb. 17.

16,432. A. Schemidt. Production and purification of acetylene gas, and apparatus therefor; also connected with portable lighting apparatus for burning such sand. Feb. 3.

19,979. A. Rolf. Incandescent gas lights. Feb. 10.

20,501. H. Hill. Manufacture of thread foundations or caps for incandescent mantles. Feb. 17.

25,887. F. J. Collin. Coke furnaces. Feb. 17.

25,167. B. Puchmuller. Process for the manufacture of fluid for impregnating bodies or suitable fabrics, threads, fibres, or the like, for illuminating or lighting purposes. Jan. 27.

26,304. R. Van de Ghinste. Incandescence gas lighting. Feb. 3.

27,086. S. D. Gillet, G. Forest, and J. E. O. Bocande. Burner for lighting by acetylene or other gases rich in carbon. Feb. 3.

27,627. H. H. Lake.—From V. Hanotier and G. Hostelet. Acetylene gas generators. Feb. 3.

29,500. E. Quelle. Apparatus for generating acetylene gas. Feb. 17.

30,134. J. Schumacher. Apparatus for use in generating gas. Feb. 17.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Applications.

1934. J. E. Whiting. Improvements in the processes for vulcanisation of wood and other organic substances or products. Jan. 25.

2515. H. J. Dawes. Improvements relating to the distillation and breaking up of liquid hydrocarbons and similar substances. Jan. 30.

IV.—COLOURING MATTERS and DYES.

Applications.

1320. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of alpha-naphthylamine in an easily diazotisable and stable form. Jan. 18.

1575. H. Baum. Improvements in the manufacture of colouring matter of the rhodamine group. Jan. 20.

1592. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of rhodamine dialkylamides. Jan. 20.

1947. C. D. Abel. — From the Actien Gesellschaft für Anilin Fabrikation. Manufacture of new dyes. Jan. 25.

1962. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Improvements in the manufacture of blue mordant-dyeing colouring matters. Jan. 25.

2655. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Improvements in the manufacture of colouring matters of the rhodamine series. Feb. 1.

2874. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. The manufacture and production of new disazo dyes on cotton. Feb. 3.

3452. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of colouring matters. Feb. 9.

Complete Specifications Accepted.

1896.

5857. H. H. Lake. — From The Farbwerk Muhlheim. Manufacture of colouring matters. Feb. 17.

7313. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation. Manufacture of new raw materials and of direct-dyeing colouring matters derived therefrom. Jan. 27.

28,810. W. E. Heyes.—From The Chemical Works, Sandoz. Production of substantive trisazo colouring matters. Jan. 27.

30,015. W. E. Heyes.—From The Chemical Works, Sandoz. Production of violet and blue colouring matters of the triphenylmethane group. Feb. 3.

1897.

304. R. W. James.—From W. J. Matheson and Co., Ltd. Process for the manufacture of aceto-derivatives of the simpler aromatic amines. Feb. 17.

V.—TEXTILES, COTTON, WOOL, SILK, Etc.

Applications.

2230. W. L. Wise. — From C. A. Kottgen. Improvements in the process for weighting silk or silk waste. Jan. 27.

2549. S. Shorter and S. Stanbridge. Improvements in apparatus for the extraction, cleaning, and preparation of vegetable fibres. Jan. 30.

2736. J. Shearer. Method of improving the colour of raw jute fibre. Feb. 2.

2960. C. O'Brien and J. Shearer. Process for improving the colour of raw jute fibre. Feb. 4.

3218. A. G. Bonbon. Improvements in mercerising textile materials. Feb. 6.

3222. J. Millar. Improvements in textile fabrics or cloths. Feb. 6.

3646. J. Waugh.—From The New Augsburger Cotton Mill Co. Improvements in the washing and boiling treatment of fabrics and materials composed of vegetable fibres. Feb. 11.

3832. R. W. Strehlenert. Improvements in the method of spinning artificial silk, and apparatus therefor. Complete Specification. Filed Feb. 12. Date applied for Aug. 13, 1896, being date of application in Sweden.

Complete Specification Accepted.

1897.

198. F. Walton. Apparatus for manufacturing mosaic floorcloth. Feb. 10.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

Applications.

1263. E. Binns and I. Binns. Improvements in appliances to be employed in hank dyeing for spotted or random yarns. Jan. 18.

1629. T. Ingham. An improved dyeing process. Jan. 21.

1744. J. H. Widdicombe. The use of a mixture of gelatin and proteid with a colour powder for transferring designs to cloth and other materials. Jan. 22.

2149. E. Binns and I. Binns. Improvements in hank-dyeing machines. Jan. 27.

2505. J. Sharp. Improvements in dyeing and finishing fabrics. Jan. 30.

2620. W. Watson and E. Bentz. Improvements in and connected with the production of coloured discharge patterns on indigo-dyed cotton fabrics in calico printing. Feb. 1.

3256. H. W. Kearns and J. Barnes. Improvements in the production of fast black upon vegetable fibre. Feb. 8.

VII.—ACIDS, ALKALIS, and SALTS.

Applications.

1762. G. D. Howard.—From H. Howard. Improvements in methods of and apparatus for concentrating sulphuric acid or the like. Jan. 22.

1912. The Aluminium Co., Ltd., and A. G. Haddock. Improvements in bleaching compounds. Jan. 25.

2308. G. F. Zacher. Improved process for producing oxalic acid. Complete Specification. Jan. 28.

2651. C. H. Mehner. An improved method of producing ammonia. Feb. 1.

3592. J. E. Campbell. Improvements in apparatus for the concentration or rectification of sulphuric acid or other acids or liquors. Feb. 10.

3795. G. G. M. Hardingham.—From T. Huntington and F. Heberlein. See Class X.

Complete Specifications Accepted.

1896.

1667. T. Fairley. Manufacture and purification of ammonium nitrate and other salts. Jan. 27.

1668. T. Fairley. Manufacture of ammonium nitrate and sodium salts. Jan. 27.

2189. W. Garroway. Manufacture of sodium and potassium silicates, and of nitric and sulphuric acids. Jan. 27.

3662. A. D. Delsemme. A new or improved process for the purification of carbonic acid gas passing from lime kilns and designed for liquefaction. Feb. 3.

5011. P. Duggan. Saturators for use in the manufacture of sulphate of ammonia. Feb. 10.

6096. D. A. Peniakoff. Manufacture of aluminate of barium and aluminate of strontium, and the production of alumina sulphate of barium and of strontium and other products from the same. Feb. 3.

6835. B. T. L. Thomson. Process and apparatus for the manufacture of carbonic acid gas. Feb. 3.

VIII.—GLASS, POTTERY, and ENAMELS.

Applications.

1132. A. J. Boult.—From P. A. J. Gasse. Improvements in or relating to the decoration of earthenware, china, glass, tiles, bricks, and the like. Complete Specification. Jan. 19.

1614. L. H. Pearce. Improvements in the manufacture of glass articles. Jan. 21.

1871. A. E. Doucede. Improved method and means for connecting tubes and pipes of stoneware, earthenware, and glass. Jan. 23.

1995. T. Pfister and E. Barthels.—From A. Navarein. Improvements in cloisonne work. Jan. 25.

3376. E. W. Brock. New or improved process for transferring printed matter from paper to glass, earthenware, and other enamelled surfaces. Complete Specification. Feb 9.

Complete Specifications Accepted.

1896.

4189. F. Albrecht. Oven for burning or fusing lettering, designs, colours, and the like into or upon articles of glass, earthenware, and other glazed or enamelled goods. Feb. 3.

4945. F. Radcliffe. Furnaces suitable for use in the manufacture of glass. Feb. 17.

5772. L. A. Garchey. Manufacture of ceramic stone and objects by devitrification of glass. Feb. 3.

29,589. F. Joynson, W. Harrison, and G. T. Joynson. Machine for the manufacture of glass bottles, jars, and the like. Feb. 17.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, and CEMENTS.

Applications.

1795. La Société Metallurgique de Champigueulles et Neuves-Maisons. The manufacture of an improved cement. Filed Jan. 22. Date applied for July 11, 1896, being date of application in France.

1881. La Société Metallurgique de Champigneulles et Neuves-Maisons. Improvements in the manufacture of cement. Filed Jan. 23. Date applied for July 29, 1896, being date of application in France.

2085. C. A. Allison.—From F. P. Stone. Improvements in process of solidifying wood. Complete Specification. Jan. 26.

2195. A. Smart and G. W. Robertson. Improvements in apparatus employed in the manufacture of paving bricks or tiles. Jan. 27.

2402. W. D. Cliff. Improvements in glazed bricks and manufacture of the same. Complete Specification. Jan. 29.

2791. F. S. Winser. Improved method of manufacturing stoneware and the like. Feb. 2.

3230. H. Muller and A. B. Hunter. Thon cement. Feb. 6.

3345. C. Lucke. Improvements in presses for forming artificial stones, slabs, tiles, and the like. Complete Specification. Feb. 8.

3571. A. H. van der Vygh, H. van der Vygh, and G. van der Vygh. Improvements in the construction of fire-proof buildings. Complete Specification. Feb. 10.

Complete Specifications Accepted.

1896.

3232. J. T. Hanson. A new method of constructing fire-proof floors, flats, partitions, and walls. Feb. 10.

3972. S. Neffgen. Manufacture of artificial stone. Jan. 27.

7866. J. Hamblet and I. Parkes. Apparatus for use in manufacture of coping bricks, terracotta building blocks, and the like. Feb. 17.

18,714. J. Junbluth. Artificial stone for street pavements, pathways, and like purposes, and method of producing same. Feb. 3.

X.—METALLURGY, MINING, Etc.

Applications.

1508. R. Hodgson. Improvements in apparatus for extracting gold from ores. Jan. 19.

1515. C. I. Thorn. Sheet iron and steel castings close annealing heating furnace. Jan. 20.

1593. B. Becker. Improvements in the treatment of gold and silver ores. Jan. 20.

1778. C. Wichmann. Improvements in extracting gold from ores. Jan. 22.

2166. L. Yardley and G. Yardley. A new or improved method of enamelling aluminium articles. Jan. 27.

2201. H. E. Fry and R. H. Everitt. Improvements in the extraction of lead with silver, gold, and other metals from ores. Jan. 27.

2226. T. Doherty and P. D. Crerar. Improvements in or relating to the process of melting iron. Jan. 27.

2256. S. J. Robinson and S. Pope. Improvements in the construction of gas-heated steel-melting furnaces. Jan. 28.

2350. H. Wolff. Method for the preparation of zinc amalgam containing aluminium or magnesium. Jan. 28.

2922. A. Gutensohn. A new or improved process for recovering precious metals from metalliferous ore or sand. Feb. 3.

3037. K. A. May and F. Eldridge. An improved process for the extraction of gold and silver from their ores. Feb. 4.

3303. F. W. Streatfeild. Improvements in extracting metals from their ores or in obtaining solutions of same. Feb. 8.

3609. J. Robson and C. A. Joel. A new and improved aluminium alloy. Feb. 10.

3795. G. G. M. Hardingham.—From T. Huntington and F. Heberlein. Improvements in the treatment of sulphide ores of lead preparatory to smelting the same, and incidentally in obtaining sulphurous acid. Complete Specification. Feb. 12.

3926. H. Niewerth. Improvements in the manufacture of iron and steel. Feb. 13.

3932. M. Hinzelmann. An improved process for applying leaf metal. Complete Specification. Feb. 13

Complete Specifications Accepted.

1896.

2931. J. S. Wallace and J. Castell-Evans. Obtaining metals from refractory ores. Feb. 10.

3086. J. B. Torres. Extraction of gold, silver, and other metals from ores and the like. Feb. 17.

4422. The Exploring and Gold Mining Association, Ltd. —From S. H. Emmens. A process for recovery of zinc from blende ores. Jan. 27.

4667. F. F. Barnes and F. Campbell. Soldering of aluminium or metals alloyed with aluminium. Feb. 17.

6572. J. Imray. From M. A. J. Roux. Apparatus for separating volatile metals from their alloys. Feb. 17.

7615. R. Farley and E. Tonks. Manufacture of steel and iron sheets. Jan. 27.

18,417. C. Phillips. Dry process or method of ore-dressing and gold-separating, and apparatus therefor. Feb. 3.

23,958. G. W. Petersson. Process of manufacturing briquettes of iron ore or other iron compounds and briquettes thus made. Jan. 27.

25,133. A. Macdonald. *See* Class XIII.

26,066. D. White and T. M. Simpson. Method and apparatus for extracting precious metals from slimes or other finely-divided material containing the same. Feb. 10.

XI.—ELECTRO-CHEMISTRY and ELECTRO-METALLURGY.

Applications.

25,360a. F. H. Snyder. Improvements relating to the coating or covering of metals and other substances with metals and other materials by the aid of the electric arc. Complete Specification. Filed Feb 5. Date claimed Nov. 11, 1896.

1279. W. Rowbotham and C. Levetus. Improvements in and relating to accumulators or storage batteries. Jan. 18.

1369. W. Rowbotham. Improvements in and relating to accumulators or storage batteries. Jan. 19.

1430. E. J. Wade. Improvements in and connected with electrical storage batteries. Jan. 19.

1535. A. James. Improvements in the electric precipitation of gold and silver from their cyanide solutions. Complete Specification. Jan. 20.

1572. H. Leitner. Improvements in electrolytes for the deposition of zinc and for use in secondary batteries. Jan. 20.

1595. T. Hamilton. Improvements in electrical accumulators. Jan. 20.

1627. A. E. Cook. Certain improvements in apparatus for the electro-deposition of metals. Jan. 21.

1657. G. A. Webb and W. A. Thoms. Improvement in the electro-deposition of metals and alloys. Complete Specification. Jan. 21.

1714. W. Rowbotham. Improvements in and relating to accumulators or storage batteries. Jan. 22.

2207. J. Greenwood. Improvements in and relating to the decomposition of alkaline salts by electrolysis, and in apparatus therefor. Jan. 27

2253. A. E. Porte and J. R. Sykes. Improvements in electrical accumulators. Jan. 28.

2272. J. L. Dobell. Improvements in or connected with electric batteries. Jan. 28.

2291. M. Engl. Improvements in insulating frames for electrodes. Jan. 28.

2378. W. Rowbotham. Improvements in and relating to primary batteries. Jan. 29.

2412. H. J. Sheldon. An improved method of depositing aluminium by electricity. Jan. 29.

2418. E. W. Bonson. An improved form of electrode for galvanic or secondary batteries. Jan. 29.

2447. P. W. Northey and The Epstein Electric Accumulator Co., Ltd. Improvements in and relating to accumulators or storage batteries. Jan. 29.

2572. C. R. G. Smythe and M. Crawford. Improvements in and connected with electric accumulator and battery plates. Feb 1.

2709. W. E. Heys.—From E. Dumoulin. Improvements in and relating to the electro-deposition of copper and other metals. Feb. 2.

2710. W. E. Heys.—From E. Dumoulin. Improvements in the method of and apparatus for the electro-deposition of copper and other metals on rotary mandrels. Feb. 2.

2711. W. E. Heys.—From E. Dumoulin. Improvements in and connected with apparatus employed in the electro-deposition of copper and other metals. Feb. 2.

2712. W. E. Heys.—From E. Dumoulin. Improvements in and connected with apparatus for the electro-deposition of copper and other metals. Feb. 2.

2789. H. S. Lloyd. Improvements in storage batteries. Complete Specification. Feb. 2.

2790. L. Bresson and P. Pacotte. Improvements in electric furnaces for use in the manufacture of calcium carbide, applicable also for obtaining metals from their salts. Filed Feb. 2. Date applied for Sept. 15, 1896, being date of application in France.

2796. P. C. A. Laglenne. Improvements in electric batteries. Feb. 2.

3053. E. S. New. Improved separator for the plates of secondary batteries. Feb. 5.

3113. R. R. von Perks and J. Benger. Improvement in electrical accumulators. Feb. 5.

3235. I. A. Timmis. Improvements in secondary batteries. Feb. 6.

3346. C. Payeu. Improvements in secondary batteries. Feb. 8.

3488. E. Schattner. Improvements in secondary batteries. Feb. 10.

3526. J. E. Hartley.—From F. W. Zingsem. Improvements in apparatus for the electro-deposition of metals. Feb. 10.

3831. A. R. Adams. Improvements in or relating to electric batteries. Feb. 12.

Complete Specifications Accepted.

1896.

4957. E. Clark, W. Clark, and F. King. Plates, grids, or supports for the elements of secondary batteries. Jan. 27.

5673. A. S. Elmore.—From J. O. S. Elmore. Apparatus for refining metals by electrolysis. Feb. 17.

7259. W. P. Thompson.—From J. Julien. Construction of soluble metal electrodes for accumulators. Jan. 27.

12,149. T. J. Holland. Electrolytic apparatus. Jan. 27.

23,663. P. Garuti and R. Pompili. New and useful improvements in the apparatus for the production of oxygen and hydrogen by electrolysis. (System Garuti.) Feb. 17.

25,799. F. W. Schneider. Construction of electrodes for accumulators. Feb. 3.

25,804. Dieffenbach. Electrolytic production of zinc and alkalis. Feb. 17.

29,307. A. Heil. Peroxide of manganese and carbon electrodes, and method of producing the same. Feb. 10.

29,907. C. Brault. Manufacture and production of elements or plates for electrical storage batteries. Feb. 17.

XII.—FATS, OILS, and SOAP.

Applications.

1305. T. C. Brynn and B. J. Sharpe. Washing solution. Jan. 18.

1355. E. A. Ruch. Improved method of concentrating glycerin. Complete Specification. Filed Jan. 18. Date applied for July 18, 1896, being date of application in France.

1448. G. Varley and A. Jervis. Improvements in means for deodorising petroleum and like oils. Jan. 19.

1554. J. C. F. Müller. Process and manufacture of a cleaning and caustic agent. Complete Specification. Jan. 20.

2324. J. Twinch. An improved process for the refining of linseed oil and the manufacture thereof. Jan. 28.

2662. W. E. Pearson. Improvements in and connected with the production of prepared tar, mineral, and other oils for pharmaceutical, veterinary, technical, and other purposes. Feb. 1.

3207. E. J. Ellis. Improvements in or relating to soap or detergents. Feb. 6.

3700. J. David. Improvements in the treatment of oleic acid. Feb. 11.

3702. F. J. Billingham. An improvement in the manufacture of soap. Feb. 11.

Complete Specifications Accepted.

1896.

799. H. Hinterberger. Apparatus for separating oil and air from water and recovering the oil. Feb. 17.

2511. J. Jack and M. Blake. Extracting and recovering oil from fish, fish refuse, or other oil-producing animal or vegetable matters. Jan. 27.

7379. W. R. Harrison and E. Stephenson. Method and apparatus for extracting oil from seeds or other oleaginous substances. Feb. 10.

25,472. J. Davidson. Manufacture of fatty matter from certain animal tissues. Jan. 27.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

2108. G. L. Burnham. An improved paint for ships' bottoms. Complete Specification. Jan. 26.

2667. A. J. Boult.—From F. Fritsche and Co. Improvements in preservative paints, pigments, and the like. Feb. 1.

3116. J. de F. Maunder. Improvements in the manufacture of zinc oxide. Feb. 5.

3576. J. C. Mewburn.—From J. C. Taylor. Improvements in the manufacture of anti-fouling compositions or paints. Feb. 10.

25,133. A. Macdonald. Manufacture of white lead pigment and in the separation of metallic lead from lead sulphide. Feb. 10.

XIV.—TANNING, LEATHER, GLUE, and SIZE.

Applications.

1442. W. J. J. Hawkins. Improvements in printers' roller composition or the like. Jan. 19.

1587. T. D. Bowman and J. Bowden. Improved manufacture of imitation leather. Jan. 20.

2746. A. J. Boult.—From C. M. Higgins. Improvements in or relating to adhesive compounds. Complete Specification. Feb. 2.

3073. J. Howden.—From J. Tyciak. Improvements in glue compounds and in applying the same to barrels and the like. Complete Specification. Feb. 5.

3180. S. Millar and C. E. Millar. Improvements in treating hides and skins. Feb. 6.

Complete Specifications Accepted.

1896.

3901. F. B. O. Hawes. Treatment of leathers for their better preservation and surface preparation. Feb. 3.

4199. J. Hall. Apparatus for treating skins, hide, leather, and like substances. Feb. 17.

10,263. A. Levinstein. Tanning. Feb. 17.

10,390. A. Levinstein. Tanning apparatus. Feb. 17.

XV.—AGRICULTURE and MANURES, Etc.

Complete Specifications Accepted.

1896.

2678. J. G. Wiborgh. Treatment of apatite and other mineral phosphates, and manufacture therefrom of products suitable for fertilising purposes. Feb. 10.

21,776. O. Heymann and A. Nitsch. Process and apparatus for the drying and disintegration of superphosphates. Feb. 3.

XVI.—SUGARS, STARCHES, GUMS, Etc.

Applications.

1338. A. Verley. Improvements in the manufacture of sugar. Jan. 18.

3438. T. E. Forman. Improvements in the manufacture of extracts and preparations to be used in the conversion of starch, gluten, and for other purposes. Feb. 9.

3472. F. L. Calmant. An improved process for the purification of alcohol in general and of raw beetroot sugar and residue by means of carbonic acid and vegetable carbon. Feb. 9.

3900. A. Weickmann. See Class XVII.

Complete Specifications Accepted.

1896.

3613. E. Shaw. Boiling sugar and other syrups. Feb. 17.

28,188. A. J. Boult.—From L. Janssens and Co. Production of sugar. Feb. 10.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

1455. A. Kinder. Improvements in or relating to malt kilns. Complete Specification. Jan. 19.

2287. E. E. M. Payne, T. O. Kent, and E. E. Pullman. Improvements in compositions or mixtures to be used for the preservation of malt liquors such as ale, beer, stout, porter, and other like saccharine liquids, and in the methods of using and applying such compositions or mixtures. Jan. 28.

2434. J. Sinclair. Improved valve fittings for use in fermenting vats or "stone squares." Jan. 29.

2553. J. L. Dewar. A new or improved method of treating beer, ale, stout, and other fermentable liquors for bottling. Jan. 30.

2872. B. Tabrum. Improvements in the process of preserving hops. Feb. 3.

3472. F. L. Calment. See Class XVI.

3900. A. Weickmann. A process for utilising the solid refuse of breweries, distilleries, starch, sugar, poudrette factories, and the like. Feb. 13.

Complete Specifications Accepted.

1896.

2150. R. Cunliffe. See Class I.

21,022. A. E. Feroe. Process and apparatus for treating malt liquors. Feb. 10.

22,403. C. H. McEuen.—From J. T. Bennett. Apparatus and process for removing saccharine and other impurities from wine and spirits. Feb. 17.

28,763. J. Y. Johnson.—From The Electric Rectifying and Refining Co. Apparatus for purifying and decolorising saccharine solutions and other liquids. Feb. 17.

XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

Applications.

A.—Foods.

2670. J. L. W. Thudichum. New and improved modes of producing and treating extracts of flesh or meat and of other parts of animal bodies. Feb. 2.

3810. H. Schowell. Improvements in or relating to the preparation of lupines for yeast manufacture, cattle food, and similar purposes. Feb. 12.

3830. B. Iribarnegaray. An improved process for use in preserving butter and other foodstuffs. Complete Specification. Feb. 12.

B.—Sanitation.

1437. W. J. Engledue. Improvements in the treatment of sewage. Jan. 19.

1722. P. R. Candy. Improvements in apparatus for use in the purification of sewage and polluted waters. Jan. 22.

2228. F. W. Strong. Improvements in tanks and apparatus for use in connection with the same for the purification and treatment of sewage and impure waters. Jan. 27.

2757. E. S. Beaven. Improvements in processes for the treatment of sewage. Feb. 2.

C.—Disinfectants.

2774. I. Werber. Improved antiseptic or disinfectant preparation. Feb. 2.

Complete Specifications Accepted.

A.—Foods.

1896.

5749. D. Finkler. Preparation and extraction of albuminous substances from animal or vegetable mixtures and products. Feb. 17.

6496. W. Nageli. Process for sterilising and preserving food products and beverages. Feb. 17.

25,204. H. D. Perky. Machines for the reduction of cereals and other products to useful, artistic, and convenient forms for food. Jan. 27.

28,708. A. J. C. Rayson. The manufacture of a substitute for coffee. Jan. 20.

B.—Sanitation.

1896.

3047. J. B. Petrie. Construction of settling tanks or lodges, used in the purification of sewage and other foul waters, and in the clarification of manufacturers' liquid compositions. Jan. 27.

5955. W. E. Adeney and W. K. Parry. The utilisation of by- and waste products from iron and steel industries for the manufacture of improved precipitants for the purification of sewage and other waste liquids. Feb. 17.

C.—Disinfectants.

1896.

5381. P. O. Dowd. Composition for utilising and disinfecting sewage matter. Feb. 10.

5705. J. N. Spence. New or improved insecticide. Feb. 3.

5955. W. E. Adeney and W. K. Parry. *See* Class XVIII. B.

14,852. M. Crawford. Manufacture of disinfecting, deodorising, and bleaching agents, and apparatus therefor. Jan. 27.

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Applications.

1390. D. Pearson and D. N. Bertram. Improvements in paper-pulp refining engines. Jan. 19.

1550. P. H. Hansen and J. K. Westengaard. Improvements in the manufacture of artificial horn and the like. Jan. 20.

Complete Specifications Accepted.

1896.

1556. C. Raymond. Manufacture of gelatinised paper or other fabrics suitable for the reproduction, by printing with fatty inks, of writings, drawings, and the like. Feb. 3.

8309. H. Thame. Manufacture of hollow pulp-ware, particularly applicable to buoyant articles. Feb. 17.

26,464. J. A. Wilkinson, F. C. Wilkinson, and A. T. Wilkinson. Machine for softening paper. Jan. 27.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

Applications.

1339. C. Goldschmidt. The application of piperidine for effecting the dissolution of uric acid. Jan. 18.

2472. J. Y. Johnson.—From C. F. Boehringer and Soehne. Improvements in the preparation of hydroxycaffeine by the direct methylation of uric acid or of certain mono- and dimethyl derivatives thereof also obtainable in the process. Jan. 29.

2556. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of nitroalphylpyruvic acids. Jan 30.

3118. A. Walker. Improvements in the manufacture of malt extract. Feb. 5.

3499. W. Mills. Manufacturing the nitride of carbonyl. Feb. 10.

3500. W. Mills. Improvements in manufacturing nitrides. Feb. 10.

3501. W. Mills. Improvements in manufacturing urea. Feb. 10.

3957. J. Y. Johnson.—From C. F. Boehringer and Soehne. Improvements in the manufacture or preparation of dichloroxypurine. Feb. 13.

Complete Specifications Accepted.

1896.

2205. A. J. Boult.—From C. Schmid. A new benzene sulphonic acid and artificial musk. Feb. 10.

3555. J. C. Richardson. Manufacture of camphors or like compounds. Jan. 27.

15,295. W. E. Heys.—From The Chemische Fabrik vormals Sandoz. The manufacture or production of bismuthoxyiodidemethylgallate. Feb. 10.

28,969. L. Lederer. A process for the preparation of acetyl and lactyl derivatives of aniline and phenitidine. Jan. 27.

XXI.—PHOTOGRAPHY.

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2269. T. Miller. A mode of developing photographic negatives without the aid of a dark room. Jan. 28.

2305. H. H. Lake.—From H. C. Fairchild. Improvements in methods of producing photographs in relief. Complete Specification. Jan. 28.

3121. G. J. Sershall. Improvements in photographs with coloured effects, and in the method of producing same. Feb. 5.

Complete Specification Accepted.

1896.

29,437. E. Edwards. — From O. Moh. Process for making photographic negatives. Feb. 17.

XXII.—EXPLOSIVES, MATCHES, Etc.

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1603. W. G. Newton. Improvements in safety fuses. Jan. 21.

2557. M. Bielefeldt. Improved manufacture of explosives. Jan. 30.

2785. A. J. Fredrikson. Improvements in and relating to the manufacture of matches. Feb. 2.

3002. H. Maxim.—From R. C. Schupphaus. Improvements in smokeless explosives and processes of their manufacture. Feb. 4.

3135. H. R. von Dalmen. Improvements in blasting compositions. Feb. 5.

Complete Specifications Accepted.

1896.

4509. W. Greaves and E. M. Hann. Explosives. Feb. 3.

5575. A. Pain. Improvements in ships' signal lights, rockets, and similar fireworks. Jan. 27.

5815. G. Craig. Manufacture of nitrate of ammonium and apparatus therefor. Feb. 10.

6052. G. Schwiening. Manufacture of igniting compound for matches. Jan. 27.

6937. W. Greaves and E. M. Hann. Explosives. Feb. 3.

25,711. C. Mortier and H. A. Sandon. Manufacture of gunpowder. Feb. 3.

XXIII.—ANALYTICAL CHEMISTRY.

Application.

3673. G. S. Newth. Improved means of detecting potassium compounds in analysis. Feb. 11.

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THE JOURNAL

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NOTICES.

Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, part of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows:—

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ANNUAL GENERAL MEETING.

The Annual General Meeting will be held in Manchester on the 14th, 15th, and 16th July next. Tickets of Membership will be issued in time for the Meeting, and will form, as heretofore, vouchers for visits to works and excursions.

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Deaths.

Kunheim, Dr. H., 32, Doratheeenstrasse, Berlin. March 22.

Molineux, Jno., C.B., Selsley House, Battersea, S.W. March 27.

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SESSION 1896–97.

April, 5th, 1897.—Mr. Oscar Guttmann, Assoc. M. Inst. C.E., F.I.C. "The Chemical Stability of Nitro-compound Explosives."

Meeting held on Monday, January 18th, 1897.

THE CHARACTER OF THE LONDON WATER SUPPLY.

BY W. J. DIBDIN, F.I.C., ETC.

(*This Journal*, 1897, 9.)

Discussion.

Mr. W. T. Burgess, having had considerable experience of London water examination in Dr. Frankland's laboratory, was deeply interested in the figures put before the meeting. He had had no experience in working the micro-filter, but looking at the figures before them he thought it at least possible that the results might be influenced by the length of main through which the water passed before examination. For instance, the Southwark Company had to send its water through a very considerable distance of main, and until one had exact information as to the points at which the samples were taken, one could not properly criticise them. Referring to Table IV., which gave percentages of nitrogen in suspended matters and sludges of different origin, he thought it a pity the author had not included some suspended matter or deposit taken from the Welsh rivers. He doubted Mr. Dibdin's correctness in assuming that the improvement in the water supply during the last few years was the result, to a great extent, of the L.C.C. examination made in 1891. If the records of the examinations made by Dr. Frankland for the Local Government Board were examined, it would be seen that the year 1891 was relatively a bad one for the Company's operations. The years immediately in front of 1891 were much better, and the results then obtained compared very favourably with those got in 1893, 1894, and 1895. His own experience of the combustion process led him to think that the author's figures for organic nitrogen were decidedly too high. The author's results showed how well, so far as organic matters were concerned, London filtered water compared with the Welsh river water.

Dr. Dupré congratulated the author on the admirable working of his micro-filter. He would confine his remarks to the bacteriological aspect of the question. In his opinion, water known to be contaminated by sewage should be rejected entirely for domestic purposes. He thought that the principal danger to London lay in the possibility of an epidemic occurring among the population higher up the river. Instancing the cholera epidemic at Hamburg, Dr. Dupré said that the Hamburg people had drunk the water of the Elbe for many years with impunity, but the cholera infection coming down with the river water had caught the city undefended. Altona—a suburb of Hamburg—it was thought was secure on account of its filter beds, but on one occasion the filter beds broke down, and the number of deaths that occurred would have been equivalent to 5,000 in the London water area. If such a thing were to occur in London it would occasion a terrible and justifiable outburst of indignation. Dr. Koch had fixed 100 micro organisms per c.c. as a measure of effective filtration which should not be exceeded. It was quite clear from the tables before them that that was a limit which was very frequently exceeded in the London drinking waters. Dr. Koch also urged that whenever, during an epidemic, the number of microorganisms rose above that limit, consumers should be informed, so that they might take preventive measures; but unfortunately it took at least two days to discover the condition of the water, and therefore that precaution was not all that could be desired. He would admit that, on the whole, filtration of the London water supply was extremely well conducted, but it was susceptible of improvement, especially in the direction of more frequent and careful tests. Every filter ought to be watched daily. If the people of London, knowing the risk they ran, chose to submit to it rather than incur the expense of protection, well and good But the public ought at least to have the case put fully and clearly before them, so that they might make their choice with their eyes open.

Mr. A. E. Fletcher considered that Mr. Dibdin had shown clearly that not only an immense improvement had been made by the water companies in the matter of more careful filtration, but that this improvement might be carried still further, and the water supply of London rendered much more suitable for consumption by the adoption of Clarke's process for softening the water. It was difficult to understand how the people of London could submit to use a water notorious for its hardness, and sometimes for its pollution also, when a process for purifying and softening it was available, which process had been proved efficient by the experience of the last 50 years. Using as he did a supply that was so treated he felt the advantage of having a water which did not corrode the supply pipes and kitchen utensils. Even the economy thus effected would compensate to a great extent the necessary outlay. The main point, however, was the benefit to the health of the people that would follow from the more efficient purification of their water supply if so treated; and, taking into account the enormous dividends which the London water companies now realised, he thought that they should not hesitate on the ground of expense to adopt any process which would secure that end. He thought that this Society ought to send forth an emphatic endorsement of Mr. Dibdin's recommendation in this paper of the adoption of some process for softening the London water.

Mr. Shirley Murphy said his own duty was to consider the effects of the water supply on the health of the inhabitants of London. In former times, when the water for domestic purposes was drawn from the rivers, where they were subject to tidal influence, the effects of these waters was manifested in times of cholera prevalence in the mortality returns; but since the companies had gone above the locks for their supplies, this evidence had been wanting; and, except in 1866, when cholera was undoubtedly spread by water drawn from the Lea, it had not been shown that the river waters had added to the death rate. More recently they had been able to study this subject more minutely, as, owing to the system of notification of cases of infectious disease in force in London, the subject could be considered in the light of cases instead of deaths. He had in a recent report given the figures of notified cases of enteric fever relating to a year in which there had been exceptional floods in the rivers, and these figures raised the question whether the London river water had not on that occasion, although in very much less degree, behaved in a manner similar to that in which Mr. Barry had shown the Tees water had behaved. He agreed with Mr. Dibdin that the water supplied from the rivers was capable of great improvement.

Dr. J. A. Voelcker had worked according to the author's method, and had found it extremely useful. It seemed to him, however, to fail in one important point, namely, that it did not distinguish between quantity and quality. It was said that suspended matter might be

disregarded if it did not exceed 1 mm. per litre. His own opinion was that if a river were in flood and gave more than that amount of earthy matter, this might be unpleasant, but not dangerous; whereas if it were only $\frac{1}{2}$ m.m. per litre, and that $\frac{1}{2}$ mm. consisted of dangerous matter, it would make all the difference. Therein lay the whole crux of the question, *viz.*, quality as compared with quantity of suspended matter. With respect to the physiological side of the subject, notwithstanding the great advance made in that department of knowledge, he must confess himself still an unbeliever in its usefulness until we came to know which bacteria were harmful and which were harmless. For his part he was willing to drink a good deal of suspended matter in the form of *débris*, but he would not, if he could help it, take one poisonous bacterium into his system. While admitting the force of the agruments advanced by Dr. Dupré and Mr. Fletcher, he must confess that, knowing what he did of the respective water supplies of London and of the country, he would, on the whole, rather take his chance of life with the London supplies. There was another point, namely, that after all, a greater amount of harmful matter was frequently introduced into our water supply after rather than before the distribution of it commenced. Far more care ought to be taken to keep a water supply free from the contamination to which it was subject in the cisterns of houses. Lastly, as to the suggestion that London water should be softened by means of Clarke's process, that might possibly be necessitated in the case of waters of certain districts but it would be decidedly objectionable from a health point of view in the case of London.

Mr. J. A. R. Newlands endorsed the opinion expressed by Mr. Dibdin and others that it would be highly beneficial to the London water supply to treat it by the Clarke softening process. The question of expense should not be allowed to stand in the way of the public welfare, since the public health was a matter of supreme importance. Even after the application of that process the London supply could hardly be considered a "soft" water.

Meeting held on Monday, February 15th, 1897.

FURTHER DISCUSSION ON Mr. W. J. DIBDIN'S PAPER ON "LONDON WATER SUPPLY."

Mr. Bertram Blount said that, notwithstanding the disadvantages of discussing a paper a month after its reading, there was so much of real importance in Mr. Dibdin's production that the interest of the meeting would, he hoped, be sustained. He congratulated the author heartily on having supplied for the purpose of water examination an analytical process which must be reckoned a distinct improvement on existing methods. The criticisms offered had, however, some justification, namely, that a certain assumption had to be made in estimating the quantity of collected precipitate. This precipitate was collected in a tube so small in bore that it was practically capillary; and the diameter of the tube was a factor in the measurement of the quantity of the precipitate. The determination could therefore hardly be made with extreme accuracy, because the tube itself would not always be circular or of known internal diameter. It would be well therefore if tubes could in future be standardised, to save trouble to the users. It seemed to him that the qualitative aspect of the examination was of greater importance than the quantitative, because it would give one some idea of the character of the water supply which had created the precipitate. In this connection he would refer particularly to the very large relative correction made for what the author called unavoidable "rust and dust," which seemed to him to deduct somewhat from the value of the method. Perhaps the author could tell them whether much of the detritus was ascribable to the attrition of packing materials used in the pipes, which would naturally break up in time and be caught on the micro-filter. It was very satisfactory to have it placed on record by so eminent an authority as Mr. Dibdin that London water is not always perfectly clear—that being a fact of which it was very difficult to convince the officials of the water companies. Coming to a more serious consideration, *viz.*, the significance of this material, he must dissent from the view that the small quantities of suspended matter present indicated something seriously wrong in London water. The case had been stated clearly and fairly by Dr. Dupré, who had said that Londoners should clearly recognise that they were running some risk in drinking sewage contaminated water purified by filtration. There were risks in everything, and he for one was well content to accept the present condition of London water as sufficiently satisfactory. He considered that dishonest efforts were being made by irresponsible persons to vilify the character of London water—persons who had not, like Mr. Dibdin, a genuine scientific interest in the matter; and these tactics ought to be clearly condemned. Referring to the character of the Welsh water advocated by such persons, he found that according to Mr. Dibdin's results London water would compare with it by no means unfavourably, and it was to be borne in mind that whereas for the London water they had the figures for the worst as well as for the best parts of the year, the figures relating to the Welsh water dealt with one portion of the year only, when perhaps it was at its best.

Mr. Dibdin said the figures for the Welsh water were the average of five samples taken from different places in May.

Mr. Blount, resuming, said that was an advantage to the Welsh water, as the average for the whole year would probably yield less favourable results. Then there was that hardy annual, the suggestion of softening London water. No doubt that would suit manufacturers, as it would save their boilers; but as a consumer of water for drinking and ablutionary purposes he would regard the softening process as decidedly objectionable.

Mr. E. Grant Hooper cordially endorsed the congratulations offered by Mr. Blount. The author had put before them results not available before; and in his micro-filter had devised a piece of apparatus which would be generally useful. He would have liked to see in the paper a clear statement as to Mr. Dibdin's opinion with reference to the condition of the London water supply so far as it was drawn from the Thames. Did he think the water so bad that it was imperative something further should be done in the way of purification, and, if so, was he satisfied that that something should be the adoption of the suggested softening? For himself the speaker did not think it had been shown that the present condition of London water was by any means a serious one. Dr. Dupré had referred to the risk that must always exist in taking a drinking supply from such a river as the Thames. With that opinion he agreed, for there must always be a danger of pathogenic organisms getting through the filters. But he would like to briefly review what had been shown by the paper, and in the first place he deprecated in a paper of this kind the references to the tons of solid mud in a year's supply of water. Even when to produce this equivalent of mud the total solid matter had been increased by the necessary 90 per cent. of water, he would suggest that if really impressive figures were desired it was unwise to limit the quantity to a mere year's supply. What did this suspended matter really amount to in the quantities with which individuals had to deal? His own observation, which included the summer consumption of thirsty children, would not allow him to rate the demand for actual drinking water at more than a pint per day per person. Mr. Dibdin's results showed that this pint of water contained $\frac{3}{1000}$ grain of wet mud, or about $\frac{3}{10000}$ grain of dry solid matter, more than half of which was inorganic matter. This was not a very serious quantity. But that was not all. On such quantities of suspended matter Mr. Dibdin classed no less than 30 per cent. of the Thames water samples as objectionable. What, then, should they take as a standard of purity? He thought that it was generally admitted that the Kent Company's water would supply such a standard, and that, except for its being somewhat hard, that water was commonly held up as a quality to be much desired. Nevertheless they found that when the Kent Company's water was tested by Mr. Dibdin's micro-filter process, no less than 18 per cent. of the samples were classed as objectionable. That seemed to him ample proof that the condition of the water supplies generally was not very bad. He gathered, however, that Mr. Dibdin

thought some of the companies needed careful watching and keeping up to the mark in the matter of filtration. He agreed that in so important a matter careful watching was necessary. The author had said that though the companies' supplies were constantly examined, nothing ever came of it. He ventured to suggest that that was because they were generally satisfactory. As to the suggested softening, he did not quite agree with Mr. Blount's views, though for personal use he would prefer a water moderately hard—say four or five degrees. There was no doubt that softening would result in a further purification of the water, but with the admitted degree of purity represented by the London water the essence of the matter was the question of cost, and about that the author had said nothing. It had, of course, been gone into again and again in the past. As far back as 1851 a Royal Commission investigated the question and fixed the cost at that time at 1*l.* 7*s.* per 1,000,000 gallons, using the Clarke process. This was calculated as representing an increase to the companies of 10 per cent. on what the water cost them, or 4 per cent. on its selling value. Later experiments had shown a very similar cost, though the figures might be somewhat different now. It was, however, obvious that the cost would be serious, and, unless the reason for incurring it were almost imperative, he thought London ratepayers, upon whom he supposed the cost would ultimately fall, would hardly view the proposal with favour.

Mr. W. Thorp would like to know what quantities the author had used in arriving at the given weights of suspended matter, and what method had been employed. The suspended matter in even 100 gall. of water, about 7 mgrms., would be no easy amount to manipulate. He must also object to the author's mode of illustration by multiplying the weight of dry suspended matter by 10 and calling it mud, to which Mr. Hooper had also referred. It seemed not only intended to alarm the unlearned, but it failed to convey any better idea than the actual figures would do. The only satisfactory course was to give the ratio of one number to another; to merely say that 131,000,000 gallons per day would carry down 67 tons of mud, equal to $6\frac{3}{4}$ tons of dry matter, per annum, conveyed nothing to his mind. Again, why was the Fitzroy River, in Queensland, taken for comparison? Unless that river had been as closely studied as London waters, and the figures quoted had been derived from samples taken under similar conditions, the comparison would be misleading. He also considered the "constant" deductions made by the author undesirable. For example, in deducting the amount of suspended matter in the Kent water as a constant, one was met by this difficulty—that the quality of even the Kent water in this respect was not constant. Taking the figures given on page 11, it would be seen that in some months there was only 0·0006 of suspended matter, while in other months the quantity was greater; so that for the year 1895 there was an average of 0·0011. In another year, however, the average might be something quite different, depending on the circumstances of the time. For instance, in December 1892 the quality of the water in this respect was very different from what it was in 1895, and therefore, by taking a constant deduction, one was exposed to the difficulty of dealing with a merely arbitrary figure, which might possibly on some occasions even be greater than the figure from which it was to be deducted. Then again, he would like to ask whether the volatile organic matter from which the proportion of nitrogen was calculated meant "loss on ignition," because that would vary very considerably, according to the way in which it was estimated, and would also include a good deal that was not organic matter. Again the figures given on page 12, fourth column, had perplexed him considerably, and he did not approve of the calculation from which they appeared to have been derived, namely, the multiplication of the percentage number of samples containing objectionable matter by the average weight of the suspended matter contained in the water. Seeing that this "objectionable matter" must vary greatly in character from time to time, it appeared that the author was using two figures to calculate a third, the figures used not being commensurate one with the other. The calculations to which he had referred seemed to imply an attempt to formulate his analytical results so that they should be understood by those who were not expert. He regarded such a course as quite hopeless in so complicated a matter as water analysis. Figures that meant one thing when associated with certain other figures, would imply something totally different when the circumstances of the case differed.

Mr. W. Lawrence desired to point out, with regard to the proposal to soften the London water, that it was quite unnecessary to carry out any such proposal in respect of the whole of the supply, much of it being used for purposes for which a hard water was quite as suitable as a soft water. Even for boiler purposes it was not so much the hardness of London water that rendered it objectionable as the sulphates which it contained, and which could be dealt with more readily in the presence of the carbonates than if the latter were removed.

Mr. E. J. Bevan endorsed the remarks of Mr. Hooper with respect to the Kent and New River supplies. Mr. Dibdin himself had admitted that those supplies were beyond suspicion; yet he had found that in the case of the Kent Company 18 per cent., West Middlesex Company 24 per cent., and the New River Company 21 per cent. of the samples were rejected as being impure. According to the author's tables New River water in June showed 0·001 grains of suspended matter per gallon, yet the suspended matter observed in the micro-filter was absolutely nothing. Again, the author had called attention to the close concordance between the results obtained by his micro-filter method and those of the chemical and biological processes of examination (page 12). He would like to call attention to figures which to his mind showed that that concordance was not by any means striking. The amount of nitrogen in the suspended matter in the case of the West Middlesex and New River supplies was stated to be 2·86 per cent.; yet, whereas in the case of the former the oxygen absorbed was 0·072, in the case of the New River water it was only half the amount, namely, 0·041. Again, taking the relation of the number of the bacteria found to the amount of suspended matter, in the Grand Junction water the suspended matter was 0·0023 grain per gallon, bacteria 170; in the East London water the suspended matter was practically the same as the Grand Junction, but the bacteria found rose to 277 per c.c., while the Chelsea water, which showed more suspended matter, was found to contain only half the number of bacteria found in the East London water. These criticisms were not intended as suggesting that the paper had no value. On the contrary, he regarded the paper as a monument of industry, care, and application on the part of the author. At the same time he protested against too much reliance being placed on figures, of great value in themselves, but liable to be misinterpreted.

Mr. W. Whitaker said that he could not treat the subject from the point of view of the chemist, but perhaps it was as well that other views should be heard. As Mr. Dibdin admitted, the water companies had for many years done their best to provide a pure supply. It was certainly to their interest to do so; but he thought that the quality of the water should be considered not only as it was at its source of origin, but as received by the consumer; and the risks of impurities which were met with in its course down the river could not be neglected. The risks to which the water supply was subject could be minimised through careful watching, and he quite agreed with the author that the water companies needed watching, just as probably the London County Council or any other authority would. If the source of supply and the preventive measures of the companies were not constantly and carefully watched, there was always a danger that contamination would come in. The question was whether it was better and cheaper to go on with the present supply, subject to such risks, or to get supplies from another and purer source. That was purely a question of finance. He thought that the water supply of London should be, as far as possible, beyond the risks of contamination, and that if we were to continue to draw our drinking water from the Thames further improvement was necessary. In the first place a more powerful conservancy body was necessary, the present body

being neither strong enough nor rich enough to do what is required. Then there was the question of softening, of which he had had some experience. At Southampton they had the largest water softening works in the country, and the engineer thereof had roughly estimated the cost of the process at a farthing per thousand gallons. That was the presumed cost of reducing a very hard water of about 18 degrees down to about 6 degrees or 8 degrees, and they found that such a course gave satisfactory results. So far, the only softening process that had been referred to was the Clark, but he would be surprised if that were adopted in London, as the plant for it occupied a great deal of ground, which would involve heavy expenses. There were, however, modifications of the process, wherein the plant employed was more manageable. He was sure that any members interested in the subject could obtain valuable information by visiting the works at Southampton.

Dr. Bernard Dyer speaking as one who had to make water analyses, wished to express his admiration for the very neat and useful apparatus devised by Mr. Dibdin as an aid to microscopical examination. It did not matter, practically, whether the apparatus enabled one to estimate exactly the amount of sludge in a given quantity of water, but as a qualitative method showing the kind of impurities absorbable in water it was a most excellent device. He had read not only the paper, but the interesting report lately submitted by Mr. Dibdin to the L.C.C., and while fully recognising the importance of the results embodied in that paper he must admit that he felt regret that the method of examination adopted consisted in drawing from the mains instead of from the filter beds themselves. If Mr. Dibdin had not been able to obtain the consent of the companies to the latter course that would explain matters, but he thought that such a course should have been adopted if possible. The contention put forward was that they did not want to know what came from the filter bed but what the public actually got. He did not agree with that view in a case like this. It was well-known that the Thames was largely fed by surface contaminated water, and that despite all the care of the Conservators impurities containing pathogenic organisms might get in. But was the water so filtered that the total bacteria present in the water as it entered the beds were reduced to a minimum as it left them? If it were true, that, say, 16,000 organisms per c.c. were reduced to 50 or some such small number, most Londoners would be ready to run the very remote risk of their being a pathogenic organism in that number, but they wanted to know also whether the large number of organisms often found by Mr. Dibdin had really eluded the action of the filter or whether they were the result of mere multiplication of the few organisms remaining in well filtered water, or whether they may have had some other origin. It was well known that whatever care might be taken to protect domestic cisterns from dust, &c., it was necessary to thoroughly cleanse them from time to time, when a slimy growth was found on the sides and bottoms of the tanks. Probably something of the same sort existed in the water mains, the growth being no doubt fed by perfectly harmless organisms. Possibly the occasionally large quantities of matter found in the water as drawn from the pipes was due to the disturbance of this slime by adventitious causes, and he thought, therefore, that it was a pity that the author had not made his determinations on lines exactly parallel to those made by Dr. Frankland and other analysts, so that the public might have been in a position to judge as to whether the results recorded by those gentlemen indicative of so high state of efficiency with regard to filtration were confirmed or not by Mr. Dibdin.

Mr. C. E. Groves, referring to the fact that the author had taken his samples from the companies' standpipes, observed that special precautions were necessary in collecting water for bacteriological purposes, and would like to know, therefore, what method had been followed. From the report made to the L.C.C., it appeared that some of the samples had been analysed both by Mr. Dibdin and by an independent authority, and it seemed probable that the very great differences in the respective numbers of colonies of bacteria found were due to faults in the method of collection. With regard to the actual amount of solid matter present that was of slight importance, as in drinking a pint of water, one would imbibe but a minute fraction of a grain, whilst the dust on the drinking glass itself would no doubt be 10 or even 50 times the amount of the solid matter actually present in the water. The nature of that solid matter, however, was far more important. Most people regarded all bacteria as harmful, but this was not the case. By far the larger number of bacteria were non-pathogenic, and they assimilated and decomposed the organic matter dissolved in water which in fact served as food for the bacteria, being converted by them into non-organic matter; therefore the quality of a river water was actually improved by the bacteria present in it. The only way of making a fair comparison between the London supply and the Welsh water would be by running some of the latter through the mains, but of course that was impossible. The severe weather of two years ago had provided many of them with an opportunity of inspecting the interior of the mains which had been destroyed by the frost, and so far as his observation went they were anything but immaculate; it was possible therefore that the extraneous organic matter found by the author had been to a very large extent generated in the mains, but that point could only be settled by comparing samples taken from the mains with others taken from the clear water wells at the filter beds Mr. Bevan had referred to the variation in the proportion of oxygen absorbed to albuminoid ammonia; the speaker had often observed this to be the case in effluents and had found that it was due to the presence of nitrates.

Mr. Bevan explained that he had referred to the total nitrogen present.

Mr. Groves, resuming, said that the question of the advisability of a softening process introduced the engineering question which was a very large one. He might mention, however, that the Royal Commission had advised the construction of large storage reservoirs up the Thames where the water could be collected in time of flood, and he believed that a partial softening of such waters in the reservoirs would greatly accelerate the subsidence of the suspended solid matter.

Dr. S. Rideal said that the question resolved itself into one of the suitability of the present supply, and unfortunately many gentlemen who differed from the author on that point were not present. Dr. Dupré had said that it was simply a question of running a risk, but he had not sufficiently pointed out that the method of softening proposed was one by which the risk attaching to the present supply would be reduced, and the suitability of the water would be improved, as the process would remove a large proportion of the pathogenic organisms of all kinds. Dr Murphy, who spoke at the last meeting, had not brought out as strongly as he might have done the fact that two years ago an outbreak of typhoid in London was obviously due to the flood water distributed by the companies. The area supplied by the Kent water at the same time was free from such sporadic cases. It seemed to him that the proper course was to supply a perfectly sterile water, not generally, but that portion which was actually consumed for drinking purposes, and this could be easily effected by Pasteur filters being included as part of the compulsory water fittings supplied by the companies. The difference in the oxygen consumed and nitrogen found mentioned by Mr. Bevan was often met with. A more important point was the determination of the organic nitrogen in the suspended matter, since it was the condition of the nitrogen in the water that threw a light on its hygienic character. The value of the micro-filter seemed to him to be somewhat discounted by the fact that no close relationship existed between the number of millimetres and the actual weight of deposit when the large quantity of 1,000 gallons was taken. Perhaps that was because only a litre was examined out of the total originally taken. But the figures when working with a larger quantity were very variable as showing the difference between the nitrogen contents. He had occasionally to examine the waters of the Kent, Southwark, and Lambeth Companies, and he found that the oxygen absorption value was the sharpest method for differentiating

between them. Roughly speaking, the waters derived from the Thames contained five times as much organic impurity as waters like the Kent Company's. He attached no importance to the *number* of organisms present. Any limit must necessarily be an arbitrary number, and after all it was simply a question of whether they were pathogenic or not. What one wanted to know was whether they were organisms which would live at the blood heat. Decided in that way there was a great difference between waters derived from sources like the Thames and the chalk waters of Kent. If one made a plate cultivation and incubated at blood heat, a pure water would show no organisms after 50 hours, whereas the organisms that existed in a polluted water like the Thames or Lea would multiply at that temperature, so that the differences between a good and a bad water were accentuated by such a method, and rightly so. Further he had noticed in the case of deep well waters with gelatine plate cultures that after some days the number of bacteria were increased as compared with the plates from a polluted water, and even in Kent or other hard water a marked increase in their number would appear after a longer period. That was due to the fact that they contained Cladothrix dichotoma, an organism which was very slow in incubating, but would appear in great numbers after five or six days. This might explain the difference found by different examiners. One of the most important points in the paper was the percentages of nitrogen found in the suspended matter, but he thought this information could be ascertained in a simpler way by using a Pasteur filter. Moreover, the yield of such a filter diminished rapidly, and it seemed to him that the diminished yield in a given length of time and with a given head of water might be taken as a measure of the suspended matter in a water. He had no data on the subject, but it was obvious that the rate of diminution must be proportionate to the choking of the filter. One would then take the deposit and subject it to the Kjeldahl method, and so get the percentage of nitrogen. The ratio of the total percentage of nitrogen to the percentage of oxidized nitrogen was a valuable factor in judging whether the water was in a harmless condition. In very pure waters the nitrogen was oxidised to the extent of at least 95 per cent. In the Kent waters the ratio of oxidation was from 95 to 98 per cent., as compared with considerably less in the other London waters. He had not noticed this factor recorded in water analyses and suggested that "the percentage of nitrogen oxidized," or "ratio of oxidation," should always be given as the information was of considerable importance.

Dr. Mott congratulated Mr. Dibdin on his admirable method of the micro-filter. In perusing the paper he had been struck with the similarity between the deposit in the micro-filter of the unfiltered to the filtered water. Did this arise from a defect of the filter or from the accumulation of the material in the mains? He entirely agreed with the last speaker as to the character of the deposit. Micro-organisms were always with us, and fortunately so, for they played an important part in the economy both of nature and of the human body. There was no question, however, as to the danger of the organisms which got into the water rendered impure by sewage contamination, and there could be no doubt that the Thames was so contaminated. They would not be justified, therefore, in accepting the statement often put forth that a twelve mile run of the water would destroy the typhoid organisms contained in it, and that other bacteria being present in the filter prevented these typhoid ones from going through. He did not think that typhoid was due so much to the impurity of the water as contained in the mains as to the unsanitary arrangements in the consumers houses; and if any new system of water supply would lead to a complete eradication of these unsanitary conditions it would be one of the greatest improvements ever effected by any city. Visitors to Vienna were struck by the splendid water supply possessed by that town, and that was simply due to the fact that owing to an epidemic of cholera the Viennese had changed the source of their water supply and had at the same time got rid of the unsanitary conditions existing in their houses. Liverpool obtained its water from Wales and the results were such as justified the action of the town in taking that course. It would be of interest to ascertain with the micro filter the number of bacteria in this water. He quite agreed with the remarks of the last speaker on the bacteriological aspect of the question; particularly he advised culture on Aga Aga at the temperature of the body.

Mr. W. J. Dibdin, in reply, said that Mr. Burgess had referred to the amount of deposit in relation to the length of mains traversed, and seemed to think that the longer the main the greater the amount of suspended matter. He had since looked into that matter and could find no ratio. When he commenced his investigations, his ordinary qualitative examination for the suspended matter always showed a considerable quantity to be present which was verified by subsequent measurement. But when it became known that he was taking that course a considerable reduction took place, and at present working under precisely the same conditions he could hardly find any. He mentioned that as a simple fact and did not see that it had anything to do with the mains, many of which had run so long that if they gave merely an infinitesimal quantity of matter to the water they would have been dissolved away long ago. With regard to the Welsh river water he had been anxious to make further examinations but had not had an opportunity. The figures were the average of five samples taken in May. He was not prepared to say that they would not have been better at one time and worse at another; he gave them for what they were worth, merely pointing out that they represented an unfiltered water. He did not say that it was absolutely necessary to soften the London water supply; but he did think that it would be of advantage from the point of view both of comfort and economy. His friend, Mr. Bevan, reminded him of the fact that while lime was worth only 14*s*. per ton, soap was worth 20*l*. That was a fact worth reflecting upon. If the course he had suggested was adopted, and London water was softened, the difference between it and Welsh water would be practically nil—a most important matter. With regard to the suspended matter, Dr. Frankland, who was respected by no one more than by himself, used to report occasionally "moving organisms," he seemed to get tired of the phrase; then "bacteria" came into the field, and moving "organisms" moved off. But suspended matter was not even referred to by two eminent authorities who reported on the quality of the London water on behalf of the water companies. That seemed to him to be regrettable. It was perfectly true that the *average* of suspended matters was so small that it was almost negligible. But the moment of danger came when the water was in its worst condition (just as the strength of a chain was that of its weakest link), and one could only judge the safety of the community by the water supply at its worst, and he maintained that the weakest link in the water supply of London was the amount of suspended matter; for speaking generally the number of bacteria did bear a relation to the quantity of suspended matter. That was in accordance with Dr. P. F. Frankland's results and his own, and reasonably so. If that was the case why was it neglected? he was sorry that men who were looked up to as leaders of the scientific world did neglect such an important fact. They had analyses in official reports giving results to a thousandth part of a grain of carbon and nitrogen; but when they had, not $\frac{1}{300}$ grain but $\frac{1}{30}$ grain of suspended matter loaded with bacteria it was not mentioned. That was not right, and in pointing it out they had done useful work. They had put the companies on their guard against a danger of which they appeared to be unaware. He had no animosity against them; he admitted that the moment they were made aware of certain objectionable things they met the position and remedied the defect. If, however, they got into a comfortable state again they might recur to $\frac{1}{37}$ grain of suspended matter per gallon again, or even—as it doubtless was in 1892 or thereabouts—about $\frac{1}{10}$ grain per gallon. Mr. Thorp had inquired as to the quantity used in obtaining the weight in suspended matter. The determinations were made upon samples of about 200 gallons. A stream of water was allowed to run on to a large filter at such a rate that the filter did not overflow. At the end of the day the filter was washed down with a fine jet and the deposit collected in a small bottle, the deposit being sterilised

at once with a little bichloride of mercury. Next day they proceeded in the same way. Thus in 10 days about 2,000 gallons would have passed through, and thus they had a very considerable quantity of deposit to work on. The miscroscopical examination of some samples was very startling. Some resembled "flue" as if swept up from a floor. This could not have come from the mains, for it was both fresh and decomposed organic matter, as to the method of stating the total suspended matter in terms of tons that might be open to the charge of extravagance; but it had served in calling attention to the fact that these in the aggregate were considerable. It might not matter to Mr. Thorp but to some people it was very important to have facts in the language of everyday life that they could understand. Mr. Thorp had also referred to the Fitzroy river. The fact was that some years since, wishing to get a factor to represent the character of suspended matter in a river absolutely uncontaminated, he had asked a brother living in Queensland to collect some samples, which he did in conjunction with the curator of the local museum, using the precautions laid down by himself, these were properly dried and sent to him direct. Dr. Voelcker thought the micro-filter failed to distinguish between quantity and quality. Of course it did. It was simply a useful means for collecting suspended matter for subsequent examination by the microscope. As to the varieties of bacteria, he did not pretend to do more than count the colonies. As to the results obtained in the years 1891–92 and in 1894–95, they were arrived at in exactly the same way. But the numbers of bacteria found at that time were very different, and the Royal Commission came to the conclusion that some of the filters used were very unsatisfactory and required improvement, as much sewage was at that time going into the river. He had answered most of Mr. Blount's points already. As to standardising the micro-filter, when Mr. Blount had done more work with it he would probably abandon the idea of standardising the thickness of the walls of the glass tube. In respect to the correction for rust and dust, however, Mr. Blount had touched upon a point of great importance. The correction made was on the assumption that the suspended matters or "rust and dust" in the Kent Company's waters was accidental. He was by no means sure that it was unavoidable, for at the present time the company was avoiding it, and he had only used the expression because he was anxious to give the companies the benefit of the doubt. As to risk from the existing supply, he did not think there was much to fear as long as the companies kept things up to their present standard; but again it was a question of the strength of the chain at its weakest link, and they must bear in mind what might happen at some future time if matters were not looked after. Things were in a very good condition at present, but he quite agreed with Mr. Whittaker that whoever was in charge of the water supply it was necessary to have the policeman constantly on the beat. He quite admitted the force of Mr. Hooper's remarks about the small quantity of suspended matter consumed per head per day; but the same argument applied equally to the carbon, nitrogen, &c. Again, referring to the deduction for what he had called "unavoidable rust and dust" in the Kent waters, he would ask how it happened that anguilulæ occurred in it? Was it from dust in the reservoirs or from dirty mains? There was, in fact, something mysterious at times about the Kent company's water which he could not explain, and had an itching to investigate. As to the loss on ignition there was only one way of determining it, and he thought Mr. Thorp might have given him credit for using that process with the usual care. The solids were dried at 100° C., carefully ignited at a low red heat, and re-carbonated with ammonium carbonate. Then as to the microscopical statements, Mr. Bevan seemed to think that he was pushing matters too far in saying that there was a ratio between the microscopical and the chemical results which fairly agreed. Of course there were isolated cases in which the lines crossed, but it was remarkable how close the agreement was. It must be remembered that this was the first time that the microscopical examination had been put into numerical form, and at the best this could be but an assumption. If the percentage of objectionable samples were calculated into weight as representing a definite proportion of the total known to be present, and these results formed a fairly uniform curve with the chemical factors, there must be some basis of agreement between them. Doubtless the microscopical examinations would be those most needing revision, as they were not at first made with the intention of working the observations into a numerical statement.

Mr. Bevan: You cannot average discordant figures.

Mr. Dibdin replied that with reference to the micro-filter it was clear that 1 m.m. deposit in the micro-filter per litre, equalled from 0·002 to 0·005 grain per gallon in the case of the filtered waters, or an average of 0·004. In the case of the unfiltered river water containing a relatively large quantity of mineral matter, sand, &c., the ratio of weight to volume was naturally higher, *viz.*, 0·06 per mm. than in the case of the lighter organic matter in the filtered water. If the process gave an indication which could not be got otherwise, surely it was a useful one. It was already contended that the difference of a few thousandths of a grain is of no practical importance; why then cavil at such minute differences as these? By it he had been able to point out defects in well waters which he had suspected, but could not be certain about before. Dr. Dyer had objected to the examination of water from the mains instead of the filter beds. Of course he had no power to go to the companies' works for his samples, and questioned whether they would have granted permission. The question was not what came from filter A or B, but what they supplied to the consumer. Some filters might be working well while one might break down entirely. His method tested the average quality of the supply, and that was the main question. As to the collection from the stand pipes, it was made most carefully, the bottles used being thoroughly cleansed.

Mr. Groves inquired if they were sterilised.

Mr. Dibdin, continuing, said that they were sterilised with strong sulphuric acid, and the samples for bacteriological examination were taken in 2 oz. bottles, sterilised at a temperatue of 400°, and many hundred check experiments were made under varying conditions, many plates being inoculated on the spot. As to the *amount* of suspended matter being unimportant, that was quite true, but where one found a considerable quantity of decomposing matter, hairs, fibres, various infusoria, and colonies of bacteria these were at least objectionable.

ON A RELATION OF COLOUR TO VALUE IN MALT.

BY JOSEPH W. LOVIBOND.

The method of estimating the quality of malt to be described is not intended to, nor can it replace the analytical precautions now in use, it is intended to supplement the present methods by providing a means of estimating those subtle properties which, whilst materially influencing the flavour, brilliancy, and keeping properties of ale are yet too subtle in their nature to be isolated, weighed, or even named.

The estimation of malt values does not always receive the attention it merits, considering the large quantity of valuable stock which must accumulate in the brewery between the first purchase of the malt and the final judgment of the finished beer, and in cases where the finished beer is not all that can be desired, the responsibility of placing blame on the malt is not a light one, the whole process of brewing being a succession of conditions each of which has an influence for good or for evil on the final product.

Another difficulty in judging malt arises from experts not yet being fully agreed as to the exact nature and value of some of the malt constituents: for instance, certain soluble substances are sometimes classed as unknown bodies and sometimes as sugars, these substances frequently vary from 4 to 6 per cent. and as their function is undetermined, an element of uncertainty is introduced which tends to confuse the judgment of the practical brewer.

In addition to this a high percentage of diastase is usually considered an index of good value, and this may be

so when a large proportion of starch material is used, requiring for its conversion a great excess of diastatic power in the malt, but in those cases where malt alone, or only a small proportion of starch material is used, the advantage of high diastatic power is not so evident, it is even possible that in some cases a high percentage of diastase may be an index of low value, as affording presumptive evidence of a low temperature on the kiln.

Supposing that all questions of analytical apportionment were settled, there still remain to be dealt with those intangible substances already mentioned.

The method of estimating the effect of these substances on beer is based on a new law, which I call the law of specific colour absorption, the fundamental principle of the law being, that each definite substance has a power of absorbing definite rays from impinging white light, this power being constant so long as the substance itself remains unaltered.

The method of establishing a colour constant is to measure the composition of the light the substance reflects or transmits, and then to obtain the absorbed rays by difference.

A single measurement does not become a colour constant until associated with the density of the substance at which the measurement was made, and then only applies to that particular density. In order to establish the specific rate of absorption which is to be our future factor of value successive measurements must be made at regularly increasing densities, and the results arranged in a table for comparison, or more graphically plotted into curves.

It is evident that the value of the method depends on the definition of the term "white light," and on the accuracy with which the unabsorbed rays can be measured, both these points have been fully demonstrated on several occasions before this Society, and are moreover accepted as valid by those investigators now using the method, I do not therefore purpose going into these details this evening further than to recall one experimental fact, which is this, that any colour no matter how complex can be quantitatively described by means of two colour terms and one light term.

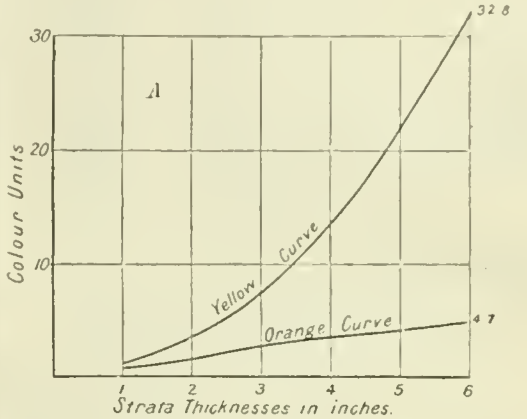
This experimental fact is the outcome of a law which possibly has not received the attention its importance merits, the law being that the colour of a beam of light depends on the one preponderating ray if the colour be pure, and on the two preponderating rays if the colour be complex. That the depth of colour is in proportion to the preponderance of the one or the two rays, and that the tone of the colour is dependent on the quantitative relations of the two preponderating rays towards each other. This holds good in all cases within the power of colour perception by the vision, whether the coloured beam be reflected from a mixture of pigments or whether it be produced by a mixture of coloured lights. Postponing for the present the consideration of the light term, *Yellow* and *Orange* are the two colours which in one proportion or another apply to the description of colour in all malts and malt extracts. It is the varying proportion of these two colours which, when co-related to the beers they produce supply the means of estimating the quality of the malt so far as the unknown constituents are concerned.

The method adopted for preparing the samples for colour measurement is to mash 100 grms. crushed malt in a 500 c.c. flask with 350 grms. of water at 74 c., digest in a water-bath at 63 c. for 48 minutes, cool and make up to 500 with water, filter, and measure, beginning with a 1-in. stratum and progress at 1-in. intervals up to 6-ins., when the colour becomes too dark for reliable reading except in the case of very pale malts, the two colour terms may be tabulated in their numerical values, or plotted into curves, the colour units supplying the ordinates and the densities the abscissæ.

On comparing a number of malt specific colour curves with the ales they produced, it soon became evident that the best results were associated with a high proportion of yellow over orange, and inferior results with a low proportion of yellow over orange, this was so constant that the proportion of yellow to orange is now adopted as an index for valuing pale malts.

The two examples, A and B on Fig. 1, used to illustrate the co-relation of colour differences in the malts, with the differences in quality of their finished ales, were selected from a number of similar instances, because they were the last experiments of a series, and they could also be associated with their chemical analysis by Mr. Heron.

Fig. 1.



Specific Colour Curves of two Samples of Malt (A and B).

The ale brewed from the malt marked A, brightened readily, was fine flavoured, and improved by keeping; that brewed from the malt marked B, brightened with difficulty, was inferior in flavour, and did not improve by keeping; there was nothing in the appearance of the malt to suggest any real difference in quality, nor does the chemical analyses appear to suggest a sufficient explanation of the wide difference between the quality of the ales produced.

On comparing the specific colour curves of A with those of B on chart 1, it will be noted that the initial of yellow for A at 1 in. is 0·5 of a unit more than B, whilst it terminates at the 6-in. stratum 2·6 units more than B, showing a higher rate of increase in the yellow for increasing densities. In the orange curves, A is 0·5 of a unit less than B at the initial inch, and 1·9 units less at the terminal 6 inches, showing a slower rate of increase in the orange for increasing densities, fully bearing out previous experiences as to the co-relation of a high proportion of yellow with fine qualities in ales, and the co-relation of a high proportion of orange with inferior qualities.

In estimating the proportion of the two colours towards each other, the variations in the curves of different malts became somewhat confusing, and it was found more convenient to plot a complementary curve showing the varying colour proportions in a single line. The ordinates for this complementary curve are supplied by the number of times the orange is divisible into the yellow at each measurement, and the abscissæ by the stratum thickness of each measurement, the variations in the curve then exactly represent the

variation of proportion between yellow and orange in the malt extract.

Mr. Heron's Analyses of Two Malts.

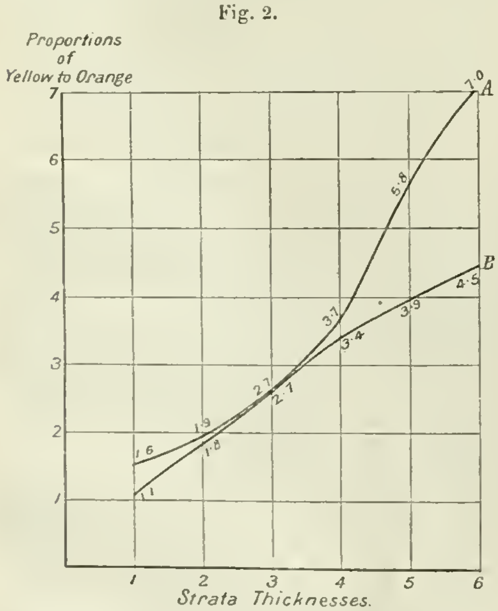
——	A.		B.	
Dextrin	9·51		14·87	
Maltose	43·91		35·52	
Invert sugar	3·14		5·02	
Cane „	2·58	59·14	2·62	58·03
Albuminoids (soluble noncoagulable).	2·51		2·86	
Acids (aslactic)	0·22		0·28	
Mineral	1·02		1·64	
Moisture	5·76		3·83	
Unknown bodies	5·67		4·50	
Grains by difference	25·68		28·86	
	100·0		100·0	
Extract in lbs. per qr.	91·08		91·90	
Diastatic capacity	40·0		25·0	

Colour in Tintometer Colour Units.

——	A.		B.	
	Orange.	Yellow.	Orange.	Yellow.
1-in. stratum	1·0	+ 1·6	1·65	+ 1·75
2 „	1·9	+ 3·7	2·8	+ 5·2
3 „	2·61	+ 7·0	3·6	+ 10·4
4 „	3·44	+ 12·8	4·7	+ 16·3
5 „	3·9	+ 22·2	5·6	+ 22·2
6 „	4·7	+ 32·8	6·6	+ 30·2

The complementary curves of A and B are shown together in Fig. 2; there is an important difference in their type, as they curve in opposite directions, A curving upwards and B downwards.

Fig. 2.



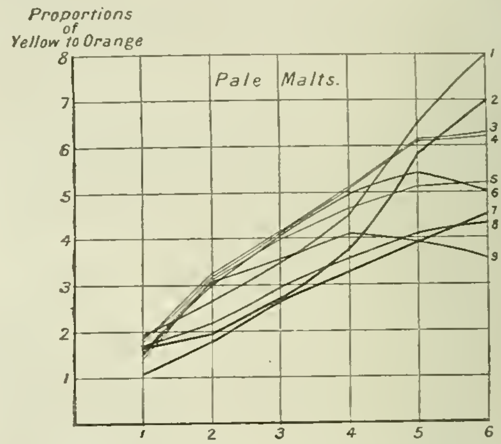
Secondary Curves showing Proportions of Yellow to Orange in A and B Malt Solutions.

A comparison of these two curves show that the measurement of a malt extract at a single density does not give a sufficient basis for a reliable estimation, it may even prove misleading, as in the examples before us, where two malts widely differing in quality give similar proportions of yellow to orange at the 3-in. density where the curves meet, but diverge at all other points.

I am only prepared this evening to deal with the curves of pale ale malts, and for this reason it was found necessary to establish a separate normal for the higher dried malts, as the type of specific absorption curves alters in these malts, appearing to have some relation to the temperature of the kiln, until more work has been devoted to the co-relation of quality to the new specific curve, little more can be said on this part of the subject.

How necessary this co-relation of colour to values is will be realised on examining the collection of nine complementary curves of pale malts on Fig. 3, with the six complementary curves of higher dried malts on Fig. 4; they are arranged and numbered in their order of merit as determined by the rule of yellow proportion over orange already defined.

Fig. 3.



Secondary Curves showing Proportions of Yellow to Orange.

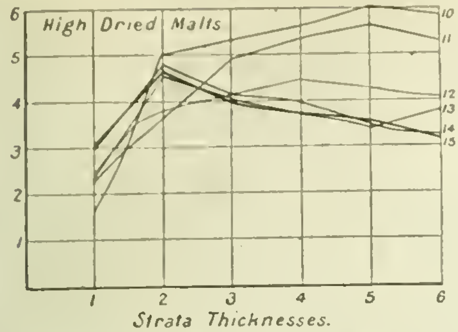
First, dealing with the nine complementary curves of pale malts in Fig. 3, the two extremes are distinctly different in type as they curve in opposite directions, and according to our rule of procedure No. 1 represents the highest type and No. 9 the lowest type. It will be seen that No. 2, the second in quality, is the specific curve of the A malt, which was chosen as an example for illustration, and was known to produce excellent results in the finished ales, in this way confirming the co-relation of fine quality with this particular form of specific absorption curve.

It will be also seen that No. 8 is only one degree better in quality than the worst example No. 9, and was known to produce unsatisfactory results, confirming previous experience as to the co-relation of this type of curve in pale malts of inferior quality. I may say that when these curves were selected as examples there was no previous knowledge as to the position A and B would take in the scale; this was only revealed after the charts were made.

Accepting Nos. 1 and 9 as the result of two distinct methods of malt curing, it is quite easy to suppose that the intermediate curves may be mixtures of these two methods brought about by uneven management on the kiln or, if the kiln had been evenly managed, it is quite as easy to imagine that the intermediate curves are specific for the particular stage of curing where one type was merging into the other. This, however, is pure speculation, the question can only be settled by actual experiment with malt during the process of curing.

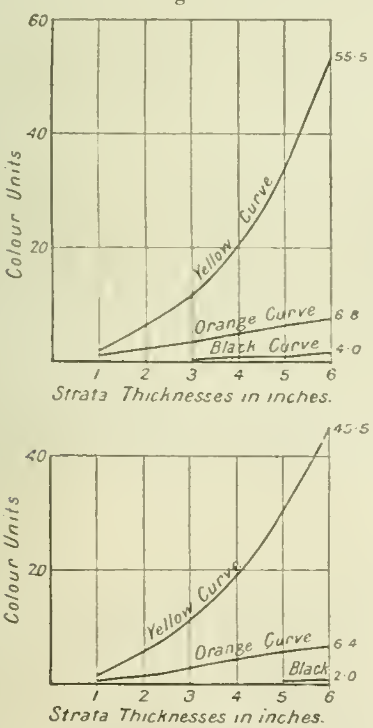
Although I do not now propose dealing with the higher dried malts on chart 4, as the co-relation of qualities to curves has not been yet established, it will be observed that there is here also a change of curve character in the highest dried malts Nos. 13, 14, and 15, and necessarily the intermediate curves are open to the same contingencies of uneven drying. A reference to the table of measurements will show the degree of colour of each malt extract at every density.

Fig. 4.



The consideration of the light factor already spoken of as one of the three terms necessary for quantitatively describing some complex colours, was deferred because this part of the question as applied to malt extracts is still under investigation. The factor itself does not frequently occur in malt extracts, and is sometimes found in malts which are otherwise satisfactory. It is represented by two examples on Fig. 5 by the black line beginning in one instance at the 3-in stratum, and in the other instance at the 5-in. The term used to indicate the presence of this factor is black and the effect of its presence is to dull the brilliancy of the colour according to its proportion.

Fig. 5.



Specific Colour Curves of two Samples of Malt showing Black Absorption Curves.

It is not yet decided whether this black factor causing a decreased brilliancy arising from a slightly less effective filtering, or whether it is an inherent property in some barleys. Should the latter prove to be the case, it would be analagous to the loss of brightness found in wheaten flour made from imperfectly ripened wheat.

Discussion.

Mr. E. Grant Hooper thought that the paper could only be adequately discussed after careful consideration when in print. There were, however, a few points on which he would like information and on which he might offer something in the nature of criticism. In the first place, had the facts been isolated? They had been told that the beers produced by the two malts of which particulars had been given were in the one case good, and in the other unsatisfactory, and they were asked to conclude that these results were entirely due to those differences in the malt which were indicated by the colour analysis of the wort they respectively yielded. It seemed to him that two classes of malt had been dealt with, which on brewing would result in two distinct classes of wort, each yielding a different proportion of alcohol. The tabular results before the meeting, headed "Analysis of Malts," he understood indicated not the composition of the malt but of the wort or extract yielded by the two samples. Here they found that in one case the maltose was 43 per cent., and in the other 35 per cent., and that the dextrine was in the first instance 9 per cent. as against 14 per cent. in the other. It followed, therefore, that in one case there would be a greater attenuation with the production of a larger proportion of alcohol than in the other, and if up to the end of the primary fermentation other things were equal, two beers so different would exhibit different degrees of stability. It was necessary to bear these facts in mind before speaking of the soundness of a beer derived from such malt. But in connection with the comparison in the matter of soundness he should like to hear from Mr. Lovibond that the precaution had been taken to use the same yeast. If the experiments were made at different times it was quite possible that the instability in the one case was due to the yeast. He would suggest as an explanation of some of the differences found that the orange colour was a product of caramelisation. From the colour curves he judged that different proportions of caramel would be found, and would suggest that the difference in appearance represented the precipitation of a caramel body. So far as he could understand, the curves meant that the lighter the colour of a malt, provided it were completely dried, the better the malt was. Was there evidence that these colour constituents indicated intrinsic soundness, or did this orange colour merely point to the presence of caramel constituents with a tendency to fall out of solution under certain conditions? The more highly coloured a malt was the more the diastase would be affected, and the less complete would be the conversion with a resulting variation in the composition of the beer produced. Had Mr. Lovibond taken care to eliminate the effect of this last difference, and secured such a uniform basis as would permit a reliable comparison?

Mr. Bryan Corcoran had had considerable experience in the construction of malt kilns. He also had some acquaintance with the tintometer and quite believed in its tests, but he would like to know what the author meant by the curing of malt, and whether the quality depended on the quality of the barley or upon the way in which it was malted, and also whether the length of steeping had anything to do with it. Some people steeped for 40 hours, others for 60 hours. On the question of drying, Mr. Lovibond had spoken of the temperature in the kiln. He thought that was not a very sound way of estimating the drying, as it was possible to have a kiln at a high temperature with very little draught, or one with a good draught and a low temperature, the latter generally producing the best malt. The colouring of the malt was a very difficult question to understand. Mr. Lovibond had spoken of the highest dried malt. Did he mean the malt with the least moisture in it? He had found that the lower temperature generally produced the best results and therefore gave most satisfaction?

Mr. E. S. Beaven had found the tintometer of great use in keeping a check on the drying of malts. He was not prepared at that moment to criticise the results, but if they proved to be corroborated by further investigation, they would undoubtedly be of the greatest possible advantage. He would venture to suggest, however, that it would be a great gain if in the place of what was usually known as "Series 52" of Mr. Lovibond's tintometer, the series which was used in recording the tint depth of the caramel colour and of malt extracts, determinations were

made in the standard primary colours. Mr. Lovibond had not that evening referred in any way to that series of standard glasses in which the colour tint of malt extract was usually quoted by analysts. He had standardised that series himself with the red and yellow, giving the orange and yellow curves which Mr. Lovibond had referred to, and he found that as the series progressed upwards the ratio of yellow to orange increased, following in that respect what happened with normally dried malts, and also with different densities of caramel. If, however, it became customary to state and tabulate the colour of malt in terms of the primary standards, it would, he considered, facilitate the elucidation of the colour analysis of malts. The single standard afforded nothing on which to draw any deductions, but a combination of the red and yellow glasses would do so. He had before known of the truth of what Mr. Lovibond had stated, *viz.*, that it was possible to match any transmitted or reflected colour by means of the standard glasses, and it was tolerably evident that irregularities in the drying of the malt were made apparent by the different character of the tints which were revealed. If measurements were taken with those glasses considerable benefit might accrue and the results be further tested.

Mr. J. Heron had listened with very great interest to the paper, because it opened up a new field in connection with the analysis of malt, caramel, and sugars, the colour of which consisted solely of a combination of red and yellow in varying proportions, its principal use hitherto in a brewer's laboratory appeared to have been exceedingly limited, being employed merely to determine the relative depths of colour of malt extract, sugar solution, &c., but this operation, simple as it was, showed a very great deal about the way in which the malt had been dried and cured on the kiln. The figures so obtained, and compared with the diastatic capacity of the malt, would tend to show the operations to which that malt had been subjected from the time it went on to the kiln to the time it came off, and he claimed that those determinations threw a good deal of light upon the history of a malt from the time it went on the kiln until it came off, but this was as far as they could go at present. Mr. Lovibond drew his attention two years ago to the bearings which the combination of red and yellow had upon the quality of black malts, and it occurred to him afterwards that those observations might be applied to pale and high-dried malts as well. If an analyst carefully examined the colour of malt extracts by the scientific method brought forward by Mr. Lovibond, he would obtain a good deal of information as to the quality of the malt he was dealing with. He had invariably found, and in that he was corroborated by the observations of those brewers who had used the tintometer in the manner indicated, that those malts whose extracts showed a large proportion of orange as compared with yellow, produced beers which did not turn out satisfactory. How did the tintometer values correspond with the values ascribed by the chemist from an analytical point of view? He was fully convinced that there was some close connection between them, but he should not like to say anything about that just yet, because he did not consider that sufficient experiments had been made in this direction. So far, however, there was a decided connection between the results as revealed by analysis and the results revealed by the tintometer, and he could only hope that it would be Mr. Lovibond's, and perhaps his own, pleasing duty at some future time to give the results of investigations in that direction. He had had nothing to do with the preparation of the paper just read, but there were two or three points in it on which he begged to differ from Mr. Lovibond. He could not agree with him in carrying on his mash at such a high temperature. He had found that a cold water mash and one mashed with hot water under standard conditions (*i.e.*, at 65° C.), provided the extract was not boiled afterwards, would give practically the same results in the tintometer. The colour of the malt was not increased by mashing in water at 65° C., except perhaps, to a point that was negligable. He had no doubt that although the total units of colour obtained by a hot mash were the same as those obtained by a cold mash the relative proportion of orange and yellow might be altered to a slight extent, and he would therefore suggest that whatever be the density used the mash should be carried on with cold water at, say, 60° F. There were a great many small differences which would creep in when making a mash at a high temperature, and which could be avoided by making a mash with cold water. The temperature of 74° C. was a point at which the diastase was considerably crippled, and they might look upon 75° to 80° as the critical temperature. 74° was also very much higher than was usually employed in mashing operations on a commercial scale. He would prefer making a mash at 65° C., equivalent to about 150° F. Mr. Lovibond had referred to the black factor creeping in in the case of certain malts. He had found that with some malts mashed under standard conditions at 65° C. a brilliancy could be obtained in the beer with the greatest ease, while with others it was impossible to eliminate a certain cloudiness which was more easily revealed by examination in the polarimeter than in any other way. This was a very good index as to the brightness of a solution, because although the solution might appear to be absolutely brilliant to the naked eye, yet, when examined in the polarimeter, the slightest degree of cloudiness would be revealed. In a thin strata of liquid this peculiarity would not be perceived, but in a strata of 6 inches or more it would appear, and would indicate certain bad qualities in the malt, but what those bad qualities were it was impossible yet to say. No doubt the lines which Mr. Lovibond had taken up in the paper, and the field of further research thrown open by him, would be productive of great benefit, and when brewers on these lines began to

TABLE OF SEPARATE MEASUREMENTS.

Numbers of Samples.

—	1.	2.	3.	4.	5.	6.	7.	8.
	Orange. Yellow.	Orange. Yellow.	Orange. Yellow.	Orange. Yellow.	Orange. Yellow.	Orange. Yellow.	Orange Yellow.	Orange. Yellow.
1″	1·15 + 1·8	1·0 + 1·6	1·2 + 1·8	1·6 + 2·6	1·5 + 2·3	1·32 + 2·15	1·0 + 1·6	1·65 + 1·75
2″	2·3 + 5·3	1·9 + 3·7	2 5 + 5·5	2·7 + 8·8	2·6 + 8·2	2·2 + 6·4	1·9 + 4·0	2·8 + 5·2
3″	3·2 + 11·2	2·61 + 7·0	3·0 + 11·0	3·8 + 16·2	3·9 + 15·6	3·5 + 14·5	2·6 + 7·4	3·8 + 10·4
4″	4·3 + 20·0	3·44 + 12·8	4·3 + 19·5	5·1 + 36·9	5·0 + 23·9	4·32 + 21·5	3·3 + 13·0	4·7 + 16·4
5″	5·3 + 35·4	3·9 + 22·2	5·2 + 31·5	6·6 + 40·4	6·2 + 32·2	5·22 + 27·2	4·0 + 16·6	5·6 + 22·2
6″	6·8 + 55·0	4·7 + 32·8	6·4 + 45·5	8·4 + 52·6	7·8 + 41·2	6·65 + 33·2	4·7 + 21·0	6·6 + 30·2

—	9.	10.	11.	12.	13.	14.	15.
	Orange. Yellow.	Orange. Yellow.	Orange. Yellow.	Orange. Yellow.	Orange. Yellow.	Orange. Yellow.	Orange. Yellow.
1″	1·8 + 3·2	2·3 + 5·9	2·2 + 5·2	2·1 + 5·3	2·6 + 6·4	2·2 + 6·0	2·7 + 8·1
2″	3·0 + 9·5	4·14 + 20·7	4·2 + 15·3	3·7 + 13·5	4·18 + 19·6	4·2 + 19·0	5·0 + 23·6
3″	4·5 + 16·1	5·9 + 31·8	5·8 + 28·5	5·8 + 24·6	6·54 + 27·0	6·81 + 29·0	8·0 + 32·8
4″	5·6 + 22·4	8·3 + 48·0	7·8 + 42·5	7·3 + 33·0	9·34 + 36·0	9·1 + 36·4	11·0 + 42·5
5″	7·2 + 27·8	10·3 + 61·5	10·0 + 56·0	9·3 + 41·0	13·1 + 46·0	12·22 + 43·0	14·2 + 49·02
6″	9·0 + 33·0	11·4 + 67·5	13·0 + 69·0	11·8 + 48·5	15·25 + 59·0	15·55 + 52·5	17·7 + 59·0

make a periodical examination of their malts they would find that by a simple operation of mashing and examining the resulting wort in the tintometer, they would after a short time be able to predict what the character of the resulting beer would be like. A great advance in our knowledge of some of the characteristics of malt would then be made, and the thanks of the brewing world generally would be due to Mr. Lovibond for initiating research in what must be regarded as heretofore unbroken ground.

Mr. William Fyffe said that he gathered from Mr. Heron's remarks that he had considerable faith in the correctness of Mr. Lovibond's theory, *viz.*, that the quality of a malt could be judged by the ratio of yellow to orange. Mr. Lovibond in his paper had told them that he based his theory upon many experiments, and found that the best beers were produced from malts where the tintometer showed a relatively high ratio of yellow to orange. Under these circumstances he wished that brewers' analysts, instead of telling them simply the tintometer reading 4°, 5°, 6°, or whatever it might be, would state the ratio between the yellow and orange, so that brewers might have an opportunity of testing the correctness of the theories enunciated by Mr. Lovibond. The tintometer was becoming a valuable instrument to brewers, who were finding out that the quality of a malt could be judged by the ratio of tintometer reading to diastatic capacity. He himself had found that a malt having a tintometer reading of 3°, combined with a diastatic capacity of 42°, did not produce so satisfactory a pale ale as a malt having a tintometer reading 4°, combined with a diastatic capacity of 37°; the former giving a beer more liable to frets and wild yeast attacks in summer, and not fining so readily as beer produced from the latter. Perhaps the gentleman who had previously spoken would say that the condition of his yeast may have had something to do with the difference noted in the quality of the respective beers. But the experience of other brewers led him to believe in the correctness of what he had stated.

Mr. J. W. Lovibond, in reply to Mr. Hooper's first question, said that he need hardly assure the members of that Society that every care was taken to have the conditions of the experiments identical; in fact, they were extracts taken from their usual work. This series extended over three months, and embraced twenty times as many examples as had been shown in the charts. Those shown were not selected for any particular reason, except that they were the last of the series, and could be accompanied by the chemical analyses. The colour measurements had nothing to do with Mr. Heron's analysis, and were independent results. With reference to the standards 50 and 52, they were arbitrary standards, and were simply the nearest glasses which, in the early days of the tintometer, he could get in accord with the caramels and malts. They fairly met the case, although they were not scientific, and were not based on the equivalence of the six colour rays in a beam of white light, as the new standards were. He should like to see a change made from the 50 and 52 series. They had answered the purpose up to the present time, but the accumulation of facts showed that they did not meet the requirements of present discoveries. The mash was made as nearly as possible under the conditions obtaining in brewing. The temperature might be high, as Mr. Heron had stated, but in dealing with small quantities there was always a loss of temperature in manipulation. No diastatic destruction took place, because, in fact, the extracts obtained were such as the malts might be expected to yield. In reference to Mr. Corcoran's question as to the quality of barley and malt, and the drying: the malt made by his firm was from barley of good malting quality: the other samples of malt were from good makers, tendered for sale in the ordinary course of business. The drying was a very important problem, however. For his own guidance he had divided the kilning process into two portions: the first he called the drying, and the second curing. The curing was the key to the whole position, and on the care with which it was carried out depended the proportion of yellow and orange, which he had no doubt whatever was an index to high quality.

Mr. Fyffe asked how many days it was on the kiln.

Mr. Lovibond replied that he had assumed that the malts were fairly made on the floor, and went to the kiln in the usual workmanlike manner; four days was the usual time, but subject to some variations under the exigencies of sudden rise in temperature. With reference to the colour in the high-dried malts, one might get an average colour depth with desperately badly cured malts, burnt in one corner and uncured in another. If mixed together, an average tintometer result may be obtained, and it was not until comparing the proportions of the curve that the true history of the curing was shown. He could take a dried malt and say without hesitation whether the curing had been uneven or not, from the proportion of orange and yellow. Mr. Beaven had spoken of the desirability of replacing the arbitrary standards by the scientific standards. With that he quite agreed, and thought that until it was done it was not possible to compare different results. In reply to Mr. Heron's suggestion of a cold-water mash: it did not matter what process was adopted if the results were similar; and if different, the only difficulty which could arise would be that a separate normal would have to be established for each process. If cold-water extract gave a similar result to the hot-water extract, there would be no necessity for establishing a separate normal. Mr. Fyffe had touched on a most important point when he spoke about unsatisfactory beers. The old tintometer standards would not discriminate between the qualities of the malt, but would simply give the depth of colour; and whether that was arrived at by the quality of the barley or the drying, it would not indicate. If the malt mentioned had passed through his hands, he could have told by the nature of the absorption curve whether the results would be likely to prove unsatisfactory, and whether the malt had been unevenly dried.

HEHNER'S BROMINE TESTS OF OILS.

BY J. H. B. JENKINS.

The following comparative results were obtained by the bromo-thermal and bromo-gravimetric tests of oils, the experiments being conducted under the conditions laid down by Hehner and Mitchell in the *Analyst* of March and July 1895. The figures are compared with the iodine-values of the oils as determined by the Hübl process:—

For the bromo-thermal tests a vacuum-jacketed tube was used. The iodine values of the oils are derived by multiplying the rise of temperature in degrees C. by 5·7, the factor for the particular tube, &c. used in the experiments. In the gravimetric process the percentage increase of weight of the oil after bromination, *i.e.* the bromine value, is converted into the iodine value by using the factor 1·587 ($= \frac{127}{80}$).

In the gravimetric experiments the brominated oils were dried first for 5 hours in the water oven, say at 97° C., and afterwards for a further 10 hours in an oil oven regulated to 125° C., successive weighings being taken after each hour's exposure. The deportment of the oils during drying is shown in the following table, in which the weights found after stated intervals have been calculated to iodine values:—

Oil.	Iodine Values.					
	Hübl.	Bromo-thermal.	Bromo-gravimetric.			
			Dried at 97° C.		Dried at 125° C.	
			3 hrs.	5 hrs.	5 hrs.	10 hrs.
Rape.........	100·2	98·6	99·8	99·0	96·8	95·5
Raw linseed .	174·3	173·9	171·1	168·8	161·8	156·9
Boiled linseed.	166·6	166·7	169·9	166·2	157·5	154·9
Castor	84·1	83·8	88·8	86·9	80·0	77·2

In the bromo-gravimetric estimations, the figures derived from the increase of weight after 3 and 5 hours' drying at 97° C., when the chloroform and excess of bromine may be assumed to have been expelled, agree fairly well with those obtained by the Hübl and thermal processes. The hourly weighings, however, showed a steady decrease of

weight throughout the whole exposure, and even after the 15 hours the loss in the case of the rape oil was 3 mgrms., which, on the 1·5 grm. of sample taken, corresponds to a loss of 0·3 in the iodine value per hourly weighing. The loss in the case of the other oils was somewhat greater. It was difficult, therefore, to ascertain when the oils had approached sufficiently nearly to a constant weight to permit the factor being applied to convert to the iodine value. The brominated oils do not seem to be quite stable at the drying temperatures.

In the following table the thermal iodine values of a series of oils are compared with the Hübl values. The specific temperature reaction figures (derived from the Maumené tests) are also attached :—

Oil.	Iodine Values.		Specific Temperature Reaction.
	Hübl.	Bromo-thermal.	
Blown rape (sp. gr. 0·9621)..	55·3	74·1	153
Blown cotton (sp. gr. 0·9639)	62·2	78·1	164
Neatsfoot	71·6	72·4	87
Olive	80·1	81·5	94
Bottlenose..................	80·6	84·2	103
Castor	84·1	83·8	105
Rape	100·2	98·6	130
Ravison rape................	109·9	114·0	162
Seal	135·5	140·5	278
Japanese wood (sp. gr. 0·9385).	165·7	133·4	372
Boiled linseed	166·6	166·7	248
Raw linseed	174·3	173·9	313

It will be seen that there is a general agreement between the Hübl iodine values (column 1) and those calculated from the thermal rise on brominatian (column 2). Pronounced exceptions are found, however, in the case of the two blown oils and also in the case of the Japanese wood oil. These oils also form exceptions in the specific temperature reactions, which otherwise generally show increasing values as the iodine values rise. Check determinations of these three oils were made by both the Hübl and thermal methods, but the fresh results tallied closely with the earlier determinations. Thinking that the abnormal calculated iodine values for these oils may really correspond to abnormal bromine values which in these instances may not be comparable with the iodine values, further tests were made with them by the bromo-gravimetric method. The percentage increase of weight, after bromination and drying for 5 hours at 97° C., was calculated to iodine values, and gave for the blown cotton oil 83·6, for the blown rape oil 75·8, and for the Japanese wood oil 189. The figures for the blown oils seem to support the suggestion that the action of bromine is more extensive than the iodine of Hübl solution. The low result obtained by the thermal method for Japanese wood oil remains, however, unexplained, but is probably due to peculiarities of constitution.

It is noteworthy that the differences in the case of Japanese wood oil disappear when the fatty acids are examined. These gave an iodine value by Hübl method of 150·1 and by bromo-thermal method of 148·3.

If an experiment be arranged with the Japanese wood oil as though for applying the thermal test, except that half of the chloroform is replaced by a saturated chloroformic solution of iodine—this being used in place of bromine,—the rise of temperature (only a few degrees) is very slow, but after a couple of minutes the whole solution rapidly thickens to a stiff elastic jelly. This effect with iodine was not noticed in the case of any other of the oils in the list, which all remained clear and perfectly fluid. Bromine has not this thickening effect on the oil in chloroform solution.

In determining the specific temperature reactions, the oils were, when necessary, diluted with a thin mineral oil. The Japanese wood oil acted in a way quite distinct and characteristic : as soon as the first drops of sulphuric acid fell into the oil, a dark, tough clot formed, which so clogged the thermometer that stirring soon became impossible. When one part of the oil was dissolved in four parts of mineral oil, it became just possible to continue the stirring after the addition of the sulphuric acid, and soon the clotted mass dispersed again and dissolved in the mineral oil.

Discussion.

Mr. Peter MacEwan asked Mr. Jenkins what he meant by wood oil. There were several things which went by that name, the best known being Gurjun balsam, an oleo-resin. There was also Chinese wood oil, the identity of which with Japanese wood oil he was not at the moment quite sure of; but it struck him that the difference in the bromine and the iodine absorptions pointed to the presence of an essential oil, seeing that the fatty acids agreed. On the other hand, Mr. Jenkins' description of the oil did not agree with Gurjun balsam.

Mr. Jenkins replied that he understood that it was practically the same as Chinese oil.

Mr. MacEwan, continuing, said that the presence of a proportion of essential oil in the wood oil would account for the difference in the iodine-absorbing powers of the oil and fatty acids, as well as the difference between the bromine and iodine absorption numbers, since it was well known that many essential oils absorb bromine much more readily than iodine.

Mr. S. Hall had in the course of his work noticed that some oils, such as linseed, rose in temperature very rapidly on treatment with chloroform. It had therefore occurred to him to use carbon tetrachloride instead of chloroform because of its higher boiling point. In some experiments made under those conditions that day the figure for linseed oil was found to be 165, which compared very closely with Mr. Hehner's figure. Another curious fact was that if bromine were added to the tetrachloride by itself, the temperature went down, and this fact had to be taken into account in working out the absorption value.

Dr. F. B. Power had been much struck by the peculiar properties of the so-called Japanese wood oil. He though it probable that it was a substance bearing some relation to Gurjun oil, commonly known as wood oil, which is obtained from the East Indies, and derived from a species of Dipterocarpus. This body was not, chemically speaking, an oil at all, but an oleo-resin, consisting of a resin with an essential oil having the character of a sesquiterpene, $C_{15}H_{24}$. When heated to about the boiling point of water, Gurjun oil became perfectly solid; and another peculiarity was the very beautiful reaction which it gave on treatment by nitric acid or other oxidising agent. If Japanese wood oil had a somewhat similar constitution, it would naturally behave towards bromine differently from the fatty acids.

Mr. J. H. B. Jenkins feared that he could not throw much light on the origin of Japanese wood oil. It had been proposed to replace linseed oil because of its superior drying properties. With regard to the suggestion that it might contain a considerable quantity of terpenes, the oil on saponification yielded a large proportion of fatty acids—about 96 per cent.,—and that fact was inconsistent with the presence of any large proportion of essential oil. He had included all the ordinary data relative to this oil in his second paper, but it was not of sufficient general interest to read to the meeting. He was much interested by what had been said in the discussion ; and in connection with the high temperatures attained by the chloroform solution of linseed oil when treated by bromine, he might mention that a friend of his was making experiments with a view to obviate any error likely to be introduced by the temperature approaching too near the boiling point of chloroform. He had not the refraction value of Japanese wood oil, but there was not the slightest danger of its being mistaken for linseed oil, though its iodine value was much the same. Its specific gravity was somewhat higher than that of linseed oil, and its viscosity was many times greater. The melting point of its fatty acids was much higher. The many peculiar properties of the oil as shown in the qualitative tests would also serve to identify it.

Mr. S. Hall remarked that some tests he had made with Japanese wood oil entirely bore out the figures given by Mr. Jenkins.

The CHAIRMAN remarked that, in view of the great attention that was being paid to the subject at present by various workers, it would be well that they should agree among themselves on the general conditions of research and particularly work with identical samples.

JAPANESE WOOD OIL.

BY J. H. B. JENKINS.

THE following further particulars respecting this peculiar oil may be of interest. It is expressed in China and Japan in enormous quantities from the seeds of the *Aleurites cordata*, and is used there chiefly as a varnish, and also for lighting (Chem. and Druggist, April 23, 1887). Efforts have been recently made to introduce this oil into Europe, and there are many directions, especially in paint and varnish making, in which it is likely to find an application. The oil was examined over twenty years ago by Cloëz (Bull. Soc. Chim. **26**, 286; **28**, 23), and more recently by Davies and Holmes (Pharm. J. 1885, 634, 636).

The present sample of the oil was clear and had a golden colour. It gave—

Specific gravity at 60° F.	0·9385
Solidifying point	below −17° C.
Iodine value (Hubl)	165·7
Saponification value (mgrms. KHO)	194
Hehner value (insoluble fatty acids)	96·4 per cent.
Unsaponifiable matter	0·44 „
Specific temperature reaction	372
Free fatty acids (calculated as oleic)	3·84 per cent.

The *mixed fatty acids* gave:—

Melting point	37° C.
Solidifying point	31° C.
Iodine value (Hübl)	150·1

For peculiarities in determination of iodine values and specific temperature reaction, see previous paper.

Viscosity.—The viscosity of the oil at 60° F. was determined with Redwood's viscometer. It required 1,433 seconds for 50 c.c. to flow through. Water requires 28 seconds and rape oil 470 seconds to flow through this particular instrument, at 60° F.

Valenta's Test.—The temperature of turbidity with glacial acetic acid was 47° C.

Sulphur Chloride Test.—To 5 grms. of the oil were added 2 c.c. of CS_2 and 2 c.c. of S_2Cl_2, and the whole stirred in the cold. In 1½ minutes the oil suddenly stiffened to a thick and sticky jelly. The product was not nearly so hard as that obtained from either castor or linseed oil.

Drying Properties.—Four grms. of the oil were exposed in the boiling-water oven in a shallow porcelain dish 7 cm. diam. After ½ hour's exposure there were evidences of a skin commencing to form round the edges. In two hours the oil would not flow, and was entirely covered by a crinkled skin. The average gain of weight during four hours' exposure was 0·36 per cent. per hour. For comparison, raw linseed oil was exposed in another dish; after four hours there was no evidence of a tendency to "skin," neither was there any sensible increase of weight.

Elaïdin Test.—By this test a brownish-red product was obtained. An oily layer rested on a lower, nearly solid portion. When stirred up, the whole would slowly flow.

Becchi's Argentic Nitrate Test.—A quarter-hour's heating with reagents in boiling water produced deep reddish-brown coloration.

Other Tests.—Strong sulphuric acid produces a black clot with the oil. Nitric acid (1·4 sp. gr.), after a couple of minutes, converts the oil into a tough mass, which on standing becomes darker and more brittle. When 1 grm. of oil is dissolved in 5 c.c. of chloroform, and 5 c.c. of a saturated chloroformic solution of iodine are added and the solution stirred, after about two minutes the whole is converted into a stiff jelly. If the amount of oil in this test be increased to 2 grms., the jelly formed is so stiff that it can be granulated. Some of the oil was heated, strictly out of contact with air, first for one hour at 150° C., then for two hours at 200° C., and lastly for a couple of hours at 250° C. At temperatures above 180° C. a very small quantity of gas was given off, but no change in the liquid condition of the oil was noticed until after the last heating to 250° C., when it became converted into a clear, solid, elastic substance. This peculiar product was insoluble in the ordinary oil-solvents, and showed no tendency to melt on being heated again up to the temperature of its formation—about 250° C.

NOTE ON THE ANALYSIS OF SUPERPHOSPHATES.

BY J. H. COSTE, F.I.C.

THE usual statement of analysis of superphosphates, though sufficient for agricultural purposes, is, from a chemical point of view, rather misleading, as it does not express the true condition of the "soluble" phosphoric acid. A sample of superphosphate which was found to contain 15·79 per cent. P_2O_5 (equal to 34·48 per cent. $Ca_3P_2O_8$), soluble in water when treated in the usual English method, was, after keeping about a month, analysed in the usual way, and also further determinations made with a view to determining the condition of the soluble phosphoric acid. The composition, according to the general method of stating results, was:—

Moisture (given off at 100° C.)	15·80
Water of combination, &c.	7·51
*Monocalcium phosphate (CaP_2O_6)	21·86
Insoluble phosphate	6·33
Calcium sulphate, &c.	46·55
Insoluble siliceous matter	1·95
	100·00
* Equal to tricalcium phosphate	34·25

The lime in the soluble and the sulphuric acid in the insoluble portion were determined, and the total sulphuric acid, lime, and phosphoric acid also determined in separate portions.

The following results were obtained:—

CaO in solution (same as soluble P_2O_5 was determined in)	10·24
SO_3 in insoluble portion	19·98
Total CaO	25·37
„ SO_3	30·01
„ P_2O_5	18·20

On combining these results, the statement of composition of the soluble part became as follows:—

Free phosphoric anhydride (P_2O_5)	7·53
Monocalcium phosphate (CaP_2O_6)	11·38
Sulphate of lime	17·05

and the amended analysis—

Moisture		15·80
Water of combination, organic matter, &c.		5·91
*Soluble	free phosphoric anhydride	7·53
	monocalcium phosphate	11·38
†Insoluble	tricalcium phosphate	2·03
	ferric phosphate	4·30
Calcium sulphate		51·10
Sand		1·95
		100·00
* Equal to phosphoric anhydride		15·69
† „ „ „		2·51

Nearly one half the phosphoric anhydride soluble in water was present as phosphoric acid, and not as monocalcium phosphate.

Liverpool Section.

UNIVERSITY COLLEGE, BROWNLOW STREET.

SESSION 1896-97.

April 7th, 1897:—
Election of Committee and Officers.
Dr. Hugh Marshall. "The Manufacture and Industrial Uses of Persulphates."

Meeting held on Wednesday, March 3rd, 1897.

DR. C. A. KOHN IN THE CHAIR.

MICRO-ORGANISMS: THEIR EFFECT AND INFLUENCE IN CERTAIN CHEMICAL INDUSTRIES, MORE PARTICULARLY IN THE MANUFACTURE OF LEATHER, WINE, AND BEER.

BY GUSTAF SCHACK-SOMMER, PH.D.

MICRO-ORGANISMS may be divided into three classes: bacteria, yeasts, and moulds, all of which play a more or less important part in pathology, as well as in many chemical industries. The aim, therefore, of the manufacturer in whose industry these organisms take part, should be directed towards promoting the increase of the beneficent and destruction of the harmful.

One of the subjects I originally intended to include in this paper was brought before this Society by Dr. Voelcker, in London, in November last (this Journal, 1896, 767). I refer to the inoculation of soil with organisms found in nodules on roots of leguminous plants, whereby the assimilation and utilisation of the free nitrogen of the atmosphere by the growing plant is promoted. I therefore omit the subject of artificial manuring through the aid of micro-organisms, and pass at once to the consideration of the first-named industry at the head of my paper, namely, tanning. The importance of the leather industry may be gathered from the fact that it is now said to rank fourth amongst English industries. In the process of tanning, the hides are first soaked for weeks in lime pits, so as to loosen the hair and the fleshy matter which adhere to the skin, and to transform the hide into gelatin with which tannic acid combines to form tannate of gelatin, or leather. As some skins, such as that of the calf, turn black in the tan pit should all the particles of lime not have been previously removed, and since for some purposes the pores, which the lime bath has caused to shrink and close, need opening, the skins or hides, which have been previously swelled by the liming process, require to be brought into a condition which allows of the easy and thorough removal of the lime and lime soap. To obtain a uniform shrinking of the swelled hides, the skins go from the lime pits into dog or pigeon-dung baths. The process consists in treating the hides for some time (sometimes four or five hours) in a vat containing a lye formed of solutions of dog, pigeon, or poultry dung. In some cases the dog-dung acts best, and pigeon or poultry-dung in other cases, but no satisfactory and sound explanation has as yet been given as to the precise kind of action which occurs.

In 1896 Dr. Popp and Dr. Becker, of Frankfort-on-Maine, were granted a patent for a process which resulted from the discovery that the action of these lyes is the result of bacteriological processes, and not, as has been very generally assumed, of the action of phosphoric acid. They examined dog, pigeon, and poultry dung bacteriologically, and showed in the first instance that even after removal of all other constituents which, according to existing views, entered into reaction, the result was the same. Continued research disclosed a second result, viz., that a number of micro-organisms exert considerable influence on the lye, partly beneficial, and partly detrimental. They say that, generally speaking, the non-peptonising bacteria act beneficially, while the peptonising ones generally have a detrimental effect. The specification states that in connection with this discovery, two methods of utilising them have presented themselves (this Journal, 1896, 911).

In the December and January numbers (1896, 910, and 1897, 52) of the Journal there have appeared articles entitled "Fermentation Phenomena in Tan Liquors," by F. Andreasch, to which I beg to call your attention.

In the preparation of tobacco the presence of the right kind of micro-organism has been found to greatly enhance the value of the finished product; it has, therefore, been found advantageous to sprinkle the leaves at the commencement of manipulation with the liquid, which contains pure cultured micro-organisms of the required kind, and the result has been much more satisfactory than if the fermentation had been left to chance.

Although pure cultures have, to the best of my knowledge, not been employed in the manufacture of white lead, the so-called Dutch process is based on the action of bacteria contained in the dung by which the lead plates are surrounded.

Quite a number of chemical compounds, which it has been quite impossible hitherto to produce synthetically, have been brought about by the help of micro-organisms. As an instance, I have only to name lactic acid. Dr. Alfred Koelliker, proprietor of the firm of Dr. L. Marquart, at Beuel, near Bonn, has made use of pure cultivations in manufacturing pharmaceutical preparations. I understand that he ferments sugar solutions at 104° (40° C.) with a pure cultivation lactic ferment. The lactic acid, while being formed, is neutralised by adding lime or chalk during the fermentation. This calcium lactate is purified by frequent re-crystallisation, and is then either sold as such or used for making lactic acid or ferrous lactate. Colours for eatables are now prepared with pure cultures of bacteria on rice, a process which the Chinese have known for ages.

In the preparation of a liquid glue for photography, photolithography, &c. gelatin or other nitrogenous substances are peptonised with bacteria such as *bacillus prodigiosus*.

Thus far the action of the micro-organisms has been considered as the cause of chemical action, but we now come to other industries where they are not only promoters of chemical action, but are of the greatest importance in producing flavours in those substances in which they are allowed to multiply. Take for instance the manufacture of cheese and butter. It is generally known that in butter-making, pure culture bacteria (bacteria of the species producing the desired flavour, and not bacteria which produce unpleasant flavour) are used for inoculating the cream for the purpose of making good butter. The bacteria favourable to this purpose are already in the market, two or three different kinds now being used in this country, the result being that the butter-maker can depend with more certainty upon the product.

For the manufacture of cheese the proper selection of bacteria is of still greater importance than in the case of butter. The curd itself is practically tasteless; it is set to ripen, and it is during the process of fermentation (ripening), which is brought about by micro-organisms, that the taste developes. The different kinds of cheese are mainly due to the growth of different kinds of bacteria in the cheese. Theory in this latter industry is, however, yet ahead of the practice and much good work can still be done in this direction.

The most important industries in which fermentation plays a part are brewing and distilling, and the manufacture of wine. In the first and second fermentation is started artificially by adding yeast, whereas in the third the grape juice ferments without any such addition. Brewers have learnt from experience that with the same raw material they may get products with different flavours, and they have known for a long time that it is advantageous for them to get now and again a fresh supply of yeast from breweries where an avowedly well-flavoured beer is produced. In the same way experience has taught distillers that the yield of alcohol from the same raw material may vary several per cent. The wine manufacturer too found that in certain years his must would not ferment, although by chemical analysis its composition did not differ from that of former years, and so he attributed the failure to the difference in the ferments.

Pasteur, Hansen, Joergensen, and others have shown that these differences are due to the quality and variety of the species of yeast used. They have arranged methods by which the yeast varieties may be distinguished, and by which the most suitable can be picked out. As a consequence certain establishments have adopted the pure cultivation and rearing of special varieties in their works, and the largest and most scientifically and intelligently conducted breweries work now with these pure culture yeasts with the very best results. They find that if they are uniformly careful in the selection of their raw material and in the manner in which they conduct the fermentation, the use of pure culture yeast enables them to have a regular and uniform product of a characteristic similarity of taste. In distilling the same experience has been gained. Preference is now given to those yeast varieties which give an especially high yield of alcohol, and pure cultures are made accordingly. The conditions here, as in brewing, are specially advantageous, as the mash is boiled, and so sterilised, before the addition of the yeast.

Must cannot stand sterilising as it would spoil its flavour, but it is all the better for pasteurising, that is, heating for half an hour in an enamelled or tinned vessel at a temperature of from 130° to 149° F. (60° to 65° C.) and then cooling down to 68° to 72° F. (20° to 22° C.). To that must, which does not contain sufficient nitrogen for the nourishment of the yeast, some should be added. For instance, it is nearly always deficient in cider, perry, and must made of berries, such as elderberry, currant, or gooseberry; to these 15 to 20 grms. of chemically pure chloride of ammonium should be added to every 1,000 to 1,200 litres of must.

Germany has taken the lead in adopting the discoveries of Pasteur, Hansen, and Joergensen in wine manufacture. Messrs. Popp and Becker, to whom I am indebted for much valuable information embodied in this paper and a great many yeast specimens, have the best known institute for the pure cultivation of yeast in Germany. In France, Messrs. Schloesing Frères and Co., Marseilles, are working on similar lines. The former state, and prove their assertion by many examples, that the quality of the wine that has been fermented with pure yeast culture is not only improved, but that the influence on the character of the wine is most telling. The discoveries which were first made in the laboratory, that some varieties of yeast produce more alcohol and glycerine than others, that they have a great influence on the taste, flavour, and bouquet of the wine, and that by the addition to the must of pure culture yeast the fermentation is more rapidly accomplished than if left only to the influence of its natural ferments, have been confirmed on a large scale.

As there are many kinds of fruits and cereals, each of which has its proper name, such as cherry, plum, apple, wheat, and barley, so there are a similar number of varieties of yeast, which will cause a fermentation in sugar containing solutions like must and maltwort. The products of these fermentations, however, differ much from one another. Whilst some produce alcohol and carbonic acid gas, others produce nauseous compounds. Yet even the fermentation of those varieties which produce principally alcohol differ very much both in the energy and speed with which they start fermentation and in the time they require to accomplish their task and in the extent to which they can decompose the sugar. Whilst some start growing and evolve carbonic gas early and stop after several days when the yeast settles at the bottom and leaves the liquid comparatively clear, other varieties start slowly with very little escape of carbonic acid gas, dragging on the fermentation for months, and preventing the liquid from becoming clear. All these varieties of yeast, the good as well as the bad, are generated in and on the soil, and scatter their spores through the wind on to the grapes from which they get into the must. If the beneficent kinds predominate, the wine ferments energetically and quickly and is soon clear, but if the others are in the majority, or even numerous, no matter from what cause, then the must will ferment badly and slowly and will not show signs of becoming bright after months have elapsed.

By using a properly selected pure culture yeast, peculiar to the same kind of grape or one of another particular species, the resulting wine will be of a better quality than if the must had been left to chance fermentation. It would be a mistake to assume that it would be possible to make from any grape juice the choicest wines, for although the influence of pure culture yeast on fermentation is very great, the slower or quicker development of the fermentation and the composition of the finished wine, is amongst others dependent on the quality of the must.

As an instance of the influence which particular varieties of yeast had on the same must, Drs. Popp and Becker send me the following analysis:—

Yeast.	Alcohol.	Glycerine.	Unfermented Sugar.
	Per Cent.	Per Cent.	Per Cent.
A	10·00	0·74	0·07
B	9·85	0·71	0·12
C	8·37	0·56	2·72
D	9·17	0·70	1·13
E	9·68	0·58	0·13
F	7·86	0·50	3·72

In this case yeast A was the best in every respect. It yielded 10 per cent. alcohol, 0·74 per cent. of glycerin, and only left 0·07 per cent. sugar undecomposed, whereas yeast F only produced 7·86 per cent. alcohol and 0·50 per cent. glycerine, and left 3·72 per cent. sugar undecomposed. They add, "naturally we choose yeast 'A' as one of our specimens from which to make pure cultures."

Pure culture yeast has been applied with great advantage in cider and perry manufacture, with the result that these lose their peculiar rough taste and become more winelike. A still more favourable result has been obtained with wines made from berries, especially in the speedier finishing of the product. A wine made from bilberries frequently takes three to four years before it is ready for drinking. This entails a great loss in interest and no small risk. With a suitable pure culture yeast the fermentation will take place much more quickly and much better. Currant and gooseberry wine, which must contain a high percentage of alcohol, should only be fermented with pure culture yeast specially adapted for a thorough fermentation.

The fact is thus established that it is not the geographical position alone and the soil on which the vine grows that accounts for the characteristics of a wine; but that just as great importance should be attached to the kind of yeast used, for not only does the yeast decompose the sugar in solution and so form alcohol and carbonic acid gas, but it assists as well in the decomposition of malic acid and in the formation of succinic acid, glycerine, and various aromatic compounds, to which the flavour of the resulting product is due.

Having arrived at these conclusions, it naturally followed that the chemist went a step further, and tried the effect of the yeast of the choicest wines on other raw products, such as beerwort. Besides the scientific value of these experiments, they promised, if successful, a new field of industry, namely, the manufacture of beverages similar to wine in countries where the grape does not grow, beverages, too, which while containing a larger percentage of nourishing properties, rival in flavour and excellence many of the best known wines of southern climes.

Pasteur was the first to try the fermentation of beerwort with wine yeast. The result as far the aroma went was satisfactory but was very disappointing as regards flavour. M. Jacquemin of Paris, and Dr. Sauer of Schoeneberg, near Berlin, picked up the threads where they were dropped by Pasteur, and went to work in the eighties to investigate the same problem. Jacquemin took out a patent in England in 1887 for a process he discovered, but which I understand has never been exploited (this Journal, 1888, 397).

I find in Jacquemin's specification, that he added a suitable quantity of bitartrate of potash to the wort with the object of producing an acidity similar to that of the grape, and thereby obviated the necessity for introducing lactic ferment, one which brewers have great difficulty of avoiding and which he said was detrimental. He then introduced the elliptical ferment of wine yeast. But the above-mentioned addition of bitartrate of potash was not the only addition of a chemical; he introduced tartaric acid into the cane-sugar solution for the purpose of inverting it, and this solution after having been boiled was then added to the wort to accelerate the fermentation and produce a higher percentage of alcohol. He contends that the said inversion of the sugar by an organic acid is essential, as otherwise the fermentation would be too slow to give good results. What Jacquemin said of lactic acid in 1887 was quite right then, for impure lactic acid retards a good and pure alcoholic fermentation, and I suppose the addition of the various chemicals had a good deal to do with the fact that Jacquemin's "Vin d'orge" has never become a popular beverage.

Dr. Sauer some five years ago applied for a patent for a process embodying the results of his discoveries. He succeeded in solving the problem of producing a wine in every way equal in character, flavour, and aroma to similar wines made from must, and moreover, succeeded in freeing this product from all malt taste or smell. He discarded the yeast of the vines of his native country and of those of France, and made pure cultivations from yeasts that have a greater power of fermentation, probably arising from the fact that they are gathered from grapes of southern climes which contain a much higher percentage of sugar. Where however, he had the greatest advantage over Jacquemin, was, that in the meantime, the process of producing pure lactic acid by the pure culture lactic acid ferment had been discovered and it had been ascertained that pure lactic acid had a favourable influence on alcoholic fermentation whilst checking the growth of other micro-organisms. Probably on that account Dr. Sauer was enabled to experiment on the pure malt decoction, generally called wort, without adding any chemicals whatever and only introduced pure cane-sugar into the liquid, as required for raising the alcoholic strength. In his process of fermentation, which I shall describe more fully presently, he produced beverages containing 18·5 volumes per cent. of alcohol.

Dr. Sauer chooses his malt according to the character of wine he wishes to represent and prepares his wort in the copper in the same way as it is done in every brewery. The malt-wort used in brewing beer is as a rule somewhat acid, yet seldom reaches more than 0·1 per cent., and even that quantity is undesirable in the manufacture of beer. In the case of making Dr. Sauer's malt-wine the desired effect is brought about by adding a pure culture of lactic acid ferment. This is allowed to act on the malt-wort heated up to 122° F. (50° C.) for 24 hours in which time it will have changed 0·6 per cent. to 1 per cent. of the wort and produced the lactic acid required. It is then pasteurised, that is, heated quickly to 167° (75° C.) and so the lactic acid ferment is destroyed. At this point it is necessary to cool the hot wort down to the proper temperature, that is, 77° F. (25° C.) and then add a purely raised wine yeast, to induce fermentation. These yeasts are peculiar wine yeasts (*sacch. ellipsoideus*) which are specially selected. They are raised from a small quantity, if possible only one cell, on sterilised, acidified malt-wort. The selection of these wine-yeasts should be made in accordance with the particular kind of malt-wine it is desired to produce; in other words, in choosing the yeast one must be guided by the flavour and aroma required.

During fermentation the embryo wine tastes somewhat like "must." The fermentation is accelerated and intensified by the gradual addition of raw sugar, and when this is completed, the preparation must be so treated as to eliminate from it everything that can suggest to the taste or the smell its malt origin. This is done by the action of the oxygen of the air.

The difference between low-fermented liquors which contain under 12—13 volumes per cent. of alcohol (under which head are included all beers, and by far the greater number of the grape wines of Central Europe) and the high fermented alcoholic beverages which contain over 12—13 volumes per cent. of alcohol is that the latter will mature if they are judiciously exposed to the air, whereas the former spoil if the air is not carefully excluded.

The malt-wine is kept for some weeks in contact with constantly renewed outer air, at a temperature of 122° F. (50° C.) or over. The higher temperature not only hastens the maturing process, but, at the same time nullifies the baneful influence of micro-organisms in conjunction with the acidity and alcohol present in these beverages. While beer is invariably shut off from all contact with the atmosphere, at a very low temperature, and matured entirely by after-fermentation, malt-wine is, on the other hand, matured as above stated, after its fermentation is completed.

A peculiar odour arises from the aromatic properties of the malt and the alcoholic contents, the exact composition of which has not yet been determined. Whether this odour, which is also present in the very best old selected Tokay, is acetal as discovered by Von Liebig, must be left for future investigation. Certain it is, however, that the malt aroma disappears entirely, and is replaced by a very agreeable one.

After the precipitate of albumin has settled, the liquid is put into casks and left to ripen. After this the malt-wine is ready to be bottled and will keep for any length of time.

The wine, to begin with, is akin to beer with which its mode of manufacture is practically identical. Its ingredients are also of such a nature as to allow of malt-wine being classed for excise purposes among the beers. It differs from them in the relative proportions of the ingredients, and especially in containing a large proportion of alcohol, in which property it compares with the high-fermented genuine wines. It also differs from the known beers in appearance and taste, in both of which it resembles the proper grape product, justifying the assumption that it may take the place of genuine grape wine, although the chemical composition is not the same.

The analyses by Fresenius show the following results:—

Character of the Wine.	Sherry.	Madeira.	Malaga.	Tokay.
Specific gravity	1·0203	1·0327	1·0594	1·0909
Polarisation in 220-mm. tube direct.	+17·75°	+18·3°	+31·9°	+29·48°
Polarisation in 220-mm. tube after inversion.	+17·75°	+18·0°	+32·3°	+21·12°
	Grms.	Grms.	Grms.	Grms.
Alcohol (percentage in weight)	14·78	14·22	12·13	11·41
Alcohol (percentage in volume)	18·62	17·91	15·28	14·37
Extract	11·27	14·24	20·50	25·50
Mineral substances	0·24	0·23	0·35	0·37
Free acid (calculated as lactic acid).	0·64	0·75	0·73	0·82
Volatile acid (calculated as acetic acid).	0·062	0·077	0·064	0·061
Non-volatile acid (calculated as lactic acid).	0·55	0·64	0·063	0·73
Glycerine	0·31	0·34	0·20	0·19
Nitrogen	0·062	0·059	0·190	0·101
Corresponding to albumen	0·387	0·369	1·188	0·631
Phosphates	0·0654	0·0690	0·1515	0·1371
Raw sugar	..	..	..	5·86
Reducing sugar (invert sugar, dextrose, maltose) expressed as dextrose).	5·00	7·37	10·25	13·14
Dextrine	2·86	2·12	5·00	2·17

The tests for salicylic, tartaric, citric, and malic acids gave negative results. The acids present may, therefore, be assumed to result from fermentation, and consist of

lactic and succinic acids. The above analyses indicate that these preparations are beverages of a sweet-wine character, and with respect to the amount of alcohol and sugar they contain, are absolutely identical with sweet wines. They are distinguishable from the latter in containing a not inconsiderable quantity of dextrin, and of extractives peculiar to malt, whereas the extractives of the genuine grape sweet-wines are absent. With regard to their nutritive qualities, they are on a par with extract of malt beers, but their stimulating powers are fully equal to those of sweet wines.

Owing to the presence of the nourishing extractives of malt (which include nitrogenous substances, dextrin, as well as phosphates), they should replace the so-called medicinal wines now in use. One of the advantages claimed for these wines is that they are free from sulphates, which are present in sherry and similar southern wines, owing to these having been clarified with gypsum.

Discussion.

The Chairman asked whether the use of specially cultured germs for tanning had been tried on the large scale, and if so, whether the difficulty of getting even results with the different kinds of hides employed had been overcome by thus adjusting the tanning materials? In regard to tobacco also, had any practical application really followed the investigations referred to by Dr. Schack-Sommer.

Respecting the wines made from malt he (Dr. Kohn) felt that Dr. Schack-Sommer had laid too much stress on the value of the chemical analyses. Did the percentage of alcohol really mean anything in regard to the nature of the wine? The difference between pure brandy and that adulterated with potato spirit was not recognisable, he believed, by chemical tests although very evident by taste to a connoisseur.

Dr. Schack-Sommer had also referred to the oxidation of the must as a part of the process described by him. Was that not a process likely to destroy the most characteristic properties of good wine? Some time ago one heard of maturing wines by means of oxygen, and he (Dr. Kohn) had experimented with Italian wines with this object. The result was certainly to do away with the harsh taste of the wine, but the original bouquet was entirely destroyed. He was therefore inclined to think that this portion of the process was likely to prevent the preparation of any of the better qualities of wine from malt.

Mr. E. K. Muspratt remarked that Dr. Schack-Sommer had spoken of pure cultivations of yeast that were used for the manufacture of cider. Did that explain why large quantities of apples were taken from Normandy by rail to Frankfort, converted into cider, and the cider exported? He thought it quite possible owing to the excellent quality of the cider produced by these methods.

Dr. Schack-Sommer replied that it was curious that it was at Frankfort where those improvements took place. They certainly delivered pure wine cultures, and so far as he knew that had not been done in England.

Mr. Muspratt said there was no doubt they got very excellent cider in England, which could be improved by the adoption of scientific methods of manufacture.

Dr. Hurter asked whether the amount of sugar which must be produced in one way or the other by the soil could be raised in this country at such a cost that it could really compete with the great quantity of cheap wines now imported; and was there really any prospect of making wine materially cheaper, taking into account the amount of the excise duty which would have to be paid on home produce just as much as on wines imported from abroad.

Dr. Schack-Sommer in reply to the Chairman's question as to whether bacteria had been used practically in tanning and in the curing of tobacco on a large scale, answered in the affirmative. Amongst others, a large firm in London, having tanneries in England and America, had worked very successfully with this new departure: and further, the curing of tobacco by micro-organisms had long ceased to be in the experimental stage, and been very generally adopted by the trade.

To those who criticised the wine-like beverages brought before them from the standpoint of wine connoisseurs, who would pay 10s. or more for a bottle of port, sherry, or Tokay, he submitted that such a high standard was not aimed at. The object had been to produce a wholesome, nourishing, palatable, wine-like beverage at a low price that would compare favourably with the average quality of the wines usually sold of the kinds mentioned above.

The less said the better as to the power of chemists to distinguish the differences between high class and ordinary wines and spirits of the same kind. All those who had studied the Blue Books containing the report of Lord Playfair's Commission on wines and spirits would agree to their inability to achieve this. The analysis by Professor Fresenius showed that there were no sulphuric acid or sulphates present, and these were always to be found in sherries. The analysis showed the composition of the wine, and from this the medical profession on the Continent concluded that the beverages under consideration must be nutritive as well as stimulating.

It was suggested that forcing air through the finished product might remove the bouquet; he proposed to the audience to use their olfactory organs and so convince themselves from the samples submitted to them, that this was not the case. He wished to draw their attention to the fact that these samples were rather sweet, for the reason that they were made in Germany for German tastes. The wines could be prepared just as well dry, so as to suit English tastes.

To Dr. Hurter, who had asked if it were possible to get sugar cheap enough to justify the starting of this industry, he would reply that there was no country where sugar was as cheap as in England, nor could a better place be found for manufactures in which it was an important raw material. When in Hamburg last spring, he was surprised to find in the large "Muster Lager" (Sample Exhibition), the whole collection of sweets and fancy confectionery goods were from England or Scotland, and when he asked how that was, as they had such large works in Germany, he was told that they could not compete in price for export with British produce. If this was true for sweets, it would be equally so in the manufacture of wine. The question of the duty to be paid to the excise authorities had been submitted to the officials at Somerset House, and they were of opinion that the tax would have to be levied in the same way and to the same amount as in an ordinary brewery, here being practically no difference in the processes.

Manchester Section.

Rooms of the Chemical Club, Victoria Hotel.

Chairman: George E. Davis.
Vice-Chairman: Peter Hart.
Committee:

F. H. Bowman.	J. M. Irving.
R. Forbes Carpenter.	M. J. Langdon.
R. S. Dale.	J. Lewkowitsch.
R. Le Neve Foster.	E. Schunck.
B. W. Gerland.	W. Thomson.
H. Grimshaw.	D. Watson.
J. Grossmann.	

Hon. Local Secretary:
J. Carter Bell,
Bank House, The Cliff, Higher Broughton, Manchester.

SESSION 1896-97.

April 2nd:—
Election of Five Members of Committee.
Sir Thos. Wardle, F.G.S. "Silk Adulteration."
Dr. Carl Otto Weber. "Hot Vulcanisation of India-rubber."

Erratum.

THE FIXATION OF PIGMENTS WITH ALBUMIN.

BY R. J. FLINTOFF, F.C.S.

(*This Journal*, 1897, 109.)

1st par., lines 9 and 11, for "rags" read "rays."

Newcastle Section.

Chairman: F. S. Newall.
Vice-Chairman: G. T. France.
Committee:

A. Allhusen.	W. Martyn.
P. P. Bedson.	John Morrison.
T. W. Hogg.	John Pattinson.
H. Louis.	W. W. Proctor.
T. W. Lovibond.	W. L. Rennoldson.
N. H. Martin.	T. W. Stuart.

Hon. Local Secretary and Treasurer:
Saville Shaw, Durham College of Science, Newcastle-on-Tyne.

SESSION 1896-97.
Date of Meeting.—April 29th.

Meeting held in the Chemical Theatre, Durham College of Science, on Thursday, February 25th, 1897.

MR. JOHN PATTINSON IN THE CHAIR.

MICRO-CHEMICAL EXAMINATION OF LEAD-ANTIMONY, TIN-ANTIMONY, TIN-ARSENIC ALLOYS, &c.

BY JOHN E. STEAD, F.I.C.

The microscopic study of metals was instituted and successfully carried out by Dr. Sorby, of Sheffield, and his first paper on the subject was read before the Sheffield Literary and Philosophical Society in February 1864, or 33 years ago, but his studies were confined almost exclusively to iron and steel. For many years little or no notice was taken of this pioneer's researches, and practically nothing has been done in this country until quite recently, although the microscopic structures of iron and steel have been more or less studied by our friends in America and on the Continent. Prof. Martens and Dr. Wedding, in Germany; H. Behrens, in Holland; F. Osmond, in France; L. Garrison and A. Sauveur, in America, have all communicated valuable articles on the structures of irons, spiegeleisens, and steels; and quite recently, within the last few years, Messrs. Guillemin and Charpy, and Prof. J. O. Arnold have made most important microscopic researches on the structures of alloys.

Prof. H. Behrens (Das Mikroskopiche Gefüge der Metalle und Legierungen) is, I believe, the only gentleman who has written a book solely dealing with the micro-structures of metals and alloys. This book is most instructive, and we can only regret that it has not yet been translated into the English language from the German.

In this work, Prof. Behrens describes the micro-structures of a very large number of alloys such as are commercially in use.

The valuable researches of Mons. F. Osmond, which are to be found in the Transactions of the Société d'Encouragement pour l'Industrie Nationale of Paris, have enabled us to determine, with a fair amount of precision, by the microscope alone, the heat treatment to which steel has been subjected; and we are now able, on the examination of very small pieces of structural steel, to determine whether or not it has been properly dealt with by the workmen during its manipulation. It is not my intention to dwell upon or discuss the structure of iron and steel, but rather to describe briefly researches which have recently been made upon alloys, and afterwards to put before you my own work.

One of the most valuable articles on the micro-structure of gold and copper, to which had been alloyed small quantities of other metals, was published in *Engineering*, February 7, 1896, by Prof. Arnold, of Sheffield. In this article it was clearly proved that the reason why bismuth, to the extent of only 0·2 per cent., in gold and copper made the whole mass so brittle and weak, was that the bismuth, or a compound of bismuth with gold or copper, separated out from the metals when solidifying, in such a way that every grain or primary crystal of the pure metals which had freed themselves from the impurity became enveloped, more or less completely, with a brittle "cement" of low melting point.

A polished and etched specimen of gold containing 0·2 per cent. of bismuth, presents the appearance of polygonal grains or crystals of yellow gold, cemented together with a red-brown substance, which Prof. Arnold has provisionally called bismuthide of gold. The thickness of this red-brown track or cement is out of all proportion in volume to the quantity of bismuth added, and Prof. Arnold is no doubt correct in assuming that the bismuth combines with a proportion of the gold, forming a fusible alloy. According to the same authority, tellurium and lead, when alloyed with gold, give very similar micro-structures to that produced by bismuth.

The bismuth in copper appears to separate out in a free state, and is found between and surrounding the grains of copper.

Prof. Arnold has also studied the micro-structure of copper after melting it with small quantities of cuprous oxide, cuprous sulphide, silicon, arsenic, and antimony, and in each case it was found that the impurity was found between the grains. This paper, in my opinion, is one of the most valuable ever communicated on the micro-structure of metals.

It will have been observed that Prof. Arnold has assumed, probably most correctly, that the bismuth and some of the other impurities actually form chemical compounds, but at present I do not believe that he has actually separated them and verified this hypothesis.

Prof. Martens, in his most valuable paper on the micro-structure of ingot iron in cast ingots, read at Chicago, before the International Engineering Congress, in August 1893, pertinently points out "that the true aim to be steadily and earnestly kept in view, is first to develop the methods for the microscopic resolution of the elements of structure, and to determine their chemical composition"; and then continues: "Micro-chemistry proper should set itself the task of isolating a certain element of the structure, and determining its composition by micro-chemical means, such as crystallising the element out of solution and determining the crystals or chemical reactions in a microscopic field"; and he appeals to all friends of microscopic metallography that they should pursue their labours along the course indicated.

Prof. Behrens, in his work on micro-chemical analysis, gives us some hope that to a certain extent the chemical reactions may be developed on the various structures in metals and alloys in the microscopic field; but the more certain method, and the one I have adopted, following the example of Sir F. Abel and Dr. Muller, is the employment of some reagent which will preferentially dissolve away one constituent before another, and then to make chemical analyses of the residues and solutions. It was in this way that the gentlemen just named determined the constitution of the hard carbide of iron.

Mons. Charpy, in a most valuable paper on the alloys of copper and zinc, read before the Société d'Encouragement pour l'Industrie Nationale, February 1896, after a most painstaking research on the mechanical, physical, and microscopic properties of brasses, sums up his microscopic researches by the following conclusions:—

"It" (*i.e.*, the microscope) "allows us to classify the alloys in three categories, corresponding to well-defined mechanical properties: the first category corresponds to alloys very malleable in the cold; the second to alloys of great resistance, less malleable than those of the first category, but forgeable when hot; the third category includes the very various alloys of which the common characteristic is brittleness, which is enough to make them rejected for commercial application." "Again, the study of the structure allows us to determine approximately the mechanical treatment undergone by the metal—above all, in the case of alloys of the first category."

"We shall be able to determine if the metal has been cast, and, according to the dimensions of the grain, to know

almost in what conditions (high or low temperature, moulded in sand or cases); if it has been hardened; if it has been annealed, at a high or low temperature; finally, if it has been burnt."

"The study of the structure perhaps may furnish many other indications; it really appears to us already capable of useful application, not to replace chemical analysis, but to complete this operation by information which chemistry is powerless to furnish."

In an admirable paper by Mons. Osmond, read before the International Congress at Chicago, upon Microscopic Metallography, he briefly gives the observations of Mr. Guillemin, and as this clearly shows to what extent the examination of metals and alloys have been pursued in France, I introduce it here.

Mr. Guillemin says: "Micrographic examination permits the direct classification of the usual alloys in a few categories." "Thus, the bronze and brasses are distinguished as—

- Tin-bronzes.
- Phosphor-bronzes.
- Brasses containing less than 37 per cent. of zinc.
- Muntz metal and analogous alloys containing more than 37 per cent. of zinc.
- Aluminium-bronze.
- Aluminium brasses.
- Delta metal.
- Roma bronze, &c.

"In the white 'anti-friction' alloys containing tin, antimony, and copper, it is easy to recognise the presence, and even, with a little practice, to make a close estimate of the proportion of lead.

"By examining the ingots of rose-copper composing different casts from the same smelting of ore, those which have been perfectly refined can be distinguished, and the rest can be classified according to the degree in which they have been refined."

"It is known that the mechanical qualities of brasses and bronzes are profoundly modified by the addition of small quantities of aluminium and of phosphorus." "The presence of these two elements can be detected with certainty by micrographic examination. Thus, the tracings assume invariably the form of veins in marble when the alloy contains aluminium, even in proportions so minute as to be detected with difficulty by chemical analysis." "In like manner, phosphorus produces in tin-bronzes an absolutely characteristic image, recalling a fern-leaf. This reaction, however, may be masked by the presence of zinc, in amount exceeding 4 per cent."

"Finally, for a known alloy, the microgram indicates also the conditions of casting, as well as the nature of the mechanical work subsequently performed upon the specimen. Thus, the image shows whether bronze has been cast too hot or too cold; whether it has been stamped or rolled; and, in the latter case, the direction of the rolling is distinctly marked"

"In a word, micrographical analysis enables us to determine quickly and summarily the nature of a bronze or an industrial alloy by the simple inspection of a polished and etched surface, and to recognise whether the alloy has been simply cast, or has been rolled or stamped."

Prof. Roberts-Austen, who has perhaps done more investigation into the physical and mechanical properties of alloys than anybody else in this country—his results being embodied in four consecutive reports to the Alloys Research Committee of the Institution of Mechanical Engineers—in his fourth report on compounds of copper and zinc, states that in his next report he will deal with the micro-structure of metals and alloys.

When the ternary and other more complex alloys are studied, I anticipate that it will require the combined assistance of the recording pyrometer, the microscope, chemical analysis, and the mechanical methods of investigation in order to obtain a correct knowledge of their properties, constitution, and structure; and I also anticipate that it will require the work of careful investigators for very many years to come before we have obtained the truth about them all.

During the past year I have devoted most of my leisure time in repeating the researches of Osmond, Arnold, and others, and have obtained results which practically confirm their work. It has, however, been beyond the resources of a private laboratory to experiment with gold; but as Prof. Roberts-Austen's pyrometrical observations agree with the microscopic work of Prof. Arnold, we may consider that the researches of the latter are beyond dispute.

The micrographic study of metals is of such a fascinating character, it appears to me, considering that the unexplored field is so extensive, and the results of research so interesting, it is difficult for anyone interested in true science, who once commences this kind of research to abandon it.

The whole operation of making the alloys, cutting them up, polishing, etching, and examining them, is of such a simple and inexpensive character, almost anyone with a little practice can do important research and obtain most valuable information.

It would not appear, after reading all the available literature on the micro-structure of alloys, that the compounds of lead and antimony have been studied; I therefore prepared a series of lead-antimony alloys, and the following description of my work will, I hope, be of interest to the members of this institution:—

Lead and Antimony.

As the object was to ascertain the micro-structure of alloys which are commercially produced, virgin lead and best "star" antimony, such as are regularly used in commerce, were employed in this research. Fifteen mixtures were made, in which the proportion of lead and antimony varied between 1 per cent. of antimony and 99 per cent. of lead, and 95 per cent. antimony and 5 per cent. lead. The carefully weighed metals were introduced into plumbago crucibles containing fluid cyanide of potassium, which were then placed in a Fletcher's gas crucible furnace. The lead in each case was first added, and when it was fluid and red hot, the antimony was dropped in, and as it immediately sunk below the surface of the molten cyanide practically no volatilisation of antimony took place. The crucible with the contents was then vigorously shaken, to effect thorough admixture, and was allowed to slowly cool in the crucible in the open air. When cold, the cyanide was washed away by a running stream of water, and the solid metal or alloy was then removed by striking the mouth of the crucible upon an iron slab. Of course, the same crucibles were used over and over again, as it was not necessary to use a new one for each trial. The pieces of alloy weighed about 100 grms., and measured about $1\frac{1}{2}$ in. at the top, and were about 1 in. deep. Each sample was sawn vertically through the centre, and a complete section was then taken by cutting out a piece about $\frac{3}{16}$ in. in thickness.

Grinding and Polishing the Specimens.

After many trials, it was found that the best way to obtain a perfectly flat face was to hold the section between the first finger and thumb, and to draw it downwards over the face of a smooth-cut file held vertically by the left hand, the tang resting upon a hard board or support. Having obtained a smooth surface, the next operation was to grind it upon a flat surface of Turkey stone, such as joiners use for sharpening their tools; but instead of using oil, the stone was kept continually wet by the application of clear water. In a very few minutes of rubbing backwards and forwards, the file marks were obliterated.

The following stage consisted in rubbing the specimen, with the application of practically no pressure, over a sheet of French emery paper No. 0000.

It is important that no pressure, or at least practically no pressure, should be applied; otherwise the softer parts of the metal will be forcibly dragged or torn, and will overlap the harder parts of the structure.

The final stage consisted in rubbing the specimen over smooth cloth, free from ribs, upon which had been previously placed a small quantity of diamantine powder—a most valuable preparation, made in Switzerland, for the purpose of polishing steel, and which can be obtained from almost any jeweller; a bottle costing 1*s.* 6*d.* will serve to polish more than 100 specimens. It is important that this block should

be kept continually moist, otherwise the softer metal will be torn by the friction and the specimen spoiled. It is also important that the pressure applied to the metal should be excessively light. The surface should be examined from time to time, and the polishing stopped as soon as the fine scratches left by the emery are obliterated. If the polishing is carried beyond this point, the softer constituents will be carried away, and the crystals or grains harder in character will stand in great relief, and be more or less rounded, and their true shapes rendered difficult to determine.

Etching the Polished Specimens.

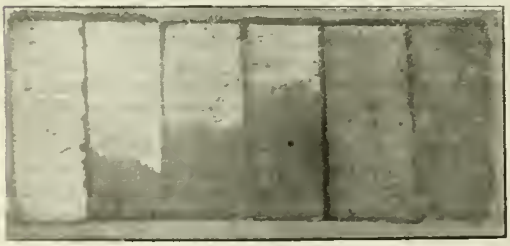
After leaving the polishing block, each specimen was thoroughly washed in water, and was at once, without drying, placed in a solution consisting of 99·5 c.c. of water and ½ c.c. of nitric acid of 1·42 sp. gr. In about one minute the structure was usually sufficiently developed for a preliminary examination. It was then removed from the acid, thoroughly washed with water, and afterwards soaked in rectified spirit, after which it was removed, the excess of alcohol shaken off by swinging the specimen backward and forward, and was finally placed on a hot plate to dry, when it was ready formicroscopic examination.

Description of Micro-Structures.

In the specimens containing between 1 and 13 per cent. of antimony, the structure appeared to be almost amorphous, and it was difficult to determine the character of the crystals; but in the alloy containing 15 per cent. antimony there was found to be a distinct layer, clearly visible without magnification, of harder metal at the upper surface, that is to say, on that part of the metal which was uppermost when it solidified. In proportion as the antimony was increased, this hard and silvery white layer increased in thickness, until when 50 per cent. antimony was present, the whole mass appeared to be bright. On examining these white layers with an objective magnifying about 50 diameters, it was noticed that they were not by any means homogeneous, but consisted of more or less well-formed crystals, apparently of cubical form, imbedded in a substance exceedingly dark in colour.

Fig. 1 clearly indicates the appearance of the various

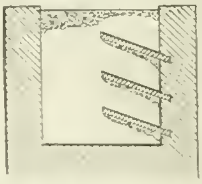
Fig. 1.



(Natural size.)
Sb = 50 %, 40 %, 30 %, 20 %, 15 %, 12 %.
Pb = 50 %, 60 %, 70 %, 80 %, 85 %, 88 %.

sections, whilst Fig. 2 is a magnification of 50 diameters of the white layer in the 80 per cent. antimony alloy.

Fig. 2.



(One-sixteenth actual size.)

Having now crystallised out a definite structure, the next question which arose was whether or not the alloy had separated into two fluid layers, one lighter than the other, previous to solidification, or whether the crystals had formed throughout the alloy, and afterwards floated up to the surface in a manner analogous but the reverse of that which occurs in crystallising salt.

Messrs. C. R. Alder Wright and C. Thompson communicated a most important paper to the Royal Society, in 1889, on certain ternary alloys, in which the metals lead, tin, and zinc in varying proportions were melted together, and the resulting mixtures, after slow cooling, were examined. It was found that if the tin is less than ⅗ths of the whole mass, the alloy divides itself into two different ternary alloys, lead preponderating in the heavier one; and they point out that this phenomenon is entirely distinct from the segregation of alloys during solidification, in consequence of the formation of other differently fusible alloys, and they also point out that the alloys separate as fluids, and then crystallise afterwards. In order to compare the micro-structure of such alloys with that I had obtained, a mixture of tin, lead, and zinc was prepared in the manner described by those gentlemen. A section of the mass was then made and was examined under the microscope. The appearance clearly showed that the alloys had separated when in a liquid condition, for there was a clearly defined line between the upper and lower alloy, which was perfectly horizontal, the alloy above having an entirely different structure from that below the line of demarcation. In the lead-antimony alloys no such clearly cut line was present; in fact, as a rule, the upper layer was not horizontal, but curved downward on each side, and the matrix which constituted the lower part of the alloy could be traced continuously between the crystals to the very top of the mass. It was also noticed that several of the white bright crystals were present at the very bottom of the alloy, and it appeared that these had attached themselves to the bottom of the crucible, and were not, therefore, free to float through the mass. Although such considerations would lead to the assumption that the crystals formed in the matrix at a temperature considerably above its melting point, and then, in virtue of its less gravity, floated to the top, it was considered unsafe to form any conclusions without more practical demonstration. In order to settle the question beyond doubt, a considerable amount of an alloy containing 30 per cent. of antimony and 70 per cent. of lead, was melted and afterward poured into a mould which had been previously heated, and which was provided with rigid shelves, attached to one side of the mould only, and which projected half way across, and which were fixed at an angle of about 30 degrees in an upward direction.

The following, Fig. 3, will make the matter more clear:—

Fig. 3.

(× 50 V.)
Lead = 20 %. Antimony = 80 %.

It may be assumed that if the lighter mass was in a fluid condition before it reached the surface, the shelves would not impede its progress. A liquid ascending would of course flow readily up the inclined planes and escape to

the surface, whereas if it crystallised in the body of the mass, on ascending to the under sides of the shelves the friction would be too great to allow the crystals to travel upwards, and on making a section of the cold alloy, from top to bottom, we should then find layers of crystals under each shelf.

After pouring the alloy into the mould, it was allowed to cool very slowly, and when cold it was sawn from top to bottom, cutting it at right angles through the shelves. The whole mass was then polished and etched, and it was found, as was naturally predicted, that the crystals on one half of the mould, where the shelves existed, were equally divided between the under sides of the three shelves and the surface. The above diagram is a graphic representation of the results obtained.

The next point of interest to determine was the composition of the hard white crystals. For this purpose a considerable quantity of the 30 per cent. antimony alloy was melted, and cooled somewhat rapidly in a shallow iron mould, the object being to obtain the crystals unattached one to the other and suspended in the eutectic compound. This bar, measuring 6 ins. by 2 ins. by $\frac{1}{4}$ in., was then filed bright, and was immersed in a 10 per cent. nitric acid solution in water, and it was allowed to remain there several days in the cold. On removing the bar from the acid, it was found to be coated with a soft, slimy material, in which the granular crystals were retained in position. By brushing off this deposit and washing with water, the dark-coloured slime was removed, and the apparently pure white crystals separated as a heavy granular deposit. In order to thoroughly eliminate all adherent matter, these crystals were boiled with dilute nitric acid, but previous to doing this they were thoroughly well rubbed with the finger and thumb to separate adhering crystals, which might possibly enclose a portion of the eutectic alloy. After digestion with acid, they were thoroughly well washed in running water, and were afterwards dried. On examination under the microscope, it was found that they consisted of more or less perfectly formed crystals of cubical form, and it was also observed that the angles had been but slightly acted upon by the acid, and as their surfaces were bright and continuous, we may be justified in assuming that the acid had not acted in any way upon the interior of their mass. The specific gravity of these crystals proved to be 6·5, which approximates to that of pure antimony, which is 6·7. An analysis showed them to contain 99·8 per cent. of antimony and 0·2 per cent. of lead; in other words, they consisted of practically pure antimony. On examining the alloys containing more than 50 per cent. of antimony, the crystals increased in volume until, when 95 per cent. antimony was reached, practically the whole field under the microscope appeared to consist of antimony, but even in this alloy, a small quantity of the eutectic compound was most distinctly visible, surrounding more or less completely the square masses of antimony. As it was impossible to separate the crystals from the alloys containing a very large proportion of antimony, I am not in a position to state from actual trial that they do not contain some lead. The relatively small areas of the dark eutectic compound would lead us to believe that the white crystals in the 80 per cent. and the 90 per cent. alloys were not pure antimony.

The next question to determine was the character and composition of the eutectic or most fusible compound.

This was done by two different methods: first, by synthesis, and second, by analysis. It had been observed that there were hard crystals in the 15 per cent. alloy, but none in that containing 10 per cent. antimony. Several mixtures were made, containing between 15 per cent. and 12 per cent. antimony, and, after melting under cyanide and allowing them to cool slowly, they were cut up and examined. As the antimony was reduced, the hard upper layer decreased, until on arriving at a mixture containing 12·5 per cent. antimony, and 87·5 per cent. lead, no separation of crystals could be detected, although they were clearly present in the alloy containing 13 per cent. of antimony. If we take the atomic weights of lead and antimony to be 207 and 120 respectively, we find that a chemical compound having the formula Pb_4Sb would contain 12·66 per cent. antimony and 87·34 per cent. lead. On now melting the metals together in that proportion and allowing to cool slowly, we obtained an apparently homogeneous mass, free from separated crystals.

There was no difficulty in obtaining this material in a perfectly pure state, as it was only necessary to cut off the upper portion containing the free antimony of any of the alloys containing above 13 per cent. of antimony, and to use the lower portion for analysis. Two carefully conducted determinations gave 12·8 per cent. and 12·7 per cent. of antimony and 87·2 per cent. of lead, and this corresponds nearly with the formula Pb_4Sb. Although it was an easy matter to obtain the eutectic alloy from the alloys containing under 50 per cent., it was impossible to separate this material from those containing 50 per cent. and over of antimony. Recourse was therefore made to another method of research, namely, the determination of the freezing or critical points in cooling the liquid alloys. Each alloy was therefore carefully melted in a small charcoal crucible to a point considerably above the fusing point, and by means of the Le Chatelier pyrometer a careful determination of the critical points was obtained. In applying the couple to the molten mass, the two twisted wires were first of all covered very carefully with moist asbestos paper, in which condition it could be most readily moulded, so as to completely protect them from actual contact with the fluid metal. Previous to the introduction of the couple into the metal, the asbestos enclosing the couple was heated to bright redness in a Bunsen flame, by which means the asbestos assumed a fair amount of rigidity. The actual thickness of this coating did not much exceed $\frac{1}{100}$th of an inch, and was so strong as to last for more than 20 determinations of temperature without breaking. The pyrometer having been previously most carefully calibrated according to the method given by Prof. Roberts-Austen and others, and the alloys having been melted, it was only necessary to lower the asbestos couple into the molten mass, and to pack loose fibrous asbestos all round it, over the surface of the liquid in the crucible, and then to carefully observe the points at which the speck of light became stationary or moved very slowly. The eutectic alloy had only one freezing point—at about 247° C. The melting point of lead being 325°, and that of antimony 630°, the mean of the two metals is 388°. It will be seen that the eutectic alloy had a solidifying point of $388° - 247° = 141°$ below that of the mean of the two metals in the mixture, and 78° below that of lead. The alloy containing 95 per cent. of antimony had a first critical point of about 615° C., and another of very short duration at 247° C., and all of the alloys had this one common critical point of 247° C., showing most clearly that the eutectic alloy in the 95 per cent. alloy was of the same composition as that containing about 13 per cent.

The specific gravity of the eutectic or lighter alloy was 10·48. Theoretically, the mean of the lead and antimony should have given a specific gravity of 10·77.

It is not surprising that the antimony crystals floated up to the top of the mass, considering that they were so much lighter than the eutectic alloy; in fact, the latter was more than $1\frac{1}{2}$ times as heavy as the antimony.

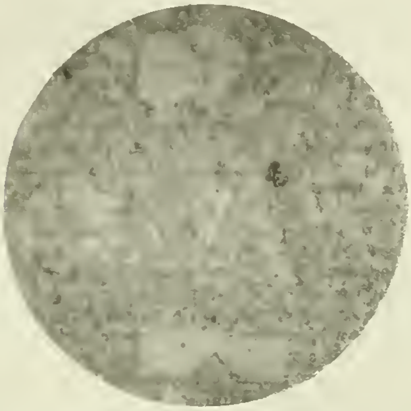
Attempts were made to determine the crystalline structure of this eutectic alloy, but, as before stated, I failed to determine it by the usual method of polishing and etching. When, however, a sufficient quantity of the material was allowed to cool or solidify very slowly, the surface of the bright alloy presented a frosted appearance, and on examining this under high power, crystals of an hexagonal form were clearly detected.

When the proportion of lead is increased above 87 per cent., the pyrometrical results indicate two melting points; the higher, or first freezing point increasing with the increase of lead, until, when the whole of the antimony is eliminated, the melting point is that of lead, or 325° C. Until that point is reached, however, there is a clear retardation at 247° C., showing that in this case the same eutectic alloy remains, as is the case in the alloys containing less than 87 per cent. lead.

In one case lead is dissolved in excess of the eutectic alloy and in the other case antimony is in excess in the same mother liquor.

That antimony and lead should combine in atomic proportions in the eutectic alloy is not contrary to the conclusions of Dr. Guthrie, who has thoroughly investigated eutectic compounds, his results having been communicated

Fig. 4.



(× 300 V.)
Eutectic Alloy of Lead-Antimony Compound (Pb_4Sb).

to the Royal Society in 1884 (Philosophical Magazine, Vol. 17, 1884, page 462), for that gentleman, although pointing out that such eutectic metals do not as a rule exist in atomic proportions, makes the following statement :—

"When we are dealing with metals, which, like antimony and arsenic, on the one hand, are themselves halogenous, or with such strongly chemico-positive metals as sodium, on the other, we may and do get alloys of atomic composition," "Metallo-metallic salts." It would not appear that Dr. Guthrie had studied antimony and lead compounds; in fact, all his communicated results dealt with the compounds with bismuth, zinc, tin, lead, and cadmium, and with mixed salts; and he clearly demonstrated that the eutectic, or most fusible constituent of each alloy did not have the metals in any simple atomic proportion. To use his own words, "The constitution of eutectic alloys is not in the ratio of any simple multiple of their chemical equivalents, but their composition is not on that account less fixed, nor are their properties less definite."

The same gentleman showed that there was a close relation between the eutectic compound metals, and the cryohydrates of common salts in water, and states, "For as a salt may unite with water, as when anhydrous chloride of calcium does so, to fix the water as crystalline water, and also may unite with water as a cryohydrate, so two metals may unite in one proportion, while they form a definite eutectic alloy in another."

Henri Gautier (Compt. rend. 1896, 123, 109—113) makes the following interesting statement with reference to the fusibility of metallic alloys :—"Fused alloys, on solidification, behave in a similar manner to fused mixtures of salts, and hence may be divided into three classes corresponding with the three classes of saline mixtures recognised by Le Chatelier."

"(1.) The metals do not combine in any fixed proportions, and the two constituents crystallise quite distinctly; for example, alloys of tin with zinc, bismuth, or lead."

"(2.) A definite compound of the two metals crystallises out, but these crystals are scattered in a crystalline matrix of the metal which is in excess; for example, copper with tin or antimony."

"(3.) Alloys corresponding with isomorphous mixtures of salts; for example, gold and silver."

"The fusibility curve of an alloy at once indicates to which of these three classes the alloy belongs."

In concluding this section it is necessary to point out that various chemical and metallurgical text books contain most erroneous statements with regard to the melting point and equivalent of antimony.

The chemical equivalent is given in many books as 122 instead of 120, and the melting point as 430° C. instead of 630° C.; in fact, my own observation proves that antimony and aluminium have practically the same melting point, namely, about 630°, and Henri Gautier gives the melting point as 632°.

Antimony and Tin.

It was not my intention to deal in this paper with other than the lead-antimony alloys, but as I have done much work with the antimony and tin compounds, although the investigations are far from complete, the results are of such interest that I am sure I will be pardoned if I introduce a brief account of the work as far as it has been carried out.

The same method of procedure was followed in making these alloys as was adopted in making those previously described, and the specimens were polished and etched in the same manner. Up to a point when the antimony did not exceed 7·5 per cent. there was no apparent separation of well-formed crystals in the mass, but the bright surface of the alloy rapidly clouded over, leaving a dark deposit upon the surface, showing clearly that at the solidifying point some of the antimony had been retained in combination, or solution in the tin, and white meshes of a harder constituent were found more or less irregularly distributed between the joints of the crystals, indicating clearly the presence of some compound which had crystallised out at the moment when the mass was solidifying.

With 8 per cent. of antimony, the matrix presented the same appearance as the 7 per cent. alloy, with this difference, a few cubical crystals, very hard, and little acted upon by dilute acid, were observed on the upper surface of the solidified alloy, when the cooling was done very slowly. When, however, the fluid mass was cast in an ordinary mould, the crystals were distributed evenly throughout the mass. With 9 per cent. the number of crystals were relatively increased; with 10 per cent. a still larger proportion of cubical masses were found distributed throughout the mass, in the moderately rapidly cooled alloy, and each addition of 5 per cent. of antimony was accompanied with a corresponding increase in the proportion of the separated crystals, until 40 per cent. of antimony and 60 per cent. of lead were present. The alloy with 25 per cent. of antimony and 75 per cent. of tin gave a really unique and beautiful section, the crystals embedded in the matrix being of the most perfect form and shape, and indicated beyond all doubt that they were perfect, or almost perfect cubes. The following Figures, 5 (omitted) and 6, clearly prove this statement.

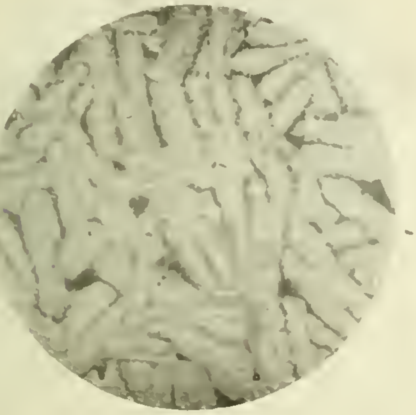
Fig. 6.

(× 50 V.)
Tin = 75 %. Antimony = 25 %.

When the amount of antimony was increased to 45 per cent. and the tin 55 per cent. the structure no longer presented the same appearance, for, instead of cubical masses, the crystalline mass evidently consisted of plates

(Fig. 6A), crossing in almost every conceivable angle, the whole surface of the mass, the dark matrix clearly

Fig. 6A.



(× 50 V.)
Tin = 50 %. Antimony = 50 %.

showing itself between the crystals. In proportion as the antimony was still further increased, until it reached about 54 per cent., the structure appeared to have the same character; but on continuing the action of the acid it appeared that the centre of the plates was harder in character than the exterior portion, and less acted upon by nitric acid. With 53 per cent. of antimony there still remained the dark coloured matrix. When the antimony was increased to 55 per cent. this dark matrix had entirely disappeared, and after the action of very dilute nitric acid, for a time quite sufficient to develop the dark matrix in the alloys just described, the field remained perfectly bright, and apparently homogeneous; but on acting for a few seconds on the bright surface with nitric acid of 1·42 sp. gr., a well-defined structure was developed, and it was clear that it consisted of two different constituents, one relatively more easily attacked by the strong acid than the other. The constituent least acted upon had every appearance of the crystalline forms which antimony assumes when crystallising out of the lead-antimony alloys; and although I have not yet actually separated them, we may be fairly safe tentatively in assuming that they actually do consist of antimony, for in proportion as more antimony was added to the alloys this harder constituent increased in proportion, until when 90 per cent. of antimony and 10 per cent. of tin were present, it occupied the greater part of the microscopic field. As the crystals in the 25 per cent. antimony alloy were so perfectly formed, and were sufficiently distributed through the matrix, a successful attempt was made to effect their isolation. This was most readily and perfectly effected by a long-continued action of comparatively strong nitric acid (1 part of acid to about 9 parts of water). The bar, after the action had been continued for several days, had much the same appearance as the lead-antimony alloys when similarly treated, and the dark-coloured slime containing the crystals was removed, and the turbid liquid decanted from the residue. The latter was rubbed up on the bottom of a mortar with an india-rubber pestle to detach the pulverulent apparently amorphous grey powder. It was then washed several times with water, and finally with dilute hydrochloric acid, to remove any mechanically adherent tin and its oxide, which was found to be present in more or less quantity. In this way a most perfect crop of crystals was obtained, and these, after drying, had their specific gravity and analysis determined. The specific gravity was 6·96; the specific gravity of tin being 7·29 and that of antimony being 6·71. If the antimony and tin were present in equal atomic proportion, and had not suffered any alteration in volume when combining, the specific gravity should have been 7·00.

Several portions were then tested for antimony and tin, using two different methods for their determination. The mean results obtained were as follows:—

	Per Cent.
Antimony	50·54
Tin	49·46

If the elements had combined in simple atomic proportions of one equivalent of tin and one equivalent of antimony, the calculated composition would have been—

	Per Cent.
Antimony	50·21
Tin	49·79

which shows most conclusively that the compound approximated to the composition of SnSb, and may be described as tin antimonide, a "metallo-metallic salt," as it would be called by Dr. Guthrie.

The following photograph (Fig. 7) was taken of one of the crystals after separation from the 25 per cent.

Fig. 7.

(× 200 O.)
Separated Crystal of SnSb (Antimonide of Tin).

alloy, and clearly indicates that it is cubical, but that as it has one corner absent, it may be considered to belong to a system between the octahedron and cube.

An attempt was made to produce an homogeneous mass of the above composition by melting the two metals in the proportions indicated, but, as before stated, such a simple compound could not be produced by this process; no cubical crystals were present, but the peculiar plates surrounded by the dark matrix were invariably the result.

A large quantity of the crystals themselves were then melted under cyanide of potassium, but with no better result, we are therefore forced to conclude that this peculiar chemical compound SnSb can only exist in presence of a large excess of an alloy containing a preponderating quantity of tin. An analogous case is well known in pig iron, or in compounds of carbon and iron, for it is now recognised that the exceedingly hard bright constituent of the most highly carburised white iron and steels consists of a definite compound Fe_3C, which can be readily separated as such from the matrix by suitable means, and that if the carbide so separated is melted in a crucible, it is instantly decomposed into a compound containing about 4·5 per cent. of carbon and 95·5 per cent. of iron, and into free graphite, which rises to the surface of the mass and is eliminated.

I have not fully completed the pyrometrical records of this series, but one point has been determined, and that is, that in all the compounds containing between 7·5 per cent. and 50 per cent. of antimony, there is a common freezing point of 256° C., which it will be noticed is close to that of

NOTE.—Prof. H. Behrens described an alloy of 90 per cent. of tin and 10 per cent. of antimony (Das Mikroskopische Gefüge der Metalle und Legierungen), but he does not appear to have examined a complete series.

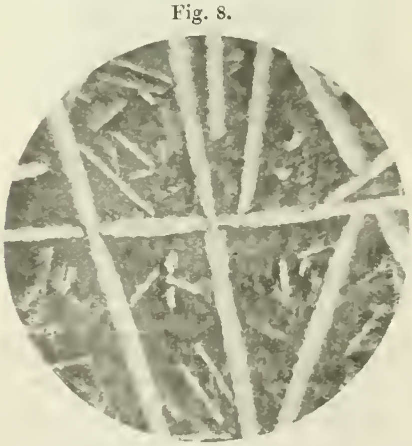
the eutectic compound of the lead-antimony series. This is an exception to the general rule, that binary alloys fuse more readily than the less fusible constituent, for tin has a melting point of 231, antimony 630, the mean of the melting points of the constituents of the eutectic compound of about 92·5 per cent. tin and 7·5 per cent. of antimony is 261° C., therefore the actual melting point is 5 degrees below this, but is 25 degrees above that of the most fusible constituent, tin. An analogous case to this has been found by Henri Gautier with antimony-aluminium alloys containing more than 2 per cent. of antimony, which are less fusible than aluminium.

This subject is receiving further investigation, and it is hoped that before long it will be completed.

Tin and Phosphorus.

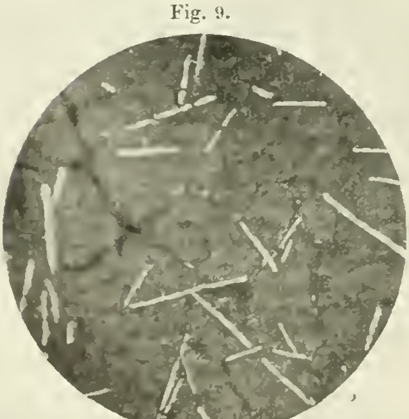
Phosphor tin is a compound well known in commerce, and is extensively used for the production of phosphor bronze. It is characterised by its extreme brittleness and its spiegel-like fracture. A section of this compound polished in the manner previously described is a beautiful object under the microscope. It has the appearance of brilliant lines, perfectly straight, which pass in every conceivable angle over the whole object, and the matrix between is of exceedingly soft character, and as is the case with all similarly constituted alloys by polishing alone, the structure is well developed. On acting on the surface with dilute acid, the matrix is slightly darkened, leaving in brilliant relief the harder constituent. A series of compounds containing between 0·04 per cent. of phosphorus and 5 per cent. was carefully prepared, and each of them

Fig. 8.



(× 50 V.)
Tin = 97·5 %. Phosphorus = 2·5 %.

Fig. 9.



(× 70 V.)
Tin = 99·83 %. Phosphorus = 0·025 %.

was subjected to microscopic examination. It was found, as one would naturally expect, that as the phosphorus was reduced, the hard constituent also decreases, until when only 0·04 per cent. phosphorus was present a few hard specks remained in between the junction of the crystals. The diagrams shown fully illustrate these structures (Figs. 8 and 9).

It was found that just in the same way that antimony and antimonide of tin could be separated from alloys by the action of dilute nitric acid, so the hard constituent in phosphor tin could be similarly isolated. Obtained in this way, it presented the appearance of brilliant white plates, very similar to graphite, but of course much brighter. Several analyses of this compound demonstrated that it consisted of a chemical compound of the composition Sn_3P_2. Fearing lest nitric acid might have acted upon one of the constituents more than on the other, an attempt was made to dissolve out the tin with mercury, as it was found that the phosphide was quite insoluble in that solvent, and that after heating comparatively large pieces of the phosphor tin with that solvent for some hours, the scales floated to the surface, and could be removed more or less mechanically contaminated with mercury, which appeared to have the property of wetting the surface of the plates without actually combining with them. By strong pressure, the greater part of the mercury could be removed. The mass so obtained was then subjected to an analysis, with results sufficiently near to those first obtained to justify the conclusion that the phosphide obtained by the first method had been unaltered in the process of separating it. This phosphide, on heating in a stream of hydrogen gas, was decomposed, free phosphorus passing off, and a compound of phosphide of tin and free tin remaining behind. On treating any of the compounds containing phosphorus, with strong hydrochloric acid phosphine gas was liberated, which burst into flame on coming into contact with the air. In cooling the fluid compounds, the phosphide of tin crystallises out before the tin, and if the quantity of phosphorus in the compound is comparatively small, and the cooling very gradual and slow, the crystallisation invariably commences at the outer portion of the alloy, and travels in straight lines towards the centre. In fact, the structure is such that the slowly-cooled alloy resembled the appearance of a section of a pan of soda crystals, the mother-liquor remaining in the centre, the crystals radiating from the exterior of the pan to the centre. If the phosphor-tin compounds are allowed to cool sufficiently slowly to a point a few degrees above the melting point of tin, the latter may be most readily pressed out from the solidified crystals of phosphide; but of course the latter cannot be obtained in a very pure state by this means. In cooling a 3 per cent. phosphorus and 97 per cent. tin compound, the following critical points were obtained:—500° and 235° C., the higher being the point at which the phosphide began to crystallise, the lower being the freezing point of the tin.

The plates, after careful separation, were examined carefully under the microscope, and the two following diagrams, Figs. 11 and 12, indicate clearly that they crystallise in hexagonal plates.

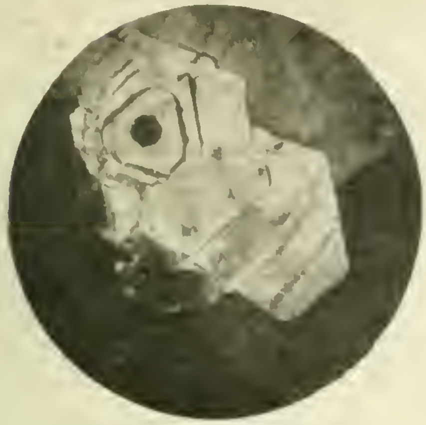
Tin and Arsenic.

Arsenic is frequently met with in commercial tin, and the microscope is a valuable means of detecting its presence. It behaves in a similar way to phosphorus, the only difference being that the plates of arsenide of tin are a little more rough on their sides than those of the phosphide, and judging from the fact that the matrix is sensibly darkened by dilute nitric acid, we may assume that, at the point of setting, the tin still contains a minute quantity of arsenic. The arsenide crystals are quite as readily obtained as those of the phosphide, and an analysis of them after separation gave a compound containing—

	Per Cent.
Tin	69·45
Arsenic	30·55

As it was found, however, that the action of acid removes a portion of the tin, leaving the arsenic still in the form of

Fig. 11.



(× 50 V.)
Tin Phosphide (Sn_3P_2). Separated from Alloy.

Fig. 12.

Tin Phosphide (Sn_3P_2). End of a separated Plate showing how the Crystals grow.

plates, I should not like to say definitely, at present, that the arsenide has that composition. It is most likely to have a corresponding composition to that of phosphide of tin, or Sn_3As_2, which would contain—

	Per Cent.
Tin	70·42
Arsenic	29·58

Further trials are in progress to definitely determine its true composition.

Unlike phosphor tin, it is possible to obtain fusible compounds of tin and arsenic, containing as much as 43 per cent. of arsenic; and I have obtained such a compound by simply dropping metallic arsenic into tin heated to bright redness. The compound was perfectly fluid at a red heat, but gave off copious fumes of arsenic when being poured into the mould, and solidified, or began to solidify, when still dull red.

The critical points of a tin-arsenic alloy with 20 per cent. of arsenic are at 530° and 235° C.

Both arsenic and phosphorus lower the melting point of steel and iron, but evidently have precisely the opposite effect with tin; at least, the first freezing point is many degrees above the melting point of tin itself.

Several photographs which were shown on the screen represent the micro-structure of tin containing varying and decreasing quantities of arsenic, and they were all prepared in exactly the same way, the same weight being made of each, and the conditions of cooling were identical. In reality, the total compound in each case was only 10 grms., and as the small crucibles were taken from the fire and placed on a metal block to cool, the time taken to set must have been of very short duration. With the higher percentages of arsenic, the plates of arsenide pass right through the section, whereas with smaller quantities, although apparently distributed at fairly equable distances apart, differ as the proportion of arsenic is reduced in their length and thickness. The plates freeze out at a very much higher temperature than the matrix, and it is quite possible to squeeze out the fusible mother-liquor, if we may so call it, leaving a mass of impure arsenide behind.

This compound crystallises in the same system and manner as the corresponding compound of phosphorus.

Bismuth and Tellurium.

Some observers have found that 0·2 per cent. tellurium in pure bismuth results in a perfect change of structure, from that of a coarse spiegel-like fracture of pure bismuth to one of fine grain.

On experimenting with small quantities of the bismuth, such as is sold as pure by chemical dealers, there was practically no difference between the pure bismuth and that containing 0·2 per cent. tellurium when they were cooled very slowly. The fractured surfaces were almost identical. The micro-structures were similar, the essential difference being that the alloy contained hard irregular shaped crystals, distributed between the grains of bismuth.

With 3 per cent. of tellurium, cubical crystals were distributed throughout the matrix on the upper portion of the alloy, and the secondary crystals below in the matrix were not so well formed as those in pure bismuth.

The results do not disprove those of others, probably the conditions of cooling are responsible for differences observed. The subject is receiving further investigation.

Nitro-Cyanide of Titanium in Spiegeleisen.

My investigations have fully confirmed those of Mr. T. W. Hogg, that this compound is present in spiegeleisen, but I found it not always necessary to dissolve the material, in order to obtain the cubic crystals. They can most readily be detected on the surfaces of the bright thin tabular plates, which are to be found in the hollow portions of the pigs These plates are interesting objects themselves without any treatment, and the cyanide crystals are easily detected in widely separated aggregations.

Micro-Mechanical Examination of Alloys.

During my investigations, I have found it of great value, after having examined sections under the microscope, to determine by mechanical means, which part of the structure, if any, reduced the strength of the whole mass. The device employed is exceedingly simple, and yet is of the most invaluable character. The section after careful examination is placed, polished surface downwards, into a small V-shaped opening cut out of a solid square bar of steel. A round piece of steel is placed on the back of the specimen, and either slow or percussive pressure is then applied, until the specimen is distinctly curved. It is then without further treatment placed under the microscope, and if there is any brittle element present, it will be at once detected, as a microscopic fracture will have been effected at that place. If the first application of pressure does not develop any fissures, it is further pressed, or struck until if necessary, the sample is bent over right upon itself, when it is examined, and the appearance carefully noted.

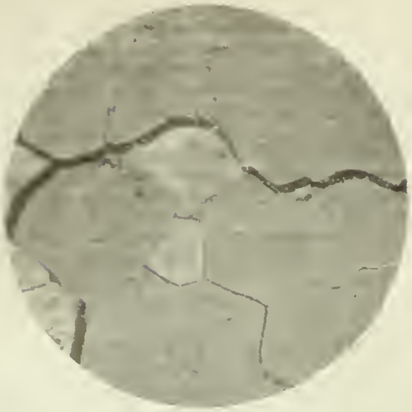
The micro photographs, Figs. 14 and 15, of the samples which have been treated in the way described, fully demonstrate the great importance of this method of examination.

The following are examples of the value of this method of testing:—

1. Vertical sections from the worn surfaces of steel rails, tyres, &c. almost always show weakness at the extreme surface, this part breaking first, and the crack thus developed, then traverses downward into the sound physically unaltered material.

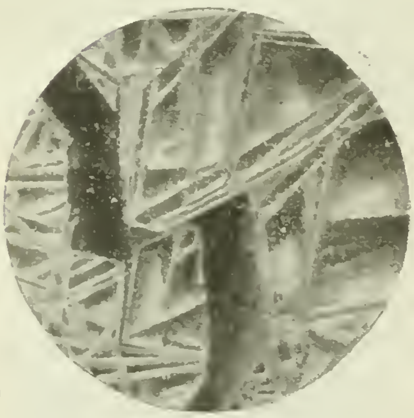
2. Micro-sections of structural steel, on being bent, first across the piece, and the direction of rolling, and second at right angles to that direction, almost always show the greater weakness in the latter case. The microscopic structure, as a rule, most clearly indicating the direction in which the rolling has been done.

Fig. 14.



(× 50 V.)
Copper = 99 %. Bismuth = 1 %. (After bending in V-block.)
(Dark lines are fissures.)

Fig. 15.



Surface of a small Ingot of an Alloy of 80 % Tin and 20 % Arsenic, showing Arsenide Crystals and fracture made in V-block.

3. Steel castings which show microscopic intergranular foreign matter, such as has been described by Prof. Arnold and Mr. T. Andrews, of Sheffield, always break away at the joints of the grains where the deposit exists.

4. Intercrystalline weakness is always detected in copper, especially when the element bismuth is present.

5. All the weak parts of white metal alloys are clearly revealed by this method. Straight lines, or plates of a brittle constituent, in a softer alloy, are split down their centres, and the complex fracture traverses these brittle tracks, which constitute lines of least resistance.

Small or great quantities of arsenic, phosphorus, and copper, in tin, give good examples of this when tested by this method.

It is not all the metals and alloys, although they contain relatively strong and weak structures, which break away when tested by this simple process, it is therefore important to find another method for testing, to meet such cases.

In conclusion, I must apologise for the great length of this paper. My excuse is that it is not nearly as great as the importance that this somewhat neglected branch of science is almost certain to attain to. More workers are wanted; the field is unbounded. The work appeals both to the analyst and to the engineer. The results which will be obtained by systematic research are certain to add to our scientific knowledge, and practical applications are sure to follow.

Nottingham Section.

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SESSION 1896–97.

April 28th (Leicester).—Mr. S. J. Pentecost, F.I.C. "The Removal of Mineral Oil Stains from Dyed Fabrics."
May 26th (Nottingham).

Meeting held on Wednesday, February 24th, 1897.

MR. F. J. R. CARULLA IN THE CHAIR.

SCHEME OF ANALYSIS FOR PHOSPHATES.

BY R. M. CAVEN, B.SC., F.I.C.

THE following table for the qualitative separation of metals whose phosphates are precipitated from acid solution by

TABLE FOR ANALYSIS OF PHOSPHATES.

If PO_4 is found to be present in the filtrate from Group II. after boiling off H_2S and adding a little HNO_3, and a precipitate forms on adding to the solution NH_4Cl aq. and NH_4HO aq., redissolve this precipitate in the least possible quantity of HCl aq., then add a few drops of $H\bar{A}$ aq. and a considerable quantity of $(NH_4)\bar{A}$. aq. If a precipitate forms, filter.

<table>
<tr><td>Precipitate may be—
$FePO_4$
$AlPO_4$
$CrPO_4$</td><td colspan="4">If no precipitate forms, Fe, Al, Cr may still be present. Add to the solution or filtrate a few drops of $(NH_4)_2HPO_4$ aq., and if a precipitate forms continue adding this reagent until no further precipitation takes place. Filter if necessary, pouring the turbid liquid through the same filter as before, if one was used.</td></tr>
<tr><td colspan="3">Residue.—Dry the precipitate on the paper and fuse it with solid KHO, free from Al, on Pt foil. Boil the mass with water, and filter.</td><td colspan="2">Filtrate or solution may contain Cr, which is not completely precipitated as $CrPO_4$ by $NH_4\bar{A}$ aq. in presence of $H\bar{A}$ aq. Add $FeCl_3$ aq. carefully until the precipitate first formed remains reddish-brown in colour; boil for several minutes and filter.</td></tr>
<tr><td rowspan="2">Residue consists of Fe_2O_3. Dissolve in HCl and add KCyS. A red colour shows Fe.</td><td colspan="2">Filtrate.—Divide into 2 parts.</td><td rowspan="2">Residue consists of $FePO_4$ and basic ferric acetate, and may contain a little Cr. It may be neglected unless Cr has not already been found, and traces are to be looked for, when a fusion with KHO must again be performed.</td><td rowspan="2">Filtrate should be free from Fe and PO_4. Add NH_4Cl aq. and NH_4HO aq., and examine by general table for groups III. B., IV., and V.</td></tr>
<tr><td>If yellow shows Cr, a trace of Mn may appear here and colour the solution green. In this case add $H\bar{A}$ aq. and $Pb\bar{A}_2$ aq. If the precipitate formed is yellow, the presence of Cr is proved.</td><td>Add NH_4Cl aq. in excess and allow to stand. A white gelatinous precipitate shows Al.</td></tr>
</table>

ammonium hydrate, is the outcome of some recent experiments on the subject; and although much originality cannot be claimed for the general method adopted, the table differs in some important particulars from any scheme which has previously been published. When ammonium acetate is added to a solution containing Fe, Al, and Cr, and in which free mineral acids have been replaced by a very small excess of HA; $FePO_4$ and $AlPO_4$ are entirely, and $CrPO_4$ is partially precipitated in the cold, provided excess of PO_4 is present. If only a small amount of PO_4 is present, the phosphates formed remain partly, or even entirely, in solution in the corresponding acetates, and the solution is not easily freed from PO_4 by boiling. Additional phosphate is therefore added, if necessary, in the form of ammonium phosphate, to complete the precipitation in presence of ammonium acetate.

PO_4 will now be in excess, and ferric chloride is added and the solution boiled, by which means all the PO_4 is removed from solution and the slight excess of iron precipitated in the form of basic acetate. If any chromium remained in solution before the addition of ferric chloride, it will be precipitated during the subsequent boiling.

The method adopted for the detection of Fe, Al, and Cr in the precipitate by ammonium acetate has been described in a former paper (this Journal, 1897, 29).

The table has given good results in the hands of several experimenters. It is susceptible of some simplification at the expense of accuracy.

Discussion.

Dr. F. Clowes said that the paper was principally interesting to him as leading up to the improved phosphate table prepared by Mr. Caven. In his old student days they had a phosphate table which they admired largely because they could not understand it, and that table survived, with some emendations, to the present time. Mr. Caven's scheme was a simplification of that table, and he thought that any elementary student would easily understand the principles involved. He should be very glad, with Mr. Caven's permission, to make use of it in future text-books with which he was concerned. There was only one point which he should be inclined to criticise. Chromium, if present, might apparently be divided into two portions; accordingly, if only a trace of the metal had to be detected, there might be risk of its detection being missed when the small amount had been thus divided. He did not see, however, any way out of this difficulty. Mr. Caven had certainly effected a very great improvement on the old method of analysis.

Mr. L. Meggitt added a few remarks on the quantitative estimation of aluminium and iron phosphates which occur in the mineral phosphates of commerce.

Dr. J. D. Granger wished to remark that it was not stated which metals were present as phosphates and which were not, in the results obtained by this table. In the old tables they were supposed to ascertain which metals were present as phosphates and which were in combination with other acid radicles. Apparently Mr. Caven's method could be carried out much more quickly that the old method. Mr. Meggitt gave them a number of methods which, for quantitative work, were very accurate, but which were too lengthy for qualitative analysis. At present, when time was of so much value, they strove for the quickest method which would give satisfactory results, and Mr. Caven's scheme appeared to comply with these conditions.

Mr. G. J. Ward asked whether the table could be used for quantitative analysis as well as for qualitative.

Mr. Caven, in reply to Dr. Clowes, said that the case of chromium presented a difficulty because of the partial solubility of chromium phosphate in acetic acid, even in presence of ammonium acetate. But it would very seldom happen in practice that chromium need be looked for twice, though, in the case of small quantities of the metal having to be looked for, the possibility of division into two portions was an undoubted disadvantage. He was very glad that Dr. Granger had remarked about the older tables professing to find certain metals present as phosphates. When once a mixture containing phosphates had been dissolved in an acid it was impossible to say how the PO_4 had been originally distributed in the solid substance; it was more than doubtful whether the identity of the original substances was in any way preserved in the state of solution. So that the use of the term "present as phosphate" was purposely avoided in this table. In reply to Mr. Ward's question, the table was intended for qualitative work only, and the author was not prepared to say how far it might be used for a quantitative separation of the metals.

THE EXPLOSIBILITY OF LIQUID ACETYLENE.

BY PROF. FRANK CLOWES, D.SC.

The author, after referring to a former paper communicated by him to the Section, stated that Mons. Pictet now prepared acetylene gas in a dry state, and in an absolutely pure condition. Mons. Pictet maintained that in this state the gas might be safely liquefied by compression, and then stored, transported, and used for illumination.

The pure gas possesses a distinct but not unpleasant smell, and produces no chemical action upon metals. It appears to give rise to no danger from explosion.

According to the researches of Prof. Dixon and of MM. Berthelot and Vieille, a local source of energy, such as an electric spark, an explosion of fulminate, or a source of heat of sufficient intensity, when applied to acetylene gas under atmospheric pressure, does not produce explosion throughout the whole volume of gas, but only an explosion immediately around the source of energy. An illustrative experiment made by the author when communicating the paper, however, proved that an explosion caused by the firing of fulminate in a small quantity of acetylene gas would develop very great energy; a glass vessel being shattered, and the glass fragments being driven through a metal screen.

On the other hand, the researches just referred to prove that acetylene gas, when compressed to two or more atmospheres, is exploded throughout its mass by the local means stated, and under these conditions a very dangerous pressure may be developed.

Liquefied acetylene is a substance of considerable scientific interest. It exerts a pressure of about 30 atmospheres at ordinary temperatures. It is the lightest liquid known, and has the greatest coefficient of expansion of any known liquid or solid. It is also possessed of less refrangibility than any known liquid or solid. It furnishes about 300 times its volume of gas by evaporation.

As regards the explosibility of liquid acetylene, it is conceded that, under suitable conditions, it explodes *en masse*. Unfortunately, numerous serious and fatal explosions have occurred from liquid acetylene, but their extraordinarily destructive effect has led to considerable difficulty in tracing the precise cause of the explosion. In a recent explosion in Mons. Pictet's own laboratory the origin has been attributed to the fact that workmen neglected to pump out the acetylene from a steel cylinder before its cap was unscrewed, and the use of a spanner on the cap appeared to develop sufficient heat to explode the acetylene. In a more recent explosion in Berlin the explosion was said to be started by friction. Until the possible causes of explosion are more fully known, it seems impossible to feel that the liquid is safe for ordinary purposes.

Mons. Pictet describes the precautions which he has taken in order to avoid risks of explosion during the preparation and liquefaction of the gas. The gas must be pure and must be cooled during the process of compression. In order to obtain the gas with safety in a pure state, the lumps of calcium carbide are allowed to fall into cooled water, so as to be at once entirely immersed, and the gas is then purified by passage through dilute sulphuric acid, calcium chloride solution, solution of a lead salt, and solid calcium chloride. All these purifying substances must be kept cool. The impurities thus removed are mainly ammonia, arsine, phosphine, hydrogen sulphide, hydrocarbons, and moisture. The dry, pure gas is then stored over heavy petroleum oil.

Mons. Pictet attributes the intense light of the flame of acetylene to the same cause as the explosibility of the gas, namely, its dissociation into its elements with production of

a temperature exceeding that of the electric arc. This dissociation occurs very near the base of the flame, and only after the elements have cooled can they subsequently undergo ordinary combustion in the higher part of the flame.

Dr. J. D. Granger, Mr. J. Golding, and the Chairman took part in the discussion, and Dr. Clowes, in reply to questions from these gentlemen, stated that the intense heat near the jet from which the gas was burnt did not appear to injure the jet, whilst the heating and contamination of the atmosphere by an acetylene flame was considerably less than that arising from the equivalent illumination produced by coal-gas. He believed that no experimental proof had been furnished that acetylene could be exploded by a distant detonation.

Discussion.

Dr. J. D. Granger, who had just returned from Berlin, referred to the extensive character of M. Pictet's work in this matter. Unfortunately two most serious explosions had taken place in Pictet's own laboratory in Berlin. It had been found that friction was more likely to explode acetylene than any other cause, especially when it was in a liquid state. In that condition the friction caused by opening the valve was enough to cause an explosion. That the acetylene was split up into its component parts, hydrogen and carbon, during the explosion was proved by the large quantity of carbon found subsequently in the cylinder. M. Pictet had succeeded in removing most of the impurities, but there was one, and that a very dangerous one, which he had not yet proved to be removed, *viz.*, phosphoretted hydrogen. M. Pictet had passed the acetylene through a solution of lead acetate or lead nitrate, and that would probably remove much of this impurity. It was necessary, however, to remove every trace of it, or there was great liability to explosion. One thing that was most lamentable was that they did not know exactly what had caused most of the explosions. In some cases they seemed to have happened without any apparent cause. It had been stated several times that acetylene acted on copper, but that was chiefly owing to impurity; and copper could be replaced by some other metal, such as nickel, in order to avoid this danger. From all that was known there could be no doubt that in the near future acetylene would replace in many places, or supplement the use of ordinary gas, but there were many things which it could not do. At the present time it had one great competitor, and that was the incandescent light. The insurance companies on the Continent were beginning to issue policies under which provision was made for lighting houses with acetylene, but the restrictions were so great that scarcely anybody availed themselves of them. Until more experiments had been made on the explosiveness of acetylene, it was hardly likely to come into general use.

Mr. Golding asked whether the heat of its combustion was not an objection to the domestic use of acetylene. Again, if the burner were tilted on one side so that a part of the flame impinged against it, would not the extreme heat of the interior of the flame fuse its orifice?

Mr. H. Forth said he believed English insurance companies had absolutely refused to allow the gas to be used. They would have nothing whatever to do with it.

Mr. F. J. R. Carulla asked in what proximity it was necessary to have the fulminate in order to produce an explosion.

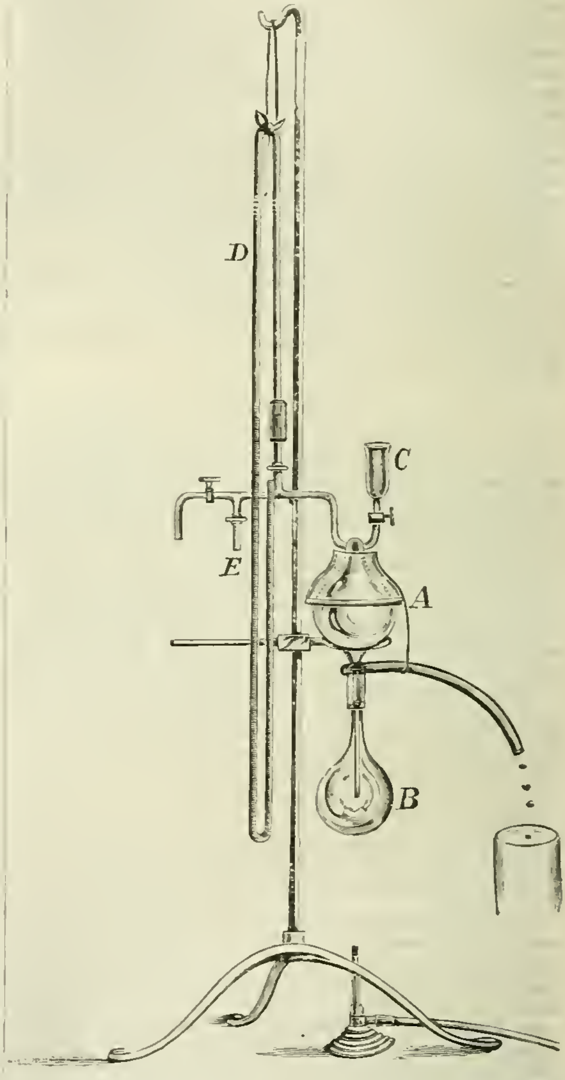
Dr. Clowes, replying to the questions asked, said he could not give any information on the last one. With regard to Mr. Golding's questions, they had tried the flames and had found, as a matter of fact, that they could hold their hands much closer to the acetylene flame than to the ordinary gas flame, for the heat disseminated in the air was much less. They had never had the difficulty of the burner being sealed up; but supposing the jet was tilted one way or another, he did not think it would play upon the jet, since the flame was a stiff one on account of the high pressure under which the gas issued. He knew as a matter of fact that there had been no trouble arising from this cause.

AN APPARATUS FOR COLLECTING THE GASES DISSOLVED IN WATER OR OTHER LIQUIDS.

BY PROF. FRANK CLOWES, D.SC.

Dr. Clowes then exhibited an apparatus devised by Dr. Truman for expelling and collecting the gases contained in solution in water, or in any other liquid.

The apparatus is represented in the accompanying figure :—



The flask B which receives the measured volume of liquid is connected by a jacketted rubber joint with the condensing vessel A, over which is spread a piece of fabric which is constantly moistened by a stream of water. The flask A is connected with the vessel C, into which the liquid may be introduced in measured quantity, and from which it is then allowed to flow through A into B. The liquid is poured into C and is drawn into A and B, which are vacuous, by opening the tap at the bottom of C.

The vessel A is connected also with two tubes provided with stop cocks and with a mercury gauge.

The apparatus is connected by tube E with a mercury Sprengel pump, and by the tube to the left of E with a high-pressure water-exhauster. And the process is commenced by exhausting the air from the interior of the apparatus as far as possible by the water-pump, then completing the exhaustion by the mercury pump. The measured

volume of water is then introduced into B from C, and is allowed to stand exposed to vacuum for several hours; the gas thus liberated is removed by the Sprengel pump and measured and analysed. The remainder of the gas which is in solution may then be removed by heating the liquid in B, and may be separately collected, measured, and analysed.

The apparatus is supported on a specially designed stand and since the support is mainly by suspension the risk of breakage is extremely small. There is sufficient play and freedom of movement to prevent the jar of a sudden shock from proving dangerous to the fragile glass.

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A. Hess.	Thorp Whitaker.

Hon. Local Secretary and Treasurer:
H. R. Procter, Yorkshire College, Leeds.

SESSION 1896–97.

Meeting held at the Queen's Hotel, Leeds, on Monday, March 1st, 1897.

MR. THOS. FAIRLEY IN THE CHAIR.

THE MANUFACTURE OF AMMONIUM NITRATE BY DOUBLE DECOMPOSITION.

BY T. FAIRLEY, F.R.S.E.

Within the last ten years ammonium nitrate, formerly only used in the laboratory, or in the production of nitrous oxide, has been employed in considerable quantity in the manufacture of "safety explosives."

A "safety explosive" for use in mines must leave no solid residue after explosion, and should not be capable of being fired by any ordinary means or mechanical violence, but only by a detonator. Ordinary explosives containing fixed solid substances may send out sparks of red or white-hot material when they are fired in blasting. This should not be possible with any safety explosive since it requires much higher temperatures to make gases red-hot, especially when they are allowed to expand.

Sprengel in 1873 (Jour. Chem. Soc. 796) showed that mixtures of nitric acid with other nitro-compounds give rise to very powerful explosives when fired by a detonator, but these Sprengel explosives have not come into general use. Sprengel also mentions the use of ammonium nitrate (Jour. Chem. Soc. 1873, p. 804) in place of nitric acid—his only objection to it being its hygroscopic nature. He found that this salt added to ordinary powder increased its explosive power.

The use of NH_4NO_3 for explosives is mentioned in patent of 1872, No. 2766, Norrbin and Ohlsen, but there is no mention made of it in Sprengel's patents of 1871.

Ammonium nitrate is an endo-thermic compound—80 grms. of the salt may be exploded so as to give nitrogen, oxygen, and steam, with an evolution of 64,000 heat units or gram-degrees. The heat evolved is probably increased in presence of a small percentage of such substances as nitro-benzene on nitronaphthalene, used in the safety explosives.

An important point in the increased use of these explosives is their relative cost, and as many of them contain from 50 to over 90 per cent. of ammonium nitrate, the question hinges largely on the cheap production of this salt. Generally it has been made by neutralising nitric acid by means of ammonia, and usually the liquid acid is saturated with ammonia gas, but in Hake's patent (this Journal, 1889, p. 706) both the constituents are mixed in the state of vapour. Very pure salt may be obtained by such processes—but more costly than if the salt could be obtained by double decomposition of the cheapest nitrate—sodium nitrate.

Little difficulty has been found in carrying out the manufacture of the corresponding potassium salt by the reaction of potassium chloride, carbonate, or hydrate on sodium nitrate.

The very great solubility of ammonium nitrate in water has, however, hitherto proved a difficulty in its manufacture by similar means—especially as the presence of 1 or 2 per cent. of fixed impurity renders the salt unfit for a "safety explosive."

The development of the ammonia-soda process suggested its application to sodium nitrate instead of sodium chloride.

Thus ammonia and carbon dioxide, or ammonium bicarbonate, would react on sodium nitrate to give sodium bicarbonate and ammonium nitrate—

$$NH_3, H_2O, CO_2 + NaNO_3 = NaHCO_3 + NH_4NO_3.$$

In 1875, Gerlach patented processes based on this reaction in England (Wirth, No. 2174) and in Germany having special reference to the purification of gas liquor. Ding. Polyt. Journal, ccxxiii., p. 82. J. Chem. Soc. xxxii., p. 236. Lesage and Co. in 1877, and Chance, in this country, in 1885, also took out patents for the application of the ammonia-soda process to sodium nitrate (this Journal, 1886, p. 325).

If the above reaction could be carried out to completeness, one would obtain only sodium bicarbonate insoluble and ammonium nitrate in solution, and the latter would be pure except from a small quantity of sodium bicarbonate corresponding to the sparing solubility of this salt.

It is now, however, well understood that such reactions are not complete under any conditions hitherto obtained. When the salts are present in molecular proportions, only about two-thirds of the quantities react—one-third remaining unaltered. If either salt exceeds the molecular proportion—the excess remains also and does not materially reduce the proportion of the other salt unaffected.

If one reverses the process, the salts—sodium bicarbonate and ammonium nitrate can react on each other so as to give a mixture of ammonium and sodium nitrates.

Though there is no doubt that variations of conditions such as temperature and pressure do slightly modify the proportions of the salts reacting, yet I have found a tolerable constancy in the results of experiments carried out in the laboratory.

Neglecting the small proportions of ammonium and sodium bicarbonates, the following numbers represent the proportions I have obtained:—

Application of the ammonia soda process to sodium nitrate.

Mixed salts obtained. NH_4NO_3 and $NaNO_3$.		$NaNO_3$.		Per Cent.
59·10	containing	19·75	corresponding to	36·80
58·06	"	19·44	"	33·48
56·70	"	18·76	"	33·08
56·14	"	17·12	"	30·49
54·34	"	19·50	"	35·75

Further treatment of the product from these experiments made practically no difference in the proportions of the salts, and the lowest proportion of sodium nitrate remained as above about 30 per cent.

Other processes depending on the solubility of ammonium nitrate in alcohol and the use of this solvent to extract the salt from the products of double decomposition have been proposed by:—

1889. Roth, Eng. Pat. No. 1885, Ger. Pat. 48,705.

1889. Wahlenberg, Eng. Pat. 12,451 (this Journal, 1889, 293).

1892. Groendahl and Landin, Eng. Pat. No. 1868 (this Journal, 1893, 263).

In these processes the ammonium salts (generally sulphate) are treated with sodium nitrate (Roth also names potassium nitrate) in various ways and the product extracted with alcohol.

Roth and Wahlenberg assume that the reactions between the materials mixed in molecular proportions are complete, while Groendahl and Landin state that a certain amount of sodium nitrate remains with the ammonium nitrate dissolved in the alcohol and a certain amount of ammonium sulphate remains with the sodium sulphate. According to this, the reaction is not complete, and alcohol containing ammonium nitrate can dissolve sodium nitrate in appreciable quantity.

The incompleteness of the reaction of ammonium sulphate with sodium nitrate and the solubility of sodium nitrate in an alcoholic solution of ammonium nitrate I had previously proved conclusively, and I find that Watson Smith, in a paper read before the London Section found that "well dried absolute alcohol" extracted after two or three hours heating, ammonium nitrate containing some sodium nitrate (this Journal, 1893, 10).

Seeing the differences, in what ought to be simple matters of fact, in these patented processes, I have made a detailed examination of the processes described in each of them.

Roth's processes, Eng. Pat. 1885 of 1889 and Ger. Pat. 48,705 are best described in the latter. He heats the salts taken in molecular proportions, say, ammonium sulphate or chloride with sodium or potassium nitrate, in three different ways :—

(*a.*) With thrice the weight of the mixed salts of 95 per cent. alcohol in a suitable vessel furnished with a reversed condenser, during 8 to 10 hours to (77° C.) the boiling point of the alcohol; then he separates the hot alcoholic solution containing the ammonium nitrate from the undissolved alkaline sulphate or chloride.

(*b.*) Ammonium sulphate, chloride, or carbonate are heated in an autoclave with similar proportions of sodium or potassium nitrate and of 95 per cent. alcohol during 4 to 5 hours to a temperature of from 120° to 140° C.

(*c.*) The same mixtures of salts are heated in the dry state to 140° to 160° C. for some time. With ammonium carbonate, closed vessels are used. The resulting mass is then treated with alcohol stronger or weaker, according to the purity of the salt required.

Where the reactions were carried out in the open, ammonia was given off and the materials became more or less acid, so as to act freely on ordinary metals. This does not apply to the ammonium carbonate experiments which were necessarily carried out in closed vessels.

The results show that in each case the ammonium nitrate contained about 10 per cent. of sodium nitrate. The reactions with potassium nitrate were very incomplete, only about 25 per cent. of the salts being converted.

Salts Treated in Molecular Proportions by Processes stated in Roth and Wahlenberg's Patents.	Percentage of Sodium or Potassium Nitrates found in the Ammonium Nitrate Obtained after Completion of the Process.		
	(*a.*) 95 % Alcohol boiled Ten hours (77° C.).	(*b.*) Autoclave Heated to about 140° C. Five Hours.	(*c.*) Dry Salts Heated to 160° C. One Hour.
1. Ammonium sulphate and sodium nitrate...............	$NaNO_3$ % 11·35	$NaNO_3$ % {11·04, 10·69}	$NaNO_3$ % 10·30
2. " chloride " "	$NaNO_3$ % 11·15	..	$NaNO_3$ 11·45
3. " carbonate " "	..	$NaNO_3$ % 17·29	..
4. " sulphate and potassium nitrate............	KNO_3 % 10·20	KNO_3 % 8·44	..
5. " chloride " " .... With 95 % methylic alcohol.	KNO_3 % {4·65, 4·00, 4·90, 4·35}	..	..
6. " sulphate and sodium nitrate.......... . ..	$NaNO_3$ % {31·50, 33·80, 31·30}	..	..

The experiments under 3, 4, and 5 were very incomplete at the end of the time stated, as the greater part of the salts were unaltered.

I also find that boiling alcohol of various strengths dissolves appreciable quantities of sodium nitrate, and that after the alcohol is saturated with ammonium nitrate, the amount of sodium nitrate then dissolved is so great as to render alcohol useless as a means of separating these salts.

Thus 100 c.c. of alcohol (measured at 15° C.) dissolve :—

——	92 per Cent. (Sp.Gr. 0·817).	95 per Cent. (Sp. Gr. 0·809)-	100 per Cent. (Sp. Gr. 0·794).*	Methyl Alcohol (95 per Cent.)
	Grms.	Grms.	Grms.	Grms.
Sodium nitrate......	1·25	0·56	0·27	3·86
Ammonium nitrate .	20·00	16·00	9·10	49·00
Sodium nitrate after ammonium nitrate	3·37	1·81	0·76	9·00

Calculating the sodium nitrate as a percentage on the mixed ammonium and sodium nitrates, we have—

——	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Sodium nitrate	14·46	10·16	7·7	15·6

* NOTE.—This alcohol was carefully rectified by treatment with sodium and distilling.

In some experiments with cold ethylic alcohol, I found that it dissolved from one-fifth to one-sixth of the above quantities, and that in each case sodium nitrate was dissolved along with the ammonium nitrate when both salts were present, as in the reaction of ammonium sulphate on sodium nitrate.

On testing the undissolved residues after removal of the alcoholic solution of ammonium nitrate, quantities of ammonium sulphate or the other ammonium salt used were found, equivalent to the unchanged sodium nitrate.

The reaction is therefore an incomplete one throughout the varied conditions of these experiments, and the same remark applies to the conditions as prescribed by Wahlenberg. Thus, the statements of Groendahl and Landin apply to each of the processes in which alcohol is used by Roth and Wahlenberg.

Groendahl and Landin state that if the ammonium sulphate be in excess of the molecular proportion, a less amount of sodium nitrate is found in the alcoholic solution with the ammonium nitrate.

To eliminate the sodium nitrate, they propose filtering the alcoholic solution through granulated ammonium sulphate. If the sodium nitrate be not eliminated by this treatment, then they recommend filtering through ammonium chloride.

The following experiments have been made on this proposed process.

With an excess of ammonium sulphate in the mixed salts, the following results were obtained.

	Percentage of Sodium Nitrate found in the Ammonium Nitrate.
Molecular proportions of ammonium sulphate and sodium nitrate.	12·07
Same + 25 per cent. ammonium sulphate mixed and heated together before treatment.	10·60
Same + 50 per cent. ammonium sulphate.	10·30
Same + 100 per cent. ammonium sulphate.	10·26

Also the alcoholic solution of the impure ammonium nitrate was filtered through layers of ammonium sulphate.

Percentage of sodium nitrate found in the ammonium nitrate.............................. 8·52 and 9·5

It is therefore exceedingly difficult to purify the ammonium salt by treatment with excess of ammonium sulphate, either at the temperature of the boiling alcohol or at ordinary temperatures.

Incomplete reactions are generally reversible, so that in this case—$(NH_4)_2SO_4 + 2NaNO_3 = 2NH_4NO_3 + Na_2SO_4$—one ought to obtain similar results, whether one starts with the substances on the left or right of the equation. I find that on heating pure ammonium nitrate and sodium sulphate in molecular proportions, and then extracting with alcohol, that the ammonium nitrate contains from 8 to 10 per cent. of sodium nitrate.

These experiments may be taken to prove that under the prescribed conditions ammonium nitrate containing under 1 per cent. of fixed impurity cannot be obtained by treatment with strong or even absolute alcohol of the mass resulting from the reaction of ammonium sulphate, chloride, or carbonate on sodium or potassium nitrates; with weaker alcohol the proportion of impurity is greatly increased.

I hope to give an account of the other processes proposed for this manufacture during the session.

Discussion.

In reply to Mr. B. Watmough, Mr. Fairley stated that he had also experimented with absolute alcohol, but found that it acted similarly to that which he had used in the experiments described.

In reply to Mr. N. Farrant, who asked whether the loss of alcohol in separating sodium nitrate from ammonium nitrate is great, Mr. Fairley said he was unable from laboratory experiments to state exactly how much loss there might be on the large scale, but he thought that if it could be kept under 1 per cent. of the ammonium nitrate produced, the process could be made to pay.

Scottish Section.

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Hon. Secretary and Treasurer:
Thomas Gray, Technical College, 204, George Street, Glasgow.

SESSION 1896–97.

April 27th.—Election of Committee and Officers.

Meeting held at Edinburgh, on Tuesday, Feb. 23rd, 1897.

DR. A. P. AITKEN IN THE CHAIR.

EXPERIMENTS ON THE COMPOSITION OF TURNIPS AND THE EFFECTS OF MANURES THEREON, WITH A METHOD OF ANALYSIS.

BY JAMES HENDRICK, B.SC., F.I.C.

The cultivation of the turnip crop has long occupied a most important position in the agriculture of this country. To this crop it has been customary, under the most various systems of farming, to apply a very great part of all the manure which the farmer uses. One often finds, indeed, that all the manures, both the dung of the farm and the purchased or artificial manures, used in the rotation are applied to this crop, and the other crops get nothing but the residues left in the soil after the turnip break. More artificial manure is probably used in this country for the turnip crop alone than for all the others put together. The question of the manuring of this crop then is of great importance, not only in its purely agricultural aspects, but in its relations to the manure industry.

The profound changes, brought about by the great modern development of our commercial and industrial conditions, which our agriculture is at present undergoing, must seriously affect the position and manuring of the turnip crop. This is the most expensive and troublesome crop in our ordinary agricultural practice, and it may almost be said that it continues to occupy its present position more for its collateral advantages than for its intrinsic worth. With the improvements of knowledge and instruments, the old turnip culture must be gradually superseded by better and more economic methods. Already the manuring practice with regard to this crop, and, as a consequence, with regard to all the other crops of the rotation, is being greatly modified, and even the position and acreage of the crop is undergoing much alteration at the hands of many enterprising farmers.

As might be expected, this crop has always been a favourite one with agricultural investigators. The late Prof. Anderson published numerous papers on the composition of turnips (Trans. Highland and Agric. Soc. of Scotland, 1851 to 1868); Lawes and Gilbert investigated the same subject at Rothamsted (Rothamsted Memoirs); and the late Prof. Voelcker also published papers on this subject (J. Roy. Agric. Soc. of England, 1877). Though there are some foreign investigations on the same subject, foreign investigators have paid far more attention to the closely related subject of the composition of the sugar beet, which has been worked out by German and French workers with a thoroughness which nothing which has yet been done on the turnip can approach. In more recent times we have had investigations on the composition of turnips by Dr. Aitken (Trans. Highland Soc. of Scotland, 1884), and by Mr. Wilson, of Carbeth (Trans. Highland Soc. of Scotland, 1884 and 1886), and simultaneously with my own I find that Dr. Somerville and Mr. Smith, of Newcastle, have been carrying on investigations, which were published in the Journal of this Society, January, 1897. In the last four years, too, an enormous number of experiments have been done. The great majority of these are purely agricultural, and can hardly be said to have any real scientific value. When properly carried out these manurial trials are, no doubt, useful object lessons of considerable educational value, but it is absurd to talk of them, as is too often done, in high sounding phrase as research work comparable with that of foreign research stations. The difficulties surrounding the subject and the labour involved in any large investigations into the composition of root crops are so great that few agricultural chemists in this country have the means of doing any such thorough investigations, as have been carried out abroad, on the composition and improvement of the beet, for instance.

The experiments dealt with in this paper were carried out in the years 1894 and 1895. In 1894 the work was mainly

preliminary. The experience gained in that year convinced me that the very laborious method ordinarily used in root analysis might be somewhat simplified. Consequently, the method described below was devised, and used in the analyses carried out in connection with the 1895 experiments. It had been intended to carry out a further series of experiments in 1896, but circumstances compelled me to abandon the work for the time. Though I hope to resume the investigation in the near future, I have taken this opportunity of giving an account of what has been done so far, in the hope that at any rate the method of analysis may be found better and simpler than that presently in use, and that others may suggest means for further improving and simplifying it.

In the 1894 experiment the roots were grown at Cleghorn, Lanark, on the home farm of Mr. Eliott Lockhart. The purely agricultural part of this experiment formed one of a series of manure tests conducted on different farms scattered over the south-west of Scotland, by the Agricultural Department of the Glasgow and West of Scotland Technical College. (See Reports on Manuring Experiments, &c. conducted in 1894. Glasgow, 1895.) The agricultural part of the work was carried out with the greatest care and thoroughness. The land lies high and exposed, and was in suitably poor manurial condition. This is shown by the fact that on the plots without any manure practically no crop was obtained. The turnips grown were the variety known as Aberdeen yellow bullock. In 1895 the experiment was transferred to Easterboard, Croy, which was much more easily within my reach, and where I could therefore exercise more constant supervision. The agricultural part of the work was carried out by Mr. George Graham, to whom my best thanks are due for his minute care and thoroughness. The land obtained here was more even than at Cleghorn, and in equally suitable condition as to inability to produce any crop without manure. A chemical investigation was made into the composition of this soil especially in regard to its capacity to supply ash constituents to plants. I lay some stress on these points. In experiments on the composition of crops and on the effects of manures on it, where we are dependent on ordinary agricultural land for our experimental ground, it is important that we should choose land in as poor condition as possible, so that the effects on the composition of the crops produced may be the effects of the manures supplied, and not merely of unknown manurial substances already in the soil. Further it is important that we should learn all that present methods of chemical analysis can tell us about the composition of our soil, especially in reference to its ability to supply essential constituents to the plant. It is unfortunate that this is not always attended to, and some laborious experiments are greatly reduced in value by the use of land, which in its unmanured condition is capable of growing a large crop, and by failure to supply information as to the nature of the soil. As in 1894 in 1895 the experiment formed one of a large series of manurial tests carried out on a number of widely scattered farms. Of these a complete report has already been published. (See Reports on Manuring Experiments, &c. conducted in 1895. Glasgow, 1896.) The variety of root grown in 1895 was "Best of all" swede.

In 1894 information was sought chiefly on the following points:—(1.) The composition of the starved roots which managed to survive on the unmanured plot. (2.) The effect of size on the composition of well manured roots. (3.) The difference in composition between roots receiving only a phosphatic manure, and those receiving nitrate of soda in addition to phosphates.

Samples of roots were taken from plots numbered I. and II., which received only phosphatic manures, and from plots IV. and V., which received the same phosphatic manures and nitrate of soda in addition. From each of these plots a few roots, intended to represent the general composition of the plot, were picked. This method is very defective, and was not repeated in 1895. It is almost impossible for the person picking to pick quite without prejudice, and to select roots really representative of a large plot. In these experiments each plot was $\frac{1}{20}$th of an acre in area. The number of roots taken was far too small to furnish a very reliable sample. Two of the best manured plots were IX. and XI. On both of these many of the roots were of large size. Interspersed with these were some which, apparently on account of overcrowding due to imperfect singling, were very small. A few of the largest and a few of the smallest from these plots were chosen. Finally, a few of the best grown bulbs from the unmanured plot were taken. The greater part of the unmanured plot was bare, but here and there a few miserable plants had managed to survive. It was a mistake to pick the best of these, as they had probably some special source, such as accidental horse droppings in the preliminary working of the land, from which they drew a little manurial substance. One should rather have picked the poorest that had just managed to survive.

Table I. gives the details of this experiment.

TABLE I.

Cleghorn Experiment, 1894.

Plot.	I.	II.	IV.	V.	IX. and XI. Large.	IX. and XI. Small.	XII.
Manure and Quantity per Acre.	Superphosphate, 6 Cwt.	Basic Slag, 679 Lb.	Basic Slag, 679 Lb. Nitrate of Soda, 92 Lb.	Superphosphate, 6 Cwt. Nitrate of Soda, 1 Cwt.	IX. Farmyard Manure, 20 Tons. XI. Farmyard Manure, 10 Tons. Superphosphate, 4 Cwt. Nitrate of Soda, 1 Cwt.		Nothing
Crop per acre	13 tons 15 cwt.	14 tons 17 cwt.	17 tons 5 cwt.	12 tons 17 cwt.	..	..	0 ton 15 cwt.
Average weight of sample roots.	2 lb. 3 oz.	3 lb. 7 oz.	2 lb. 10 oz.	2 lb. 0 oz.	5 lb. 6 oz.	0 lb. 11 oz.	1 lb. 4 oz.
Moisture	88·70	89·05	88·06	87·82	89·45	86·46	88·14
Dry matter	11·30	10·95	11·94	12·18	10·55	13·54	11·86
Sugar total (as cane sugar).	5·64	6·30	6·83	6·64	6·14	8·08	6·93
Crude fibre	2·71	3·14	3·05	3·48	3·00	3·35	3·23
Nitrogen, total	0·159	0·118	0·174	0·171	0·097	0·194	0·241
" albuminoid	0·115	0·083	0·110	0·110	0·063	0·114	0·140
Albuminoids	0·720	0·520	0·687	0·687	0·395	0·712	0·874
Insoluble albuminoids	0·338	0·262	0·256	0·309	0·334	..	0·531
Ash	0·966	0·782	0·746	0·913	0·801	1·156	0·874
Percentage of total nitrogen which is non-albuminoid.	27·67	29·63	36·98	35·72	34·78	41·24	41·91

Moisture was determined in the ordinary way, by hanging up the sliced material in a dry atmosphere at a temperature below 50° C. till moderately dry, gradually raising the temperature till the material was dry enough to powder in a mill, and completing the drying with the powdered material at 100° C. Nitrogen and ash determinations were made in

this dried material. Nitrogen was determined by the Kjeldahl method. Crude fibre is the insoluble matter which is left after extracting fresh pulp with water for the sugar determination, less the albuminoids and ash which it contains. Albuminoids were determined in the dried material by Church's phenol method. As repeated experiments, both with roots and with such other plant products as contain much non-albuminoid nitrogen, have convinced me of the unsatisfactory nature of this method; Stutzer's method was used in 1895 and found much more satisfactory.

Little importance can be attached to the crops per acre given in Table I., except as showing that whereas the unmanured plot gave practically no crop, the manured ones were capable of giving a very fair crop. Disease developed in certain parts of the plots late in the season, and certain plots, such as V., had their produce very seriously diminished from this cause.

All the turnips in this experiment gave an amount of dry matter above the average. The smallest turnips give the highest percentage of dry matter, and the largest the lowest percentage. This effect of size on composition is strikingly brought out by the difference between the large and small roots from IX. and XI. Not only do the small roots contain 3 per cent. more dry matter, but there is a higher percentage of each individual constituent determined. The dry matter, too, is, weight for weight, of better composition as a food. This is shown by Table II., which gives the composition of the dry matter.

Table II.

Cleghorn Experiment. Composition of Dry Matter.

Plot	I.	II.	IV.	V.
Sugar (as cane-sugar)	49·11	57·51	57·20	54·53
Crude fibre	23·98	28·67	25·55	28·58
Nitrogen, total	1·41	1·08	1·46	1·40
" albuminoid	1·02	0·76	0·92	0·90
Ash	8·55	7·14	6·25	7·50

	Plots IX. and XI.		Plot XII.
	Large.	Small.	
Sugar (as cane-sugar)	58·20	59·68	58·42
Crude fibre	28·43	24·74	27·23
Nitrogen, total	0·92	1·43	2·03
" albuminoid	0·60	0·84	1·18
Ash	7·59	8·54	7·37

We must expect, then, that this difference in size will be felt all through the table, so that the differences in composition obtained in the other columns are not merely a result of the manuring, but of the average size as well.

This effect of size on composition is further illustrated in Table III., which gives the composition of two samples of "Achilles" turnips taken from analyses made at the same time, but in connection with some experiments on quite a different subject:—

Table III.

—	A.	B.
Average weight	5 lb. 1 oz.	2 lb. 14 oz.
Moisture	92·770	91·340
Dry matter	7·230	8·660
Sugar (as cane-sugar)	3·930	4·620
Total nitrogen	0·192	0·148
Albuminoid nitrogen	0·108	0·076
Ash	0·560	0·640
Percentage of the total nitrogen which is non-albuminoid.	43·750	48·650

In this case the amount of dry matter in both samples is considerably below the average. This may be due partly to the different variety of seed, and partly to differences in soil, climate, and manuring. These roots were liberally manured. It is noticeable that both in Tables I. and III. the smaller roots have the larger percentage of their nitrogen non-albuminoid.

The nitrate of soda used in plots IV. and V. (Table I.) has not had a very decided effect on the composition. It is to be remembered, however, that the dressings used were very moderate. We can, however, distinctly trace to its influence the increased percentage of sugar, the larger amount of nitrogen which is non-albuminoid, and the lower percentage of ash in plots IV. and V. compared with II. and I.

The small and half-starved bulbs in XII. give an interesting analysis. They contain a fair amount of dry matter, and in the dry matter a large amount of sugar. So that sugar formation has not been at all checked. Their most noticeable feature is the amount of nitrogenous matter they contain. Their dry matter is the richest of all both in albuminoid and non-albuminoid nitrogen. In this connection it may be noted that though no analysis of the Cleghorn soil from these plots was made, analyses were made of similar soil from adjacent plots on which other experiments were being conducted. These analyses showed that the soil is not remarkably deficient in nitrogen, though also not rich in this element, but is very deficient in the important ash constituents, phosphoric acid and potash, in a form available to plants. The available ash constituents were determined by Dr. Bernard Dyer's method (J. Chem. Soc., Vol. LXV.), and only 0·0039 per cent. and 0·0054 per cent. respectively of available phosphoric acid were found in two samples.

Before proceeding to deal with the experiment of 1895, the methods of sampling and analysis made use of will be described. It gave me much trouble to devise a method for obtaining a perfectly fair sample of a plot of $\frac{1}{20}$th of an acre, without having to carry off an inconveniently large sample. I found that several different methods have been used by previous investigators, all of which are open to serious objection. At last the following method was adopted, which seemed to me as fair a one as could be devised. Each plot consisted of three drills each about 110 yds. long. Four points were taken at nearly equal distances along the outside drill of plot I. From these points parallel lines were drawn right across the whole series of plots. From each of these lines a yard was measured off at right angles along one drill in each plot. Two of these yards were measured on the central drill, and one on each of the side drills of each plot. Every turnip, large or small, on these four yards was raised and included in the sample, from that plot. It may be remarked that at Easterboard the crop was very even, and quite free from disease and lent itself readily to accurate sampling. There were two unmanured plots, I. and XIII. Of these, XIII. was a complete failure, hardly a single plant surviving till the time of raising. On I., curiously enough, almost as large a number of plants were alive at the end of the season as on any other plot. But so small and miserable were these plants, that they had hardly any resemblance to turnips, and from a little distance the plot looked nearly as bare as XIII. Each sample contained from 15 to 20 roots. On arrival at the laboratory each sample was washed, dried, and weighed. From this and the number of roots in the sample, the average weight of the roots from each plot was calculated. A segment through the centre, from top to bottom, was then taken from each root, in order to provide a sample for analysis. In all the plots except I. the segment was $\frac{1}{8}$th of the root. In the case of plot I. the whole root was taken.

The ordinary method is to cut these segments into thin slices, in order that they may dry readily. They are then hung up and dried at a gentle heat till they are hard enough to grind. The ground material is then completely dried, and the dry powder used for determinations of nitrogen, ash, fibre, &c. During this drying, the material becomes very dark in colour, especially if the temperature be raised too quickly, and a certain amount of decomposition of dry material undoubtedly takes place. This vitiates all the results obtained from this dry matter. For not only has the composition undergone some change, but all the calculations are based on the assumption that this is the

true dry material of the turnip. Sugar determinations are made in a separate sample, which is pulped or rasped down and then has its juice extracted.

This method is not only inaccurate but extremely tedious. Dr. Aitken, who has great experience of root analysis, says: "It might seem from this description, that the sampling and drying of the samples of a turnip crop was a comparatively easy matter, while in reality it is a complex and laborious operation, requiring several months for its accomplishment" (Trans. Highland Soc., Scotland, 1884, p. 360) This method was no doubt necessary when one had to determine nitrogen in dry material by the soda-lime method.

Some preliminary experiments convinced me that when using the Kjeldahl method for nitrogen determinations, all the necessary determinations could be made from the fresh turnip pulp. My samples were therefore pulped by putting them through a small sausage machine, which was found very convenient for the purpose. The pulp was at once thoroughly mixed, and the samples immediately weighed out into previously weighed dishes. If this is done quickly, with two operators working, the whole process can be gone through in a few minutes, and there is thus no danger of error from loss of moisture by evaporation, or from fermentation. I found a good physical balance which turned with three or four mgrms. best for the weighings. As one is weighing a pulp containing about 90 per cent. of moisture, the samples are correspondingly large, and weighings to two places of decimals with the pulp are as accurate as, or more accurate than, weighings to three places with the dry powder.

Five quantities of pulp were weighed out from each sample:—

(1.) For sugar determination, about 100 grms. This was weighed into a small, wide-mouthed bag of close strong linen hung over the mouth of a beaker.

(2.) For moisture and ash, about 30 grms., weighed into a flat dish.

(3.) For fibre, about 30 grms. weighed into a conical flask.

(4.) For total nitrogen, about 10 grms., weighed directly into a Kjeldahl cooking flask.

(5.) For albuminoid nitrogen, about 10 grms., weighed into a beaker.

(1.) *Sugar.*—The mouth of the bag is tied up tightly. The juice is then expressed by pressure. I found a strong potato-masher, such as is ordinarily used for domestic purposes, admirable for this purpose. By soaking the bag in successive small quantities of hot water, and squeezing again, it was found that the sugar could all be extracted by the use of less than a litre of water. The solution so obtained is clarified, inverted, made up to a litre, and the sugar estimated by any suitable process.

(2.) *Moisture.* — The pulp dried much more easily than I expected. If the heat is not raised above 100° little darkening need occur. The pulp dries to a somewhat porous mass, and any decomposition which takes place is very slight compared with what takes place in drying large samples by the old method, when the drying takes days, or even weeks. The dry mass burns easily in a muffle at a low red heat, to a nice light ash.

(3.) Fibre was determined directly in the pulp by the usual acid and alkali method.

(4.) *Total Nitrogen.*—The nitrogen determinations in the wet pulp gave no trouble. With 20 c.c. of the strongest sulphuric acid and a small drop of mercury the pulp always boiled quite clear in less than an hour. It was liable to froth a good deal at first, but a small shaving of paraffin at once checked that.

(5.) The albuminoid nitrogen was at once determined by Stutzer's method with copper hydrate.

Duplicate nitrogen determinations usually agreed within 0·005 per cent. on the pulp. The maximum difference was 0·024 per cent., but that was a quite exceptional case.

Directly after the crop was raised, the soil on the unmanured plot, I, was sampled. It is a medium light loam, containing a little gravel and coarse sand. About 86 per cent. passed a sieve having 30 meshes per linear inch. This fine matter was used for the analysis given below. The "available" phosphoric acid and potash were estimated by Dr. Bernard Dyer's method.

Analysis of Easterboard Soil.

	Per Cent.
*Loss on ignition	11·390
Lime	0·403
Equal to calcium carbonate	0·720
Phosphoric acid	0·128
Potash	0·213
Silica and insoluble silicates	78·990
*Containing nitrogen	0·318
"Available" phosphoric acid	0·0096
" potash	0·0058

This analysis shows, that though moderately supplied with phosphoric acid and potash in a form which can be extracted by strong hydrochloric acid, the amount of these constituents which is in so readily soluble a form as to be of any use to plants is very small. It also shows the soil to be fairly well supplied with organic matter, and its contained nitrogen.

The Easterboard experiment consisted of 13 plots, each of $\frac{1}{20}$th of an acre. Samples were analysed from eight of these plots to illustrate the following points:—(1.) The composition of starved plants from an unmanured plot. (2.) The effect of a moderate dressing of nitrate of soda on the composition of roots receiving only a phosphatic manure. If the soil itself had been somewhat poorer in nitrogen it might, perhaps, have illustrated this point better. (3.) The effects of a rich all-round dressing of artificial manures alone, of a rich dressing of farmyard manure alone, and of a rich dressing of a mixture of farmyard manure and artificial manures on the composition. Table IV. gives the manures applied, the crop per acre for all the plots, and the number of roots per acre on some of the plots.

In order to carry out my scheme, roots were analysed from plots I., II., III., V., VI., IX., X., and XII. It had been intended to count the number of bulbs on all these plots, but we were prevented from completing this plan.

In Table V. the details of the 1895 analyses are given.

It will be noticed that the average weights, given in Table V., of the roots analysed agrees fairly well with the average weights of the total roots on the plots given in Table IV. We might expect the weights in Table IV. to be slightly higher than those in Table V. The weights in Table IV. are calculated from weighings in which the crops weighed were in an agriculturally clean condition. The weights in Table V. are derived from weighings made in the laboratory, after the samples had been thoroughly washed. The results, however, are sufficient to show that my samples represented the average sizes of the roots on the different plots fairly closely.

In this experiment, as in 1894, the roots all show a high amount of dry matter. Swedish turnips, however, have on the average a higher percentage of dry matter than other varieties. König ("Chemie der Menschlichen Nahrungs und Genussmittel," Band I.) gives, as the mean of a very large number of determinations made by a great many chemists of repute in several different countries, 87·80 per cent. of moisture and 12·20 per cent. of dry matter in Swedes.

The analysis of the starved roots from plot I. is, as might be anticipated, extraordinary. They contain a percentage of dry matter which is most exceptional. The late Prof. Voelcker records one case of Swedes giving even a slightly higher percentage, 17·78, of dry matter than this. It is evident that these roots did not remain starved for want of nitrogen. They contain a quite extraordinary proportion of nitrogenous matter, an unusually high percentage of which is non-albuminoid. This supports the view that in the case of an ordinary soil containing a moderate supply of nitrogenous matters, the turnip crop is perfectly capable of supplying itself with nitrogen without the aid of nitrogenous manure. This is supported by the remaining columns of the table. Thus it will be found that II. and III., which got no nitrogenous manure, are not deficient in that element as compared with the samples which got dressings of nitrogenous manure, some light and some

Table IV.

Easterboard Experiment, 1895 Manures and Crop per Acre.

Plot.	Kind of Manure.	Quantity per Acre.	Roots per Acre.	Tops per Acre.	Number of Bulbs per Acre.	Average Weight of Bulbs.
			Tons cwt. qrs.	Tons cwt. qrs.		Oz.
I.	No manure	..	1 8 1	0 2 3	20,500	2¼
II.	Superphosphate	6 cwt.	16 10 3	1 10 0	21,200	24
III.	Basic slag	688 lb.	14 5 1	1 4 3	..	..
IV.	Bone meal	375 lb.	13 3 3	1 3 2	..	..
V.	Basic slag Nitrate of soda	688 lb. 93¾ lb.	14 19 3	1 10 0	..	..
VI.	Superphosphate Nitrate of soda	6 cwt. 1 cwt.	17 1 0	1 8 0	..	..
VII.	Superphosphate Sulphate of ammonia	6 cwt. 85 lb.	17 16 2	1 8 1	..	..
VIII.	Superphosphate Nitrate of soda Sulphate of potash	6 cwt. 1 cwt. 1 cwt.	19 0 0	1 9 3	..	..
IX.	Superphosphate Bone meal Nitrate of soda Sulphate of potash	6 cwt. 4 cwt. 1 cwt. 1 cwt.	19 15 3	1 10 0	23,720	30
X.	Farmyard manure	20 tons	23 2 0	2 6 3	21,120	39
XI.	Farmyard manure Superphosphate	10 tons 4 cwt.	22 5 0	1 16 2	24,760	32
XII.	Farmyard manure Superphosphate Nitrate of soda	10 tons 4 cwt. 1 cwt.	22 15 3	1 18 2	..	..
XIII.	No manure	..	Complete failure	..	..	..

Table V.

Easterboard Experiment, 1895.

	Plot I.	II.	III.	V.	VI.	IX.	X.	XII.
Average weight of roots	1¾ oz.	20 oz.	14 oz.	14 oz.	23 oz.	32 oz.	39 oz.	29 oz.
Composition of the fresh roots:—								
Moisture	82·38	88·52	86·53	86·17	87·46	87·82	88·46	87·13
Dry matter	17·62	11·48	13·47	13·83	12·54	12·18	11·54	12·87
Sugar (as cane-sugar)	7·33	6·12	7·11	7·75	7·38	7·07	6·37	7·53
Fibre	2·34	1·18	1·45	1·54	1·24	1·30	1·23	1·34
Nitrogenous matter (N × 6·25)	3·71	1·00	1·19	1·13	1·18	1·06	1·21	1·16
Ash	1·12	0·57	0·67	0·62	0·65	0·62	0·64	0·65
Nitrogen total	0·593	0·160	0·190	0·181	0·188	0·169	0·193	0·185
„ albuminoid	0·243	0·120	0·145	0·126	0·103	0·089	0·087	0·085
Albuminoids	1·52	0·75	0·91	0·79	0·64	0·56	0·54	0·53
Percentage of total nitrogen which is non-albuminoid.	59·01	25·09	23·69	30·39	45·21	47·34	54·92	54·05
Composition of the dry matter:—								
Sugar (as cane-sugar)	41·60	53·33	52·79	56·03	58·86	58·06	55·20	58·50
Fibre	13·28	10·28	10·76	11·14	9·89	10·67	10·66	10·41
Ash	6·36	4·97	4·97	4·48	5·18	5·09	5·47	5·05
Nitrogen total	3·37	1·39	1·41	1·31	1·50	1·39	1·67	1·44
„ albuminoid	1·38	1·01	1·08	0·91	0·82	0·73	0·75	0·66
Pounds per acre:—								
Dry matter in crop	557	4,246	4,304	4,643	4,789	5,398	5,972	6,560
Nitrogen in crop	18·8	59·3	60·7	60·8	71·8	74·9	99·9	94·4
„ supplied in manure	..	..	..	14·7	17·6	17·6	(240)	17·6 (+120)

heavy. Indeed we find that whether we take the composition of the dry matter or of the raw material, II. and III. are markedly high in albuminoid nitrogen, while those plots which received heavy nitrogenous manuring are the lowest in albuminoid nitrogen.

Plot I. also gives a very much higher percentage of ash than any of the other plots. It had been determined to estimate phosphates and potash in each sample of ash, but, owing to an unfortunate accident some of my ash samples, including I., were destroyed before I had time to deal with them. The estimation of phosphate in the ash would have been perhaps the most interesting in the whole investigation. I turned therefore with much interest to the paper published in the January number of this Journal, in which phosphate determinations are given. Unfortunately, however, the conditions of the experiments there described, in which the soils experimented upon were in such high condition that unmanured plots gave very fair crops, were so different from mine that they cannot throw any light on the important question of the composition of the ash of plants starved for want of a sufficient supply of certain essential ash constituents. Plots II., III., V., and VI. show that while the addition of a small dressing of nitrate of soda to a phosphatic manure does not materially increase the bulk of the crop on such a soil as this, it does distinctly improve its composition in certain points. Comparing V. with III., and VI. with II., the percentages both of dry matter and of sugar are increased. On the other hand, as in 1894, the percentage of nitrogen which is non-albuminoid is also increased.

Turning to IX., X., and XII. we find that all the roots are bigger, and give a somewhat lower percentage of dry matter. In no case, however, is the average root, what can be considered very large. Plot X., which received the heavy dressing of dung, gives roots of, in nearly every respect, the poorest composition. This plot gave the heaviest crop per acre, but XII. really gives a larger crop per acre, for it gives more dry matter, and dry matter of quite as good, if not a better composition. The amount of nitrogen given per acre in the manure to plots X. and XII. is calculated on the assumption that a ton of dung contains 12 lb. of nitrogen. On this assumption, which is a fairly safe one, these plots, and especially XII., get heavy dressings of that most expensive manure constituent nitrogen, and give little return for it.

While it would be unwise to draw any hasty conclusions especially in matters of detail, a consideration of these experiments, in the light of the many others on the same subject which have preceded them, will indicate certain lines along which improvement must be sought if this most costly and troublesome crop is to remain an important feature of our agriculture. As in so many other parts of our agricultural industries we must look more to quality and less to mere quantity. Quality varies greatly with a variety of conditions, and by cultivation and rigid selection could be much improved. The turnip crop can and should have its quality improved by similar means to those by the use of which continental workers have so greatly raised the quality and value of the beet. Next we must try to find the means of greater economy in the use of manures, and especially of the most expensive manure constituent, nitrogen. It is now generally accepted that it is not economical to lavish all our manures on the turnip crop. A small quantity of nitrate of soda is useful in pushing the plant through a risky period in its life history, and improves the quality. Large quantities of nitrogen, such as are applied in heavy dressings of farmyard manure, are expensive and wasteful. A small quantity of dung along with a moderate quantity of a phosphatic manure, and, perhaps, a small quantity of nitrate of soda is more economical, and, generally speaking, gives as great a quantity of food of better quality.

Discussion.

The Chairman presumed that the intention of the experiments was to provide agriculturists with information regarding the efficacy of manures, such as might be translated into general practice. In that case he regarded it as of prime importance that the effects of the manuring should be determined only upon such plants as a farmer would desire to grow, and under ordinary conditions of farming. The composition of turnips grown upon an exceedingly poor soil, without any manure, and weighing, say, $1\frac{1}{2}$ ounces each on an average, did not, in his opinion, convey information of any value; for the plants, under such severe conditions, never attained to the condition of a normal turnip. That was, of course, an extreme case; but also, in some of the manured plots, the kind of turnips grown was so much inferior to what a farmer would wish to grow that their composition conveyed little information of practical value to the farmer. If it were desired to obtain information of scientific rather than of practical interest, the end could be attained in the open field only after prolonged investigation, whereby the many disturbing influences to which crops exposed to the weather were liable had been, as far as possible, eliminated; and the samples taken of such crops must be very large in order to be reliable.

The area under experiment in this investigation was $\frac{1}{40}$ acre for each plot, which might seem a small one, but it was double what was commonly chosen for turnip experiments, and was ample if land could be got of uniform quality. That was a difficult thing to get, and Mr. Hendrick was fortunate in getting it in this case. The Chairman could not say that he approved of the kind of plots selected, *viz.*, a three-drill plot of great length, of which only a small cross-section was taken to provide turnips representative of the bulk. When turnips were well grown they sent out their roots laterally and invaded the neighbouring drills, so that in such field experiments as he had conducted, he had always rejected the outermost drills of the plots, knowing that they had, in most cases, borrowed from the neighbouring drills. Such borrowing was especially apt to influence prejudicially the results of the experiment if the plots had not been separated by intervening unmanured drills.

Mr. Hendrick had endeavoured to overcome what he termed the personal equation in sampling, by selecting a straight section running across his three-drill plots, and using as his sample the total number of turnips included in the section. The Chairman was of opinion that that method was liable to greater inaccuracy than could be introduced by the ordinary method of selecting a sufficient number of turnips from each plot, such as was deemed fairly representative of the average growth. Large turnips that had accidentally received an unintended amount of manure, and small misthriven plants that might have received some injury, were in that way rejected and not allowed to disturb the sample, which should be made up of such turnips only as a farmer would regard as normal.

So far as ascertaining the quantity of the crop per acre was concerned, the only safe way was to weigh all the crop, or at least the half of the crop contained in each plot. On the other hand, as regards the quality of the crop, he believed it was safer to trust to a sufficient number of carefully selected average specimens.

As regards the method of analysis, anything which would simplify and shorten the work would be welcome. The labour and time absorbed in drying the samples constituted a chief objection to the usual method, and if Mr. Hendrick could show by comparative experiments that the pulping method was equally reliable it would be a valuable result; but in order to show that it was a more accurate method, it would be necessary to prove that in drying sliced samples in the ordinary way there was a loss by decomposition.

In his own experience the Chairman had found that he could not accurately determine the amount of moisture in pulped samples on account of the difficulty of getting the proper proportion of sap and solids, but his samples were taken from the pulp made with the farm pulping machine, and not with the little sausage machine which Mr. Hendrick had found so suitable. He quite agreed with Mr. Hendrick in preferring the Stutzer method of separating albuminoids to the carbolic acid method, and the Kjeldahl method of estimating total nitrogen to the soda lime one, as they were easier and more convenient, though not more accurate.

There was a point which he might take note of, in which he differed from Mr. Hendrick, *viz.*, in regard to Eastbog experiments, where from the presence of a large percentage of nitrogen in the very small turnips grown, he inferred that there must have been plenty of nitrogen in the soil. He did not think they were entitled from the growth of such plants to draw that inference. It would even be somewhat risky to draw such an inference from plants that had grown to normal proportions. He might not, however, have quite apprehended what Mr. Hendrick had said on that subject. He would be glad to hear Mr. Hendrick on the few points he had referred to, and had pleasure in expressing his gratification at the kind of research which the author had described to them. Such researches were very much wanted, and it would be advantageous to have them carried on at the various agricultural schools in the country.

Mr. Hendrick, in reply, said that no doubt the Chairman would find some of his criticisms fully answered when he had time to look into the paper more carefully. No claim was made that this described work was exhaustive; in fact, it was distinctly pointed out in the paper that this was but a slight contribution to a large subject. One was accustomed to hear the so called practical man questioning the object and use of scientific work, but it was a little surprising to hear the Chairman taking up such a position.

With regard to his method of sampling, the speaker said that one of the best points about it was that it brought out clearly what false ideas as to average size those who sampled by mere individual selection were apt to entertain. The Chairman, who had great experience on this subject, considered that the bulbs chosen were very small; that they were not such as a farmer would wish to grow. If he examined the tables he would find that the crops per acre were not, as he suggested, ridiculously small, but were good agricultural crops; nor were the numbers of bulbs per acre abnormal. The average size per acre found from these figures agreed fairly with the average size of the samples. Further, it was found that, on attempting to sample by the Chairman's own method, the samples taken were much over the average size, whether the samples were chosen by the speaker himself or by a practical farmer. The eye was attracted by the larger roots, which were picked out, and the large number of smaller ones around them were hardly noticed. It might be remarked that practical men considered the crops on plots IX. to XII. average well-grown crops for the district.

Mr. Hendrick said that he fully appreciated the difficulty as to the shape of his plots, but he had to take what he could get—not what he would like. Unfortunately he was

not in the happy position of having an experiment station and adequate means at his disposal, but had to trust to what he could persuade an intelligent farmer to give him. It was intended to take the sample for each plot entirely from the centre drill of that plot, but when it was seen that the end drills of the manured plots, both of which lay next to heavily manured plots, still gave no crop, though in one case all the plants were alive, it was decided to sample in the method already indicated. The manures were, of course, applied with every possible precaution to prevent overlapping. With regard to comparative experiments, he (Mr. Hendrick) did not think it necessary to burden his paper with the details of these. He had made sufficient to convince himself of the superiority and greater ease of working of his method. He might say, however, that he did not find a great deal of difference between the amount of dry matter found by the two methods. The moisture lost on the knife, hands, or in cutting up the turnips might be expected to counterbalance the loss due to decomposition. Two errors here neutralised one another. But in the case of the old method the subsequent determinations of nitrogen, ash, and fibre depended on this questionable determination of dry matter, while in his method they were absolute. He (Mr. Hendrick) could not conceive on what grounds Dr. Aitken described the Stutzer method as an easier, though not more accurate, method than Church's phenol method. He had given up the phenol method, though he found it rather easier to work, because he had become convinced that it was less accurate.

New York Section.

Meeting held at the College of Pharmacy on Friday, February 19th, 1897.

PROFESSOR CHAS. F. CHANDLER IN THE CHAIR.

ALUMINIUM BINARY COMPOUNDS AND THEIR REDUCTION AS CONSIDERED FROM CHEMICAL AND ELECTRICAL STANDPOINTS, INCLUDING A SYNOPSIS OF THE METALLURGY OF SODIUM.

BY HENRY S. BLACKMORE.

Under the head of aluminium we will consider at the present time only those aluminium binary compounds which are or have been of commercial importance in relation to reduction to metallic state, namely, the oxides, fluorides, chlorides, and sulphides; considering first those found in nature (oxides and fluorides) and, secondly, those only produced artificially by chemical reaction (chlorides and sulphides), it being our object to give a synopsis of the most important processes hitherto devised for producing and reducing these compounds, citing, so far as known, the difficulties met with and means devised for obviating them.

We will consider first the oxides, they being the practical basis for artificial or chemical production of all the other compounds. As the characteristics of the minerals containing aluminium oxide, such as corundum, clays, bauxite, &c., are all well known to chemists, we will only refer to the processes devised for commercial production or separation of the aluminium oxide from the minerals containing aluminium.

The production or separation of aluminium oxide may be classed under two heads—that of combining the aluminium oxide with alkali, to produce aluminates and final decomposition of the same while in solution precipitating aluminium hydroxide, and that of combining the aluminium oxide with acid to produce salts to be finally dissociated, such as sulphates. The various processes for producing aluminium sulphate from clay, or aluminium silicate by action of sulphuric acid, and by burning with iron pyrites, &c., and separation by lixiviation, filtration, or decantation, and final precipitation of iron by ferrocyanides, aluminium hydroxides, &c., are well known, so we will merely cite under this head the process for producing oxide from said sulphate. When aluminium sulphate is heated to bright redness it is dissociated spontaneously into aluminium oxide and sulphuric anhydride, the latter of which volatilises; it is found, however, that the dissociation is not perfect, insomuch as much of the sulphuric anhydride is simultaneously dissociated into free oxygen and sulphur dioxide; and here is where the fatal point is reached relative to the economical production in this way; for, whereas if the sulphuric anhydride eliminated were not itself dissociated it might be absorbed by water to reproduce sulphuric acid for further use. The said dissociation into free oxygen and sulphur dioxide precludes this to a large degree, and in a practical way not more than 25 to 30 per cent. of the theoretical amount of sulphuric acid can be thus obtained, the balance of the sulphur being lost in gaseous by-products, which can only be recovered by the expensive process of oxidation.

Some years ago I turned my attention to this process with reference to devising a means of preventing the dissociation of sulphuric anhydride into free oxygen and sulphur dioxide, and finally succeeded in eliminating the sulphuric radical from the sulphate and recovering the same with very little loss. This I accomplished by heating the sulphate in retorts to a point below dissociation—that is about 175° F.; then I introduced superheated steam in a highly heated state. The sulphuric radical was immediately absorbed by the steam, producing sulphuric acid which distilled leaving behind aluminium oxide, while the occluded heat conveyed into the retort by the steam was utilised to further the reaction directly within the porous mass in a perfect manner. Whereas without the steam at higher temperature, the sulphate would be dissociated at points first in juxtaposition to the retort, producing a layer of aluminium oxide, which, being a non-conductor of heat, would necessitate a higher temperature for further action, and thus produce gaseous by-products of comparatively little value.

It might be interesting to state how I obtained this sulphate. Starting with bauxite, which is a double oxide of iron and aluminium contaminated more or less with silica, I placed it in a reverberatory furnace and heated it to low redness, in order to drive out all moisture and make it porous. I then closed the furnace and passed superheated steam through the coal with which the furnace was heated, thus producing water-gas, carbonic oxide, and hydrogen, which was then passed through the porous mass by means of properly regulated flues and dampers. The bauxite was thereby transformed so that the iron present was practically reduced to a condition insoluble in sulphuric acid. When the mass had been treated with gas for about an hour it was removed and cooled in closed containers, to prevent further oxidation. It is then mixed with sulphuric acid, lixiviated, filtered to remove iron oxide and silica, boiled down, and cast into blocks for recovering the sulphuric acid by action of superheated steam, as before described.

The Webster process employed by the Aluminium Company, Limited, of Oldbury, England, consists in heating potash alum with pitch, and treating the product with hydrochloric acid so long as sulph-hydric acid gas is evolved. The mass is then broken up, placed in retorts, and subjected in a heated state to the action of steam and air, which burns out the excess of carbon: and there remains a mixture of alumina and potassium sulphate. The latter is separated by lixiviation and filtration, and the evaporated solution is used over again, the aluminium oxide retained by the filter being removed and dried.

The alkali aluminates are produced generally from bauxite by heating with alkali salt, such as sodium carbonate or sulphide, &c., or by heating cryolite (aluminium-sodium fluoride) with calcium carbonate. In treatment of bauxite the sodium or alkali aluminate produced is separated by lixiviation and filtration, leaving behind iron oxide. In this way, however, it is likely to be contaminated with alkali silicate, and thus the alumina finally precipitated from solution by action of carbonic anhydride contains silica.

Tilghman (British Patent 1847) introduces steam, carrying vapour of sodium chloride, into a mass of heated bauxite, thereby eliminating hydrochloric acid fumes and producing sodium aluminate. He also introduces steam, carrying sodium chloride, into heated aluminium sulphate, producing aluminate. Dr. K. J. Bayer in 1889 devised a process for precipitating aluminium oxide in the form of hydroxide from a solution of sodium aluminate by adding aluminium hydroxide to the solution and cooling, which precipitates and carries down a large part of the aluminium oxide previously combined in the aluminate in pulverent form. Sodium aluminate has also been and is now produced from cryolite (aluminium-sodium fluoride) by heating with calcium carbonate, lixiviating, and filtering, producing at the same time calcium fluoride. Another process has also been devised which consists in mixing aluminium sulphate with alkali sulphide and heating, which produces an alkali aluminate, eliminating sulphur dioxide, which is oxidised to produce sulphuric acid for further production of sulphate.

Having now referred to the most important methods devised for producing aluminium oxide, let us look for a moment at the processes devised for its reduction to the metallic state. Mr. H. Warren claims to have reduced aluminium oxide by action of hydrogen gas at high temperatures, concentrating all the heat in one spot of the tube in which the reaction took place.

I have made many experiments on reducing aluminium oxide by means of gaseous agents, thinking that perhaps a temperature might be found at which hydrogen would reduce aluminium oxide, on the same principle of variation of affinity of oxygen for iron at different temperatures, decomposing steam at a white heat, producing iron oxide and free hydrogen, and again giving up oxygen at a low red heat in the presence of hydrogen, producing so-called "iron by hydrogen." I did not, however, find any reaction whatsoever with dry aluminium oxide. I then tried dissolving the oxide in a molten bath of cryolite and passing hydrogen gas through it, thinking that by separating the molecules in solution they would be in better condition for reduction. Under ordinary atmospheric pressure I obtained no results, but upon subjecting to super-atmospheric pressure I obtained a reaction, and steam escaped from the safety valve. Upon finally opening the container, however, I found no aluminium, but instead a lower fluoride or sub-fluoride of aluminium, which only required about one-half the amount of sodium to reduce that the normal fluoride does. Upon trying the oxide under similar conditions with other fluxes I obtained no results, which led me to believe that the affinity of fluorine in the fluoride assisted to a large degree in the first case. Upon treatment with acetylene instead of hydrogen with other fluxes, I obtained aluminium in a fine state of division, and also a sub-oxide of aluminium of blue colour, which decomposed water with elimination of hydrogen gas and production of white aluminium hydroxide, the blue colour entirely disappearing. This is evidently the base of ultramarine, for if this blue oxide is melted with sodium silicate and sulphur, a permanent compound is produced, not decomposable by water. Dr. Joseph W. Richards has informed me that he has tried to reduce aluminium oxide dissolved in molten cryolite bath by means of natural gas, consisting principally of marsh gas, and also with other hydrocarbons under ordinary atmospheric pressure without result. There have been processes devised by which aluminium oxide is mixed with an oxide of another metal, such as iron, and reduced by carbon at high temperature, the object being to reduce the aluminium oxide by nascent iron or other metal-producing alloys of aluminium, the carbon removing and reducing the metallic oxide produced by said reduction; but they have proved to be of no practical commercial value, as they could not be depended on. This is about all the data of importance we have relative to the chemical reduction of the oxide, and we will now look at it from an electrical point of view.

We will refer first to the Cowles electric process, which consists principally in using a furnace, wherein high temperatures are obtained by electrical resistance. At the temperature attained by this furnace, aluminium oxide can be readily reduced by carbon, but has to be retained by some other metal, such as copper, as the aluminium volatilises at the intense temperature produced if no other metal more stable under those conditions be not present; only alloys are, therefore, practically produced in this way.

Next is the Heroult process, which consists in fusing aluminium oxide, on top of copper or other metal, by electrical heat and decomposing by electrolysis, the aluminium being absorbed by the copper which is the cathode, while the oxygen combines with the carbon anode suspended in the molten oxide. For pure aluminium the cathode consists of molten aluminium.

Then we have the Hall process, in which the oxide is dissolved in a molten bath of cryolite or other fluorides and thus electrolysed, the aluminium accumulating at the bottom while the anode is consumed by the oxygen of the oxide. This is the process employed by the Pittsburg Reduction Co. at Niagara Falls, and Mr. Hall and his associates deserve great praise for the perfection to which this process has attained. It is stated in Prof. Richards' work on aluminium that Mr. Hunt has stated that with improved facilities at Niagara they will be able to reduce the cost of production by this process to 21 cts. per pound—the lowest limit.

Finally, we have the process of Gooch and Waldo, which consists in introducing into a molten bath compounds of aluminium, which are transformed by heat or steam into oxides in the bath during electrolytic action.

Synopsis of Processes for Producing Sodium.

In 1807 Sir Humphry Davy discovered this metal by electrolysis of caustic soda ($NaOH$); shortly after this Guy Lussac and Thenard decomposed sodium carbonate with iron turnings at white heat. In 1808 Curaudau is said to have produced sodium by substituting animal charcoal for iron turnings; next Brunner replaced animal charcoal by black flux, produced by calcining argols, placing the mixture in an iron mercury flask connected with a condenser. On heating the mixture to whiteness vapour of sodium with gaseous by-products were eliminated and sodium condensed; only 3 per cent. of the mixture was obtained as sodium. It was found that sodium carbonate fused during process of reduction, and when carbon was employed as reducing agent it floated upon the surface, so that reduction only took place at point of junction. To obviate this Deville mixed it with chalk to make it less fusible. Castner overcame this difficulty by weighting carbon, so as to keep the carbon below the surface of the molten bath; this was done by coating particles of iron with carbon by heating with tar or pitch.

The great trouble experienced in processes where carbon is employed as a reducing agent with sodium hydroxide is that the carbon oxides, eliminated during reduction, combine with a large part of the caustic soda, producing sodium carbonate, which has to be reconverted into caustic, or the apparatus will be quickly destroyed by the high temperature required to reduce the carbonate.

In 1886 I invented a process for reducing sodium from caustic soda by action of nascent iron, producing the caustic soda from carbonate directly in the mixture. Later I reduced molten caustic soda by means of iron by hydrogen, or a finely-divided iron heated to redness, and gradually introduced it into the molten soda, at which time a reaction took place, sodium vapour and hydrogen being eliminated and iron oxide produced, which settled to the bottom, and was withdrawn from time to time. The soda dissolved out and evaporated, and iron oxide dried, the soda being used over and the iron reduced by hydrogen again, a portion of the hydrogen being recovered after condensing the sodium for this purpose, the process being practically continuous without formation of carbonate or other salt of soda, which was detrimental to the process, and admitting of almost

entire reduction of caustic soda. I also introduced iron in a finely-divided molten state with good results.

Dr. Curt Netto heats carbon and introduces caustic soda in molten state, so that it drops on the heated carbon, sodium and gases passing into condensers, and carbonate produced being automatically withdrawn from lower part of retort and reconverted.

The Cunningham process consisted in introducing into molten caustic soda at or near the bottom of the reducing retort finely-divided carbon in a heated state, which passed up through the mass, decomposing it.

The principal electrolytic processes devised are those of Ludwig Grabau and H. Y. Castner. Grabau electrolyses molten sodium chloride, chlorine being eliminated at the anode, and sodium at the cathode, which is allowed to distill. Castner electrolyses molten caustic soda at a low temperature, skimming the sodium off the surface of the bath. It is stated that sodium can be produced by this process for 15 cents per pound. Prof. Rogers, of Milwaukee, suggests producing alloys of sodium and lead by using molten lead for cathode and finally distilling off the sodium.

Great difficulty is experienced in employment of alkali chlorides on account of their extreme volatility at the temperature employed, as they distill off with the sodium and contaminate it. The disintegration of electrodes and corrosive action on apparatus is also a bad feature, as well as the cost of electricity required to perform reduction. In the employment of caustic alkali, as in Castner's process, considerable alkali carbonate must necessarily be produced by action of the carbon anode.

Aluminium Fluoride.

Aluminium fluoride is found in nature combined with sodium in the form of cryolite. This compound has been produced artificially by mixing solutions of aluminium sulphate and sodium fluoride, when artificial cryolite precipitates in gellatinous form, which is separated by filtration or decantation and dried. When this cryolite is reduced by metallic sodium, producing aluminium, the sodium fluoride is dissolved and used over to produce more cryolite. Aluminium sulphate has also been introduced directly into molten alkali fluoride, the alkali sulphate produced being dissolved out. This process for producing aluminium fluoride was invented by Ludwig Grabau, of Hanover, whose process for reduction will be cited hereafter.

Aluminium Chloride.

Aluminium chloride is generally produced by mixing aluminium oxide with fine carbon, working up into a dough with oil, pressing into porous bricks, which are then dried and placed in retorts. When heated to redness, dry chlorine gas is introduced and passed through the mass. This, in the presence of carbon which absorbs the oxygen from the aluminium oxide, combines with the aluminium, producing aluminium chloride which sublimes. Sodium chloride is often mixed with the carbon and alumina, in order to produce a double chloride, which is more permanent in air and much more easily handled. Aluminium chloride can be produced by passing carbon bisulphide and hydrochloric acid gas alternately over heated aluminium oxide. Camille A. Faure, of storage battery fame, invented a process in 1888, which consists in heating aluminium oxide to redness and passing vapour of petroleum carrying chlorine over and through it.

Reduction of Chlorides and Fluorides.

We will consider the reduction of these two compounds under one head, as the processes of reduction are analogous to a large extent. There have been processes devised whereby aluminium chloride was reduced by heating it, and passing metallic sodium vapour over it, and, *vice versâ*, heating sodium and passing vapour of aluminium chloride over it (Deville), also by bringing the two vapours together in a heated chamber precipitating aluminium in small globules (Frismuth), also by melting double fluoride of aluminium and sodium (cryolite), and forcing an ingot of solid metallic sodium down into it (Netto), and heating practically infusible aluminium fluoride *per se* and pouring upon it molten sodium (Grabau). But in all these processes great trouble is experienced by reason of the violent reaction which takes place while reducing the aluminium and distributes it throughout the flux in such a manner that it is almost impossible to collect it into a mass, on account of its lightness. In view of this fact, in 1889 I devised a process to overcome this violent reaction or agitation of the molten bath. I employed a carbon-lined retort in a vertical position, at the bottom of which was molten aluminium, and floating upon it the flux, carrying aluminium fluoride or chloride to be reduced. Below the surface of the molten aluminium at the bottom of the retort, I introduced molten sodium, by gravity from a reservoir placed at a high point, by means of a pipe connection. Upon the gradual introduction of sodium in contact with the aluminium it alloyed with it, passing up to the surface of the free aluminium in juxtaposition to the flux carrying the compound to be reduced, reducing the aluminium therefrom in contact with the surface of the molten mass of aluminium, with which it immediately combined without agitation, the reduced metal as it were gradually growing up from the bottom.

The processes for electrolytic reduction have, with a few exceptions, been based upon the dissociation of molten aluminium chlorides or fluorides in the presence of an anode consisting of bauxite or aluminium oxide mixed with carbon, the object being that as the chlorine or fluorine is liberated at the anode during action, aluminium chloride or fluoride will be regenerated by action of said elements upon the alumina and carbon anode, the product being absorbed by the bath to be afterwards decomposed by electrolysis depositing aluminium at the cathode, the difference between the processes being merely a little change in apparatus employed to bring about the result. Grabau suggests the presence of some alkali salt in the bath, to absorb the chlorine or fluorine liberated during electrolysis, and Faure suggests electrolysing aluminium chloride and utilising the chlorine liberated to produce bleaching powder.

Aluminium Sulphide.

Aluminium sulphide I consider as the most important compound of aluminium relative to economical reduction, as the affinity of sulphur for aluminium is comparatively slight, while the electro-negative constituent sulphur is comparatively inexpensive.

Petitjean (British Patent of 1857) describes a process for producing aluminium sulphide, which consists in heating aluminium oxide to redness and passing vapour of carbon bisulphide over it, whereby the carbon seized the oxygen of the alumina, and the sulphur combined with aluminium, producing aluminium sulphide. By this process only about one-third of the oxide was converted into sulphide, the reason being that aluminium sulphide, which is partially fusible, coated the particles of infusible oxide and prevented further action. Aluminium sulphide has been successfully produced as aforesaid by Fremy, Reichel, Comenge, Prof. Joseph W. Richards, of Lehigh University, and many times by myself.

Aluminium sulphide produced in this manner was, however, of no practical commercial value, for the reason that the waste of aluminium oxide was enormous, being about 66 per cent., while the mass was practically infusible at ordinary temperatures, and not in condition for practical reduction. When the sulphide thus produced was subjected to action of reducing agents, the aluminium was reduced in small globules throughout the infusible mass in such a manner that it was impossible to combine it into a button.

Aluminium oxide has been heated with sulphur, a mixture of sulphur and carbon, and sulphydric acid gas without producing aluminium sulphide. The reason for the reaction of carbon bisulphide, and not the elements carbon and sulphur *per se*, is that in the formation of carbon bisulphide a great amount of heat is absorbed, which is given out upon reaction with aluminium oxide, thus bringing the reaction within bounds of reasonable temperatures.

Comenge, of Paris, suggests preparing aluminium sulphide by placing aluminium oxide at one end of a clay

retort and charcoal at the other, then heating and passing in sulphur vapour through the charcoal, the product being conveyed over the aluminium oxide at the opposite end.

The process of Petitjean has also been assisted by mixing the aluminium oxide with carbon, as for producing chloride, and then passing carbon bisulphide vapour over it. Free sulphur vapour will not convert this mixture into aluminium sulphide, as chlorine does into chloride.

Realising the importance of the practical production of aluminium sulphide, I took up the matter some time ago, and have devised the following processes. In order to avoid the difficulties experienced in Petitjean's process in reference to coating of particles of alumina with fusible sulphide, I employed a molten bath of sulphides and fluorides of sodium and potassium, the mixture of sodium and potassium salts melting at a lower temperature than either of the others independently. I then introduced into the molten mass aluminium oxide, which dissolved therein; then I passed vapour of carbon bisulphide through the mass, converting the whole of the aluminium oxide into sulphide. As there were no particles to coat with sulphide to retard the reaction, the sulphide thus produced was in solution in the molten bath, and in better condition for reduction than aluminium sulphide *per se*. I also suspended aluminium oxide in a molten bath, in which it was not soluble, such as chlorides of potassium and sodium, and subjected it to the action of carbon bisulphide vapour, whereby the insoluble particles of alumina were superficially coated with aluminium sulphide, which, being soluble in the molten bath, was dissolved off, exposing new surfaces to action. I also combined aluminium oxide with alkalis, producing aluminates, which I fused and subjected to the action of carbon bisulphide vapour, converting the whole into a double sulphide of aluminium and alkali.

In carrying out these perfected processes it became evident that their commercial importance depended largely on the cheap production of carbon bisulphide, so I turned my attention to the production of that compound. I found that the principal expense in producing carbon bisulphide was occasioned by destruction of retorts, which being composed of iron were rapidly destroyed by the sulphur vapour. I then experimented with a view to employment of fireclay retorts, but found their porosity a great disadvantage, as the carbon bisulphide produced would pass through the pores, converting the aluminium oxide, silica, and other ingredients into corrosive sulphides of disintegrating nature. In order to overcome this I prepared my retorts in the following manner, taking for the basis of my operation the absorbing properties of porous matter for lead oxide, suggested by the cupelling process. I then coated the inside of the clay retorts with lead oxide, and heated to whiteness. The lead oxide was absorbed, filling the pores of the retort. I then coated again with a mixture of lead oxide, powdered feldspar, and quartz, burning as before, and finally coating with lead oxide mixed with sodium silicate, and burning in again in presence of vapour of carbon bisulphide, which converted the superficial coating of lead oxide into sulphide, thus practically preventing further corrosive action; and, after several continued weeks of test, am satisfied that the retorts will, with occasional coating of lead oxide, last for years without replacement, thereby materially cheapening the process of production.

Reduction of Aluminium Sulphide.

Comenge reduced aluminium sulphide by means of iron and copper filings. Reichel claims, also, to have reduced aluminium in this way. F. Lauterborn states the reduction with iron is brought about at red heat. Prof. Joseph W. Richards, in his standard work on aluminium, states that Dr. J. D. Lisle, of Springfield, Ohio, has reduced aluminium sulphide with zinc. Prof. Richards also states that he has himself reduced aluminium sulphide with tin and iron filings. This reduction was performed with sulphide produced by the Petitjean process, and contained only about 33 per cent. of aluminium sulphide, the balance being unconverted alumina, which rendered the sulphide hard to reach with reducing agents.

By employing the solution of aluminium sulphide in molten fluxes, as produced by my process, I have been able to reduce practically all the sulphide by action of iron. This iron I employ in a highly heated molten condition, thereby introducing considerable heat directly in the mass, and is introduced into the molten bath containing the aluminium sulphide in small shot formation, produced by passing the molten iron through a fireclay sieve. The retort being about 10 ft. deep, allows all the iron to become sulphurised before reaching the bottom, thus liberating aluminium. The iron sulphide produced accumulates in a molten condition at the bottom of the retort, from whence it is first withdrawn, then the aluminium, and finally the flux, which is employed to dissolve more aluminium oxide, to be converted as before into sulphide by action of carbon bisulphide vapour.

The reduction of aluminium sulphide by electrolysis is performed with less expense than any other aluminium compound, it only consuming one-half the electric energy required for electrolysis of the oxide. The processes devised and in operation at the present time are those of Bucherer, who produces his sulphide by mixing aluminium oxide with an alkali polysulphide and carbon, heating it to a high temperature and then subjecting to electrolysis. This process for producing aluminium sulphide was evidently invented by Petitjean, and a subject of his British Patent of 1857. Next we have the Jaennigen process (German Patent of 1895), which consists in introducing a powdered mixture of aluminium oxide and sodium carbonate into a heated chamber containing an atmosphere of carbon bisulphide vapour. As this mixture is gradually sifted into this atmosphere of carbon bisulphide at intervals, it is claimed to be transformed into a double sulphide of aluminium and sodium before reaching the bottom, at which point is situated a crucible or melting pot, in which the material accumulates and is fused by heat of an electric arc and finally electrolysed, aluminium separating at the cathode and sulphur at the carbon anode. The sulphur combines with the carbon of the anode, producing carbon bisulphide, which is distilled and used over.

The Aluminium Industrie Actien-Gesellschaft, of Neuhausen, also patent in Germany, under the same date, a process, in which they reduce aluminium sulphide at a temperature below that required to produce carbon bisulphide at the anode, and free sulphur distills which is used over again.

Discussion.

Prof. Chandler asked whether the process which had been described had gone beyond the experimental stage, and was in present operation.

Mr. Blackmore said it would be in about one week. The furnaces were all completed. It had been done on a small scale, and proved that it could be carried out on a large scale. The making of the sulphide of aluminium was carried on to quite a large extent—probably about a ton and a half to two tons of material in the retort.

Prof. Chandler presumed that the supposition was that this would be a more economical process than those processes which were now employed.

Mr. Blackmore said it would be, because from the sulphide of iron produced they could recover the sulphur; but it would be, even if they threw away sulphide of iron. It would not cost over 6 cents a pound to produce aluminium sulphide.

Prof. Chandler said he had witnessed the Castner process of making sodium at Birmingham, and without going into details as to cost, it was certain that the process was a successful chemical operation. The works were in full operation. There were long rows of iron pots containing the melted sodium hydroxide, the melting being done by the resistence to the current. About three times an hour workmen came with a kettle and ladle, opened the lid of the sodium pot, and ladled out metallic sodium. The yield of those works at the time he visited them was a ton of sodium a day, which was sold for about 200*l*. This process had been introduced in two or three places in Germany, and works were now being erected at Niagara Falls to put it into practice there.

What it cost to make the sodium, Mr. Castner was not willing to tell, nor was much sodium being sold, as Mr. Castner had devices to use it, and was really his own customer for it. A considerable portion was converted into peroxide of sodium, and sold in that form. At the present time a considerable portion was employed for the manufacture of cyanide of sodium and potassium, for gold extraction. The advantage of making cyanide of sodium and potassium in this way arose from the fact that when the yellow prussiate of potassium was heated with sodium, neither oxygen nor alkaline carbonates were introduced, and the product was an almost chemically pure mixture of cyanide of sodium and cyanide of potassium, and which actually tested, on the cyanide of potassium basis, over 100 per cent.

He had also witnessed the aluminium process as conducted at Niagara Falls, and formerly conducted at Kensington, near Pittsburg, and that, too, seemed to be as simple and beautiful an electro-chemical process as could possibly be carried out. There were iron vessels there some 6 ft. by 8 ft. broad and 2 ft. deep, lined with carbon. These vessels constituted the cathode. There were some 30 anodes of very compact carbon hanging from copper rods, in three rows of about 10, about three inches in diameter. The cryolite bath had previously been fused in another pot, and the impurities electrolysed out, so that the cryolite that was actually employed as the bath for forming the aluminium contained no silicon and no metal which would contaminate the aluminium. The pure alumina was fed into this pure cryolite bath, and the electrolysis proceeded regularly. Whenever there was a short supply of alumina, the resistance of the bath increased, and an indicator showed at once that the alumina had been consumed, and workmen simply shovelled in some more, the process consisting chiefly in shovelling in alumina and ladleing out aluminium, day in and day out.

As to the expense, that was something he had never had an opportunity to investigate, and whether these new processes would result in a large economy in the cost of production remained to be seen. He presumed Mr. Blackmore had figures, gained by experience, which satisfied him as to the result.

Dr. G. A. Prochazka noted that Mr. Blackmore spoke about aluminium suboxide as being the mother-substance of ultramarine. Without wishing to raise the question of the constitution of ultramarine at the moment, he took issue on that point. Aluminium suboxide was not the mother-substance of ultramarine.

Dr. H. Endemann had written some years ago regarding the constitution of ultramarine. He had shown that it was a substance containing more atoms of non-metallic elements than metallic elements. If they considered in the formula of ultramarine the aluminium combined with this excess of non-metallic substances, they found Al_2O_3 as a residue in the combination. There were really more non-metallic elements with aluminium in combination than there ought to be if it were a substance of the composition of alumina; hence there would not be a suboxide of aluminium. It must be a substance corresponding to even a higher oxide than alumina.

Mr. H. S. Blackmore mentioned that oxide of aluminium was very porous, so that this material would be, and any other material, such as sulphur, would be very apt to be absorbed in the material; so they could not tell whether it was actually combined or not, except by very careful investigation.

Dr. Endemann said he had carefully investigated ultramarine, which consisted of even-coloured globules of ultramarine, under the microscope. It looked like a glass. It did not give a very good colour, but at the same time it was blue throughout, and gave a blue powder on being pulverised, but it was not quite as good as the ordinary ultramarine. It had been exposed to too high heat, in his opinion. It was from the examination of this dense sample that he conceived the idea that ultramarine must be looked upon as a derivative of AlO_2.

ALCOHOL DERIVED FROM GRAIN, IN THE MANUFACTURE OF COAL-TAR DYES.

BY GEORGE A. PROCHAZKA.

Many of you are without doubt familiar with the requirements of the coal-tar dye industry with regard to grain alcohol.

Grain alcohol, as used in the industry with which I am connected, fulfils three distinct functions:—

I. Alcohol is used as a solvent pure and simple, for the extraction and purification of raw materials employed by us, and some of our finished colours.

Our firm, the Heller and Merz Co., produce an eosine of extreme purity by crystallisation from alcohol. The alcohol can be recovered pretty completely, and generally the same degree of purity as originally employed. The recovered product may be used in subsequent operations.

II. Alcohol is used as a vehicle or solvent, in which two or more chemicals are allowed to act upon each other for the production of an entirely new body. The alcohol does not enter into chemical combination, but very often a portion of it is destroyed by the secondary action of these chemicals. The balance may be recovered to be used in subsequent operations. We use it largely in the production of coloured bromine and iodine compounds (bromofluoresceins, rose-bengale). The recovered alcohol is not as pure as the original, while the unavoidable loss in this class is greater than in the first.

III. Alcohol enters into chemical combination with the compounds produced. It cannot be recovered at all, as in I. and II. The ethyl group of the alcohol forms an inseparable constituent, and could not be isolated in the form of alcohol, without destruction of the entire compound.

The consumer demands of the dyestuff manufacturer as full and varied a line of shades of colours as possible. The introduction of the ethyl group, either by itself or in place of the methyl group, produces new and striking results. The manufacturer employs grain alcohol to get his valuable ethyl group, not on account of any personal preference for grain alcohol, but because he must use it. He has no choice.

Colours with the ethyl group are produced industrially in two ways:—

A. Ethyl derivatives of coal tar are prepared that are not dyes. These are converted into dyes by more or less complex methods.

B. A dyestuff of a certain shade is converted into a dyestuff of another shade by ethylation.

The following ethyl derivatives of coal-tar, not dyes, are at the present time largely employed in the manufacture of coal-tar dyes:—Diethylaniline, Ethyl-methylaniline, Ethyl-o-toluidine, Ethyl-benzylaniline, Diethylmetamidophenol, Ethoxybenzidine, and Quinaldine. The last is included because aldehyde is used in its production.

Dyes derived from these Raw Materials.

1. *Diamidotriphenylmethane derivatives:*—
   - *Emerald Green.*—Salts: Tetra-ethyldiamidotriphenylcarbinol.
   - *Patent Blue V.*—Salts of *m*-oxytetra-ethyldiamidotriphenylcarbinoldisulpho acid.
   - *Guinea Green.*—Salts of diethyldibenzyldiamidotriphenylcarbinoldisulpho acid.
   - *Acid Green-Yellow.*—Salts of the trisulpho acid.
   - *Patent Blue A.*—Salts of *m*-oxydiethyldibenzyldiamidotriphenylcarbinoldisulpho acid.
   - *Acid Blue 6 G.*—Salts of *m*-oxydiethyldiamidophenylditolylcarbinoldisulpho acid.
2. *Triamidotriphenylmethane derivatives:*—
   - Ethyl Violet: Hexa-ethylpararosaniline. Fast Acid Violet 10 B. Alpine Blue. Acid Violet 6 B. Acid Violet 6 B. Alkali Violet. Acid Violet 6 B N. Acid Violet 7 B (S. Schultz-Julius tables, III. ed., 314—320).
3. *Diphenylnaphthyl dyes:*—
   - Victoria Blue R. Night Blue (Schultz-Julius, III. ed., 335—336).

4. Pyronine B (Schultz-Julius, 341).
5. Rhodamine 6 G G B (*ibid.* 344—346).
6. Thionine Blue G. Oestr. New Methylene Blue N (*ibid.* 442—445).
7. Quinoline Yellow.
8. The derivatives of ethoxybenzidine, known as:—Diamine Blue B. Diamine Black E. Diamine Black P O. Diamine Blue 3 R. Diamine Red N O (*ibid.* 225—229).

Dyes derived from others by Ethylation.

Chrysophenine (*ibid.* 251), Diamine Scarlet 3 B (*ibid.* 184), Diamine Yellow N (*ibid.* 230), Congo Orange R (*ibid.* 215), Rhodamine 6 G (*ibid.* 344), Anisoline (*ibid.* 347), Spirit Essence (*ibid.* 356).

The so-called Wilson Tariff Act of 1894, while increasing the internal revenue tax on alcohol from grain from 90 cents to 1·10 dols. per proof gallon, allowed a rebate on grain alcohol used for industrial purposes under regulations to be formulated by the Treasury Department. These regulations were never formulated, the tax was not refunded, and the whole matter is now in litigation. In June 1896, Congress repealed the rebate section of the tariff law, and referred the entire question to a Joint Committee of the two Houses, made up of members of the Ways and Means Committee of the House, and the Finance Committee of the Senate, under the chairmanship of the Hon. Orville P. Platt, of Connecticut, who has taken a great interest in the matter. This Committee has been investigating the matter thoroughly. A special delegate was sent abroad to examine the question in different countries of Europe. Hearings of great interest were given in New York and Washington, and it is to be hoped that the long-desired relief will at last be given to those interested.

The Effect of the Tax upon the Coal-tar Dye Industry.

Ethyl colours cannot be manufactured with taxed alcohol in competition with foreign manufacturers who get their alcohol free of tax. Our main competitors—the manufacturers of Germany—have tax-free alcohol, and are treated very liberally by their Government in that respect.

One example—the important raw material diethylaniline, and the valuable green colouring matter, "Emerald Green," derived from it—will be sufficient:—

Value of 100 per cent. Alcohol:

	Dols.	cts.
1 gall. or $6\frac{3}{4}$ lb. Tax paid	2	48
1 lb.	0	37
Tax on 1 gall.	2	20
Tax on 1 lb.	0	33

Diethylaniline.—1 lb. requires in its production not less than $\frac{1}{2}$ lb. of 100 per cent. grain alcohol, rather more.

Alcohol value in 1 lb. diethylaniline:

	Cents.
Tax paid	18·5
Free of tax	2·0
Difference	16·5

The German product, diethylaniline, made with tax-free alcohol, is worth then less than 30 cents per lb.

Emerald Green derived from diethylaniline sells for about 50 dols. per lb. for the imported article, duty paid. It contains ethyl groups, corresponding to somewhat less than 40 per cent. of grain-alcohol, or 14·8 dols. per lb. of colour in tax-paid alcohol, or more than 25 per cent. of the selling price of the colour, duty added.

Alcohol, free of tax, per lb. of colour, 1·6 cents.

The tax in this class of colours is prohibitive. It is impossible for us to manufacture them in competition with the foreign article. It also acts as a very serious bar to the consideration of ethyl alcohol in the inventive development of this important and highly scientific industry in this country.

Where alcohol can be recovered in part, the tax hampers very seriously. The cost of the alcohol used in our manufacture amounts to about 10 per cent. of the value of the colour (alcohol tax paid); with alcohol free, this would amount to a little more than 1 per cent., a very serious difference in the close competitive markets of recent years.

Discussion.

Prof. Chandler asked whether the author felt hopeful of obtaining some relief?

Dr. Prochazka said that the Select Committee appeared to be very favourably disposed towards those industries which required grain alcohol. Of course, they had strong opposition to meet from the manufacturers of wood alcohol, varnishes, and turpentine. In that way the opposition was somewhat concentrated, but he thought relief would have to come. As the meeting would see, it was impossible to tackle those products. He had brought the matter before them to awaken some additional interest in the matter.

Prof. Chandler asked if it was proposed to denature alcohol?

Dr. Prochazka said that the Committee spoke principally of methylation, which would hardly answer their purpose, because they wanted pure alcohol, if they could get it. For instance, if they took methylated alcohol when they wanted to ethylate, they might have very interesting results. When trying to make diethylaniline with methylated spirits, the results might be of an unexpected nature.

Mr. Henry Clementson said that the Western farmer found himself burning his corn, when the distiller, if he had an outlet for his alcohol, which he would have if they had alcohol free of tax, would take the farmer's corn at a good price. The farmer considered it a very serious question. In the United States they had 128,000 bushels distilling capacity per day, and under the present conditions the distillers could use about 40,000 bushels capacity only. By the statistics that the Congressional Committee got while they were abroad, they found under the free alcohol laws of Germany that the industry increased there in seven years something like 1,000 per cent. If they could get half that, the distilling capacity of the country would be utilised, and, instead of the farmer of the West burning his corn, the chemists of the country could develop their chemical factories, and the makers of dyestuffs could make dyes at home. Speaking as a manufacturer of alcohol, he thought that the regulations, instead of encouraging the industry in every way they possibly could, did everything to hinder the industry and its development from a scientific standpoint. They were confined in their hours of fermentation, and the manner in which they might manipulate it and also put to the expense of very heavy bonds in order to do the business. In fact the regulations were such, and the bonds required were of such an enormous amount, that it was only by combination that the industry could be carried on. The Government were promoting a trust in alcohol. He thought from what he learned in Washington, that the present Congress would certainly give them a modified form; that is, certain industries would be allowed to have alcohol free of tax, under certain restrictions. The concensus of opinion there, among those who had it in charge, was that the Government and the people at large were not used to the idea as yet; that it would have to be brought about gradually. For instance, probably the varnish industry would get free alcohol; perhaps they would select the coal-tar dyes as one industry, and possibly some other industries; and as time went on, and they found that those industries are very largely increased and benefit derived therefrom, they would extend it to others.

Mr. M. Toch thought the Chemical Societies of the United States, namely, this Society and the American Society, had not taken any steps towards investigating the free alcohol question. The matter was ripe and something ought to be done in connection with it. The manufacturers of varnishes and colours throughout the United States had endeavoured to obtain free grain alcohol for use in the arts, and he thought that a Society of such standing as theirs should send a deputation to Washington or communicate with those at the head of the Ways and Means Committee. Such a deputation would have great weight, and manufacturers and consumers of alcohol would stand a better chance of getting alcohol free in the arts and sciences and manufactures than they would have otherwise.

Prof. Chandler reminded the meeting that there was no alcohol tax before the war. He had a very vivid recollection of how he was affected by the tax. He was in the

habit of using alcohol in his laboratory in Union College for burning in lamps, as they had no gas. He formerly paid 16 dols. a barrel for 95 per cent. alcohol. Then the Internal Revenue Law was passed in order to raise money to carry on the war, and the next barrel he bought cost over 200 dols. The cost went up from 16 dols. to over 200 dols. The tax was then at the rate of 4 dols. a gallon for pure alcohol. That was a war measure, but it had been in force so long that the generation of people who were living when it was adopted had passed away. There were only a few left, and people of to-day had always been accustomed to this Internal Revenue tax and accepted it as the proper thing. That was the reason the inertia was so hard to overcome; and possibly the temperance societies were in favour of it and threw the weight of their influence against the removal of the tax.

With regard to the methylating of spirits, some years ago the Treasury Department, with a view to initiating legislation upon this subject, applied to the National Academy of Sciences for information as to methylating alcohol; and as to the result, Prof. Marsh appointed a committee of the National Academy of Sciences, with Prof. Remsen and himself as members, to ascertain whether it would be easy to remove the methyl alcohol, together with such bodies as usually accompany it, in case methylated spirits were allowed to come into free use. The Committee found that there was no difficulty whatever in removing every trace of the methyl alcohol and all the substances that accompanied it, and that without difficulty an entirely satisfactory alcohol could be made by the modern methods of distillation, with the aid of alkalis. The Treasury Department seemed to think this raised a serious obstacle in the way of permitting the sale of methylated spirits, and the matter was dropped at that time. They were adding in Europe, in addition to methyl alcohol, pyridine, and he understood that there was now an establishment in San Francisco engaged in manufacturing pyridine from California petroleum, to be used in denaturing alcohol in Europe.

Mr. Clementson said that that point was brought up in the Senatorial Committee. He himself stated his opinion before that committee that people who wanted to defraud the Government would not be found among the educated classes, especially among chemists. The man who started to defraud the Government certainly would not buy methylated spirits for the sake of obtaining alcohol. When he could buy a bushel of corn for 20 cents, and get $4\frac{7}{10}$ galls. of proof spirits from it, he would hardly pay, say, 30 cents for a gallon of alcohol. It would be too costly an experiment. He would require very elaborate apparatus to do that, and as a general rule illicit distillers employed very cheap apparatus, so that should they get caught they did not lose much. He did not think the Government took the right ground in regard to that point. Furthermore, there had been some experiments tried in Charles Cooper's establishment in Newark, and there, with very elaborate apparatus, they did not succeed in de-methylating spirits. The experiment showed that after very careful distillation on a large scale with 100 galls., over 5 per cent. of the methylated spirits remained in the distillate, which would make it unfit for chemical or drinking purposes.

Prof. Chandler explained that their Committee took the ground that it was just as easy for the Government to prevent purification of methylated alcohol as it was now to prevent surreptitious distillation. Therefore that was no argument against methylated spirits.

SEWAGE PRECIPITATION AT WORCESTER, MASS.

BY H. P. EDDY.

In considering the possibilities of any method of sewage treatment it is quite necessary to consider the average results of work performed on a practical scale, under ordinary conditions and extending over a considerable length of time. It will be found that deductions have frequently to be made from the results of laboratory experiments, and that these deductions often seriously reduce the estimate of the qualitative and quantitative efficiency of the process.

In this connection it may be of interest to examine the record of efficiency of the treatment of the sewage at the purification works at Worcester, Mass., where about 16,000,000 galls. are treated daily by precipitation. It must be remembered, however, that the sewage there dealt with is very dilute, averaging throughout the past year only about 0·500 part albuminoid ammonia per 100,000, and that storm water is treated, which at times seriously interferes with the treatment, and to such an extent that its efficiency is considerably reduced.

Table showing the Results of Treatment of Sewage at Worcester, Mass.

Date.	Average, December to December. Albuminoid Ammonia. (Parts per 100,000).	Per Cent. Total Albuminoid. Ammonia removed.	Per Cent. Suspended Albuminoid Ammonia removed.	Average, December to July. Albuminoid Ammonia. Parts per 100,000.)	Per Cent. Total Albuminoid Ammonia removed.	Per Cent. Suspended Albuminoid Ammonia removed.	Average, July to December. Albuminoid Ammonia. Parts per 100,900.)	Per Cent. Total Albuminoid Ammonia removed.	Per Cent. Suspended Albuminoid Ammonia removed.
1894	0·506	50·78	89·54	0·422	46·63	83·26	0·622	53·78	94·36
1895	0·518	51·63	91·11	0·504	49·69	91·73	0·538	52·93	90·75
1896	0·497	53·92	92·02	0·400	52·00	91·45	0·640	56·10	93·35
Average	0·507	52·11	90·89	0·442	49·44	88·81	0·600	54·27	92·82

This table is made up from the results of daily analyses of sewage and effluent. 24 portions of effluent taken hourly and 48 portions of sewage taken half-hourly comprise the samples for daily analysis. This table is arranged so that a comparison can be made between the results of treatment during the wet and dry portions of the year and the average for the entire year. Many conditions beyond the control of the city affect the result, so that it is somewhat difficult to draw conclusions from the figures for each year, but a satisfactory comparison may be made between the averages. It will be noticed that the sewage is much stronger in the dry season than in the wet season, and that this difference has a marked effect upon the efficiency of the treatment. In the wet season the per cent. organic matter removed varied from 46·63 in 1894 to 52·00 in 1896, averaging 49·44 per cent. In the dry season the variation was less, being about 3 per cent., or from 52·93 per cent. in 1895 to 56·10 per cent. in 1896, and averaging for the three years 54·27 per cent.

The suspended organic matter appears to be subject to the same variation. In the wet season of 1894 but 83·26 per cent. was removed, while in the corresponding season of 1896 it reached 91·45 per cent. In the dry season the variation was from 90·75 per cent. to 94·36 per cent., 1894 being the highest.

It can be readily seen that conclusions from the success of a laboratory treatment of a sample of this sewage taken during either the dry or the wet season could not be applied exactly to the results of treatment upon a large scale and extending over a considerable length of time. There are other conditions bearing upon this point, but which cannot be illustrated by any tables of figures; for example, variations in the chemicals contained in the sewage.

In recent years there has been a growing tendency toward the abandonment of chemical precipitation as a method of sewage treatment, and a general feeling that a universal remedy for sewage purification troubles had been found in the methods of land treatment. This feeling has been greatly strengthened by the admirable work done by the Massachusetts State Board of Health, under the able direction of Dr. Thomas M. Drown and Dr. William T. Sedgwick. In this subject, the action of bacteria having been found to be a potent factor in the processes of purification, as in a great many other branches of science, the general fascination for the infinitesimal organisms has led to exorbitant claims and irrational opinions. But, however exaggerated these estimates of the value of land treatment may have been, it is certainly true that much advance has been made in the knowledge of sewage treatment, and it seems probable that this vexing question will cause less trouble in the future than it has in the past, and that much more intelligent action will be possible than has been attained hertofore.

There are, however, many obstacles in the way of land treatment, and these may in many cases be overcome by the aid of precipitation. It would seem that there are conditions under which each method can be worked successfully, and conditions under which they should be used together.

A few conditions under which chemical precipitation can be successfully applied either by itself or in connection with various methods of land treatment follow:—

1. Where there is insufficient dilution afforded by the water into which the discharge is made for crude sewage, but where it is ample for a chemically precipitated effluent.

2. Where the discharge is to be into a tidal body of water and it is impracticable to discharge crude sewage.

3. Where it is not possible to provide a sufficient quantity of land to treat the crude sewage.

DISCUSSION.

Mr. EDDY, in reply to a question from the Chairman, said that the chemical treatment employed at Worcester consisted in the addition of lime to the sewage. It was not, however, what was known as the "Lime Treatment." They had large wire drawing establishments in the City, and the pickling liquids from these works were turned into the sewers, so that the addition of lime to the sewage threw down a precipitate of iron.

Mr. R. C. WOODCOCK asked whether oxidation in sewage was not only helped by micro-organisms, but whether it was not altogether due to this cause. Prof. Warington had published some very important researches on the oxidation of ammonia into nitrites and nitrates, owing to the presence of micro-organisms, and he proved it very cleverly in this way: He took tubes, about 3 ft. long, filled with earth and allowed a weak solution of ammonia salts to be filtered through when the earth was in proper condition, and if the flow of ammonia salt was not too rapid, he found that the effluent contained no ammonia whatever, the whole being oxidised into nitrites or nitrates, or both, according to the organisms he had present. Now, taking those tubes that were active in their work and passing a little chloroform vapour through them, which either destroyed or kept the organisms in a torpid condition, washing out the chloroform vapour by means of air, and then passing through the same solution at the same rate, no oxidation whatever took place. Now, taking another tube so treated and inoculating that tube with a little of the earth from one that was active, at once the earth set up activity again, which undoubtedly proved that micro organisms did the whole work there.

The researches of Tidy and Frankland also very strongly showed that most of the oxidation in flowing streams was due to micro-organisms, and not simply to the absorption of the oxygen direct by the organic matter. Was this, in Mr. Eddy's opinion, due to direct absorption of oxygen or to these micro-organisms?

Mr. EDDY stated that the oxidation of sewage was almost exclusively brought about by micro-organisms. If sterilised sewage were passed through sterile sand, no oxidation to speak of took place. An interesting experiment was to watch the oxidation of sewage in a bottle plugged with cotton to keep out organisms from the air. Upon allowing it to stand for a month a gradual change from the albuminoid into ammonia as free ammonia, and thence to nitrites, and even to nitrates, occurred in the solution. It took a considerable time. The odour, which at first was comparatively slight, increased to a maximum, and then gradually decreased. The growth of the organism increased and decreased *pari passu*. With sterilised sewage one got no change whatever and no odour.

Mr. REUBENS asked whether the author had noted some records of experiments in which iron and zinc were hung in cages in the ducts for the conveyance of the sewage.

Mr. EDDY, in reply, said that he had not seen an account of those experiments. They were quite similar, however, to experiments in the electrical treatment, only that zinc was not used. That was really chemical treatment, because hydrate of iron was thrown down as a precipitant. He did not recollect any experiments with zinc and iron.

Prof. CHANDLER, alluding to the use of zinc, said that there was a reaction of zinc with water and air, which did not appear in the text-books, recorded by a German investigator by the name of Taube. Wet granulated zinc became coated with white hydroxide of zinc; and Taube found that the following reaction took place:—

$$Zn + 2HOH + O_2 = Zn(OH)_2 + H_2O_2$$

When zinc, water, and air came together, the zinc took hydroxyl, and hydrogen peroxide was formed at the same time, so that while zinc appeared as a reducing agent under ordinary circumstances, in this case it might actually be the means of bringing about an oxidation. Iron had recently been used extensively in Antwerp and some other places to purify drinking water.

Journal and Patent* Literature.

I.—PLANT, APPARATUS, AND MACHINERY.

Steam Boilers, Preventing Scale in. Eng. and Mining J. 1897, **63**, 45.

THE introduction of kerosene, drop by drop, into the boiler is advocated on account of its cheapness and the fact that it leaves no residue therein, and has no injurious effect on the iron. An injector is described, so constructed that the amount of kerosene fed into the boiler can be very closely regulated.—A. S.

* Any of these specifications may be obtained by post by remitting 8*d.*—the price now fixed for all specifications, postage included—to Sir Henry Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

Gas Engines, Combustion of Illuminating Gas in. Haber and Weber. Ber. **30**, [2], 1897, 145.

See under II., *page* 227.

PATENTS.

Treating and Extracting Solids with Washing Liquids or Solvents, Impts. in or relating to Apparatus for. T. Fairley, Leeds. Eng. Pat. 1666, Jan. 23, 1896.

The solids are fed through a cylinder by a screw conveyor, and are mixed by the blades of the conveyor with a stream of the washing liquid or solvent flowing through the cylinder in the opposite direction. The form of conveyor employed is that of an ordinary screw conveyor divided longitudinally into two halves, the halves being adjustable on each other, so that the threads of the screw on one half may precede or follow those on the other half by any regulated distance. The apparatus may be jacketed, so that it can be heated or cooled, as desired.—R. A.

Evaporating Apparatus [*Multiple Effect*], *Impts. in.* S. M. Lillie, Philadelphia, U.S.A. Eng. Pat. 5431, March 10, 1896.

A number of evaporators, each comprising a series of heated tubes or other heated surfaces, over which the liquid is circulated by a pump, are connected together to form a "multiple effect," and are provided with suitably arranged pipes and valves to enable the liquid to be sent through the evaporators in succession, in either direction. An escape pipe is provided for conducting the water of condensation from a hotter or higher pressure evaporator to the steam chamber of a cooler or lower pressure evaporator, this pipe having a steam trap to prevent the escape of steam with the water of condensation. The invention also comprises various improvements in the construction of the evaporators and the arrangement of the circulating pumps, the pumps being provided with nozzles adapted for circulating the liquid and discharging it from the evaporators. The entrance of the liquid into the evaporators is controlled by an automatically operated feed valve.—R. A.

Vessels for holding Corrosive Substances or the like, Impts. in. P. Funfak, Leipsic, German Empire. Eng. Pat. 25,031, Nov. 7, 1896.

Vessels for holding acids, alkalis, solutions of salts, and similar substances, are made of wood or other suitable material, lined with celluloid.—R. A.

Evaporating Water Containing Salts and Calcareous Matter, Impts. in Apparatus for, and in Heating Furnaces for Removing the Incrustations from the Coils of such Evaporating Apparatus. S. Straker, 139, Cannon Street, London. Eng. Pat. 28,411, Dec. 11, 1896.

The present invention relates to evaporating apparatus of the kind described in Rayner's Specification, Eng. Pat. 5972 of 1888, and Caird and Rayner's Specification, Eng. Pat. 10,700 of 1893, for evaporating water containing large quantities of salt and calcareous matter. The box-shaped fronts which form the evaporator doors, and carry the evaporator coils, are mounted on wheels, to run on rails, so that they can be readily removed from the evaporating apparatus and run into a heating furnace, for the removal of the incrustations from the coils. The furnace employed, consists of a rectangular sheet-metal casing with a smoke-funnel at the top, and with a sliding door, and provided internally at the bottom with a disc-shaped asbestos wick, on to which petroleum is made to flow from a perforated tubular star connected by a pipe with an external supply of petroleum.—R. A.

II.—FUEL, GAS, AND LIGHT.

Naphthalene, Stoppages in Gas Mains due to. A. Hoffmann. J. f. Gasbeleuchtung, **40**, 1897, 97—98.

From observations made by the author at the gasworks of Kaiserslautern, it is shown that stoppages in mains and pipes due to naphthalene, are not entirely prevented, in spite of the adoption of the most perfect system of cooling the gas before its introduction into the mains. Large air-coolers were erected, and it was whilst repairing a defective joint at one of the coolers, that the cause of the mystery was discovered. Most gasworks having steam-boilers, make it a rule to connect all apparatus with the general steam system, in order to blow them out, should it at any time be found necessary to do this. Owing to a faulty valve communicating with the last cooler of the series, steam was introduced into the vessel; and although the temperature of the gas could be reduced to 14°, it was impossible to condense the steam entirely. The naphthalene which had been deposited in the cooler was volatilised by the steam, and carried with it into the mains. After adopting measures for preventing a recurrence of this evil, the difficulty was entirely overcome. With a view of ascertaining whether the gas, after its passage through the coolers, still contained naphthalene, the author allowed a continuous current of steam from a small pipe to flow into the town's main for two days. The experiment was attended with serious consequences, and had to be abandoned after the first day, numerous complaints about stoppages in the gas-pipes being received. Whether the trouble was due to the presence of naphthalene in the cooled gas, or was caused by the volatilisation of old naphthalene which had been deposited in the pipes before the adoption of the air-coolers, remains an open question, although the author considers the latter view the more probable of the two.—D. B.

Gas Engines, Combustion of Illuminating Gas in. Haber and Weber. Ber. **30**, 1897, [2], 145—151.

Two Deutz engines were used: A, 4-horse, with flame-ignition, and gearing arranged so that the gas-supply was either fully open or shut at each stroke; B, 2 horse, with glow-tube ignition, and geared so as to open the gas-supply partially as well as fully, and thus regulate by altering the proportions of the gases for explosion. The burnt gases were aspirated from the exit tube, and analysed. The combustible portions were burnt over platinum or palladium, first at a low temperature to burn the hydrogen, afterwards at a higher temperature to burn the methane. Qualitative analysis having shown that carbon monoxide was also present, that and the hydrogen had to be burnt together, and wire was used rather than asbestos covered with the spongy metal, as local rise of temperature and consequent combustion of methane were thus more easily avoided. The fillings and explosions were registered electrically, to avoid drawing combustible gases into the sample for analysis if an explosion should "miss."

At full load no appreciable amounts of combustible gases were found in the exhaust, as the following figures show:—

—	CO_2 in Exhaust. (1.)	Additional Amounts after Combustion of Residue. (2.) CO_2	(3.) H_2O.	"Degree of Incompleteness." $\left(\frac{100 \times (2)}{(1) + (2)}\right)$
A	2·1326	0·0046	0·0089	0·22
,,	1·4079	0·0048	0·0066	0·34
B	2·9585	0·0124	0·0106	0·42
,,	2·3226	0·0129	0·0152	0·55

At half load very perceptible amounts of unburnt gases were present, the figures for B giving 6·15 and 8·38 for "Degree of incompleteness," and corresponding with an average content of:—CH_4, 0·13 per cent.; CO, 0·14 per cent.; H, 0·12 per cent. The corresponding percentages in the case of A were 0·092, 0·085, 0·058 respectively.
—J. T. D.

Gas for Domestic Lighting, Value of. [*Incandescent Mantles.*] V. B. Lewes. J. Soc. Arts. 1897, **45**, 101—111.

The oxides from which incandescent mantles are made, do not produce a very great illuminating effect when used alone, the illuminating value per cb. ft. of gas, of mantles made thus, being as follows:—Commercial thoria, 6·0; pure

thoria, 1·0; commercial zirconia, 3·1; pure zirconia, 1·5; ceria, 0·9; yttria, 5·2; lanthana, 6·0; commercial erbia, 1·70; pure erbia, 0·6; alumina, 0·6; chromium oxide, 0·4; barium oxide, 3·3; strontia, 5·5; magnesia, 5·0. Ceria gives a reddish-yellow light; erbia, zirconia, and barium oxide, a yellow light; alumina, a whitish-yellow; and strontia and magnesia, a white light. Although neither thoria nor ceria, when pure, have any particular power of emitting light, yet, if 1·2 per cent. of ceria be mixed with the thoria, the mixed oxides emit a light 14 to 16 times greater than either of the substances used, whilst on increasing the amount of ceria, the luminosity gradually diminishes until, with equal weights of the two, the light emitted is in proportion to the illuminating power of the constituents.

C. Killing states that the luminosity of incandescent mantles is due to the catalytic action of the oxide in a state of extremely fine division, which is added as an excitant to the skeleton formed of the chief material of the mantle, and he found that traces of metallic platinum or iridium, or even gold, osmium, ruthenium, and palladium, act in a similar way to ceria when added to the thoria mantles. With regard to this statement, the author observes that if the catalytic action be the true explanation of the phenomenon, further additions of the excitant ought to increase the incandescence, and certainly would not decrease it, as is the case.

The author's opinion with regard to the injurious effect upon the eyesight of artificial illuminants, is that the best light is one essentially different from ordinary daylight, and as free as possible from the actinic rays present in sunlight. Crookes states, as a conclusion from a series of experiments, that the injurious effect of artificial lights on the eye being principally attributable to the invisible ultra-violet radiation, the cold greenish-blue Welsbach light may be considered to be 2·5 times as harmful as the warm pinkish-yellow light of the "sunlight" mantle.—A. S.

Electric Lighting, Underground. J. Daw, jun. Inst. Mining and Met., 1897, 1—5.

A portable electric lamp $2\frac{3}{4}$ ins. square and 8 ins. high has a "dry" battery in the lower part, and a 1 to 3 candle-power incandescent lamp with porcelain reflectors and switch above. The lamp bulb is protected by a stout glass cylinder, held in place by four brass uprights. The battery consists of one positive plate placed between two negative plates, each composed of plumbic oxide, pumice powder, and india-rubber solution; and the electrodes are separated by an adhesive cellulose moistened with sulphuric acid. Two of such cells are contained in the base of the lamp, and being coupled in series give a total E.M.F. of four volts. The electrical storage is equal to 7 ampère-hours. In charging, the current must not exceed 0·8 ampère. After one month's constant use, sufficient (a trifling amount of) sulphuric acid (sp. gr. 1,100) is added to re-saturate the cellulose composition. The weight of the cell varies from 1 lb. to $3\frac{3}{4}$ lb., according to the time that it will be required to supply light. A one-candle power lamp will last for 16 hours without re-charging. The lamp at present costs 25*s.*, and is expected to have a life of at least 5 years. The energy used in charging is 42 watt-hours, and since the amount of each charge is 7 ampère-hours, the actual cost of a charge to last 12 hours, is 0·062*d.*, taking the cost of generating electricity at $1\frac{1}{2}$*d.* per unit of 1 kilowatt-hour. Allowing for repairs and the renewal of bulbs, the total cost of charging and maintenance may be taken at $\frac{1}{8}$*d.* per lamp per charge.—W. G. M.

PATENTS.

Gas Burners, An Impt. in Atmospheric. J. Moeller, Westminster. Eng. Pat. 24,839, Dec. 27, 1895.

An atmospheric gas burner tube having lateral holes for entrance of air to mix with the gas, so arranged that the streams of air are directed obliquely upwards and inwards. The mixture of gas and air thus produced, burns with a highly heating flame.—A. S.

Artificial Fuel, Impts. in, and in the Manufacture of. H. C. B. Forester, Penybryn, Sketty, near Swansea. Eng. Pat. 682, Jan. 10, 1896.

The artificial fuel is prepared by crushing or reducing coal or the like solid carbonaceous body, mixing it with petroleum, tar, creosote or creosote oils, pitch, naphthalene or naphthalene salts, resin, or other solid or liquid hydrocarbon, and heating the mixture in an externally heated closed vessel under pressure. The mixture is continuously forced through the retort, and is afterwards pressed or moulded if desired.—R. S.

Furnace for producing Coke, A New; with or without suitable Apparatus for collecting the By-Products. G. W. Breffit, Leadenhall Street, London, F. G. Treharne, Wrangbrook, Glamorganshire. Eng. Pat. 841, Jan. 13, 1896.

The furnace is in the form of a muffle, subdivided into narrow chambers, into which, containers or boxes of metal or thin fireclay slabs, and containing the coal to be coked, can be passed. The furnace is initially heated by a fire grate, and the products of combustion travel internally and all around the muffle by suitable flues. When sufficiently carbonised, the boxes are removed, a cover fitted over them, and they are allowed to cool. The gaseous products from the containers may be (1st) collected, the liquid constituents separated, and the gases returned to the combustion chamber, and there burned; (2nd) led at once to the combustion chamber and burned, a suitable arrangement of flues therefor being illustrated; (3rd) allowed to collect in the muffle, and burned, when desired, by admitting oxygen; or (4th) led from one container to the next, whereby a large amount of carbon in the gases is deposited upon the coke, thus tending to enrich and harden the latter. —R. S.

Non-Exploding Mechanical Admixture of Oxygen and Hydrogen, A Process and Furnace Installation for Continuous Process of Production of a. C. Polony, Mittelgasse, Vienna. Eng. Pat. 1300, Jan. 18, 1896.

This non-exploding mixture of oxygen and hydrogen is obtained by the dissociation of steam. It may be separated afterwards into its constituents, or otherwise treated. The three essentials for producing the mixture are: sufficient heat, an impermeable casing, and friction within the producing furnace. The latter has a central chamber, five pairs of concentric semicircular channels arranged in three communicating tiers, the two semicircular pairs being separated by a wall from one another so as to form two independent furnaces as regards working. All the channels and the chamber are built of chromic iron ore. Numbering the channels from the centre chamber outwards, air or oxygen passes downwards through the fourth tier of channels, and enters a mixing or combustion chamber beneath, which also receives the products of combustion from a grate below. The mixture then passes upwards through the first and third tiers, and then across and downwards through the fifth tier, and so to the chimney. The steam for treatment is admitted to the top of the second tier, and, passing downwards, is heated sufficiently to be dissociated, and may be collected in this state. Or the mixture of oxygen and hydrogen may be led to the lower end of the central chamber to react upon any body placed therein, as sodium chloride to obtain caustic soda and hydrochloric acid. If sulphur dioxide be caused to mix with the dissociated gases, sulphuric acid is obtained. Should the temperature fall, the mechanical mixture quietly recombines to form water. If the constituents are required separately, a thin fire-brick partition, platinum in thin sheets, an electric spark, or other means may be adopted. When the first (diffusion) method is adopted, the oxygen may be used instead of air for the combustion process, whereby pure carbonic acid is obtained, or, by a modification, carbonic oxide.—R. S.

Incandescent Oil Lamps, and Igniting Devices therefor, Impts. in or relating to. A. J. Boult, London. From

The Société Dubois Frères, Paris. Eng. Pat. 2022, Jan. 28, 1896.

A BURNER in which the wick tubes are arranged so as to form, at the upper end, an annular vaporising-chamber, which is surrounded by the lower edge of the mantle, the said chamber containing a central chamber having a central conduit terminating in a funnel surrounding the wick tubes, in which funnel the mixture of carburetted air is made. A bell is arranged over this funnel, communicating with the lower part of the outside of the annular chamber and with the wick tubes, and having on its lower edge a recess for the passage of an igniter, and two other recesses for supplying air to the said igniter.

The upper edge of the annular chamber is surrounded by a ring, to prevent admission of non-carburetted air into the interior of the mantle, whilst the upper end of a tube of very small diameter enters the chamber, the lower end being turned upwards and brought under a central opening in the wall separating the burner from the body of the lamp, which opening may be closed or opened by means of a flap-valve operated from outside by means of a key. The vapour of oil heated in the annular chamber passes through this tube and through the opening mentioned, into the mixing funnel, and thence through the central conduit into the central chamber, where the mixture of carburetted air is heated ready for supply to the mantle.

The igniter has two wick-holding tubes, which are introduced so as to surround the wick tubes and central conduit, in order to start vaporisation and to light the lamp.—A .S.

[*Incandescent "Mantle Fluid."*] *The Manufacture of Fluid for Impregnating Bodies or Suitable Fabrics, Threads, Fibres, or the like, for Illuminating or Lighting Purposes; An Improved Process for.* B. Puchmüller, Schöneberg. Eng. Pat. 26,167, Nov. 19, 1896.

A MIXTURE of magnesia and baryta is dissolved in "a suitable liquid or acid" ("bismuth acid, antimonic acid, meta-antimonic acid, citric acid, aconitic acid, oxyallyltricarbonic acid," &c.), then evaporated, the vitreous or salt-like residue pulverised, again dissolved in another liquid or acid ("nitric, tartaric, dioxysuccinic, pyrotartaric, malic, succinic, or formic acids"), mixed with distilled water, and the resulting solution used for impregnating fabrics, &c. Oxide of uranium and strontia may partially or wholly replace the magnesia and baryta.—H. B.

Incandescence Gas Lighting, Impts. in Means for. R. Van De-Ghinste, Brussels. Eng. Pat. 26,304, Nov. 20, 1896.

MANTLES are to be made of wires composed of an alloy of platinum with rhodium, iridium, or palladium, or with mixtures of these. When used with a Bunsen burner the mantle may consist of pure platinum wire, or of an alloy of platinum with 2 or 5 per cent. of iridium, the wire measuring about 0·005 mm. in thickness and the meshes about 0·4 mm. in width. When an intense flame is to be employed, such as that got from a Bunsen burner constructed so as to allow of a free circulation of atmospheric air where the gas escapes from the nozzle into the burner tube, or from a burner in which an air-current is used for injecting the gas, the wires should be 0·1 mm. thick and the meshes 0·5 mm. wide, good results being obtained by employing wire of pure platinum, or of an alloy of platinum with 5—20 per cent. of iridium, or of an alloy of platinum with 10 per cent. of iridium and 10 per cent. of rhodium, or of an alloy of platinum with 10 per cent. of rhodium.—H. B.

Lamps for Burning Hydrocarbon Oil or Spirit, Impts. in or connected with. A. Kiesow, London and Berlin. Eng. Pat. 2281, Jan. 31, 1896.

IN lamps in which an S-shaped or other wick tube descends to near the bottom of the reservoir, and is sealed by the oil, it becomes necessary to admit a small supply of air to the reservoir, in order to equalise the pressure and allow the free flow of oil to the wick. The object of the invention is to connect the wick tube with the reservoir so that the flame cannot flash back through the connection into the reservoir. At the upper part of the wick tube, and at one side thereof, a horizontal channel is stamped from the inside, so as to protrude on the outside, and upon the interior of the tube this channel is covered by a light plate, which thus converts it into a tube. The channel is arched from the horizontal line, so that its ends are lower than the centre. At the highest point of the arc there is a small hole opening into the reservoir, and at each end are small holes opening into the wick tube. Thus, the interior of the wick case is placed in communication with the reservoir, and the air can pass through; but the passage is so narrow and circuitous that the flame cannot flash back, and if any oil should, accidentally or by condensation, lodge in the channel, it will flow out at the holes at each end, into the wick tube. Instead of a stamped channel being made, an arched tube may be soldered on to the outside of the wick tube, and perforated as above, or a minute tube or channel of S-form may be furnished, one end communicating with the wick case, the other with the reservoir.—H. B.

Washing, Scrubbing, and Purifying Gas; Impts. in Apparatus for. S. Hersey, K. Hulett, and Chandler, Ltd., Palace Chambers, Bridge Street, Westminster. Eng. Pat. 5686, March 13, 1896.

THE apparatus consists of a vertical column divided into a number of superposed compartments, up which the gas passes whilst the washing liquid flows downwards. The latter enters by a pipe at the extreme top, falls on to a tumbling vessel, which distributes it over a horizontal perforated plate, from which it passes between two superposed sets of corrugated vertical iron plates, the plates of each set being placed close together, but at right angles to the other set. From these compartments it flows through two more compartments connected by an overflow pipe having a regulating valve, and finally escapes from the lower of these two compartments by an overflow pipe having a valve. The gas to be treated enters at the bottom of the column, and flows upwards through the last two compartments mentioned, by means of special roses dipping into liquid. The gas next passes upwards between the corrugated plates, and then escapes. The major claim is for apparatus for the above purpose, consisting of a gas- and water-tight vessel divided into a number of superposed compartments, some of which contain roses of special construction, and some, sets of vertical corrugated plates, this vessel being provided with an inlet and outlet for the water or cleansing liquid. The employment of a tumbler to project water, &c. into the apparatus, and of a perforated plate for spreading the water, &c., is also claimed, as also the combination of parts to form the whole apparatus.—R. S.

Gas-regenerative Furnaces [*Water-sealed Valve*], *Impts. relating to.* E. Johnson and J. Thompson, Lemington-on-Tyne. Eng. Pat. 5738, March 14, 1896.

"THE novelty of this invention consists in the method of applying a water-sealed valve for changing the direction of the current of the gas or air when entering into separate flues." The flues communicate with an upright chamber, which may be circular in plan, and is halved vertically by means of a central partition, the two halves being open at the top. Round the upper edge of each chamber and across the partition runs a narrow trough containing water, to serve as the water seal. Rising from the middle point of the partition is arranged a central upright spindle carrying at right angles the valve or lid, which is large enough to completely cover one compartment of the chamber. Surrounding the underside of the lid is a downward projection, which dips into the water trough and so forms an air-tight seal to prevent leakage. When it is desired to alter the direction of the gas or air, the spindle, carrying the lid, is raised till the downward projection clears the trough, and the lid is then turned round until it comes over the opposite compartment of the chamber; it is then lowered until the projection dips into the water trough. Now the previously open compartment is covered and the previously covered compartment is opened. The valve may be used for reversing the direction of the flow of steam or other gaseous products of metallurgical processes.—H. B.

Acetylene Gas, Impts. in Apparatus for Use in the Production of. C. G. Luis, Denmark Hill, Surrey. Eng. Pat. 1332, Jan. 18, 1896.

The improved apparatus consists of a vessel divided horizontally into two partitions, the upper of which contains the water and the lower the calcium carbide. A tube conveys the gas as it is liberated, to the space above the water in the upper chamber. This chamber is provided with a safety-valve. The amount of gas generated, is automatically regulated by the pressure of the gas on a ball-float or a diaphragm actuating a conical valve, which controls the flow of water to the carbide. Suitable air-tight caps are provided for replenishing the chambers and removing the waste. The apparatus may be constructed either as a table-lamp or for the supply of several burners. A series of wire gauzes are arranged in the gas-exit pipe, to prevent lighting back under reduced pressure.—J. A. B.

Acetylene Gas, Impts. in Apparatus for use in the Manufacture of. E. B. Pym, Kensington, and J. Gore, Clerkenwell. Eng. Pat. 3219, Feb. 12, 1896.

The pressure of gas in the mains is said to vary considerably with apparatus as heretofore constructed. The patentees obviate the defect by introducing a second storage chamber provided with a float or diaphragm subject to the pressure of the gas therein. The float controls the movements of the valve which admits the gas from the first chamber; and thus the pressure in the second chamber, from which the mains are fed, is kept fairly uniform. If the pressure of gas in the first chamber rises unduly, the valve lifts independently of the control of the float, and the pressure in the second chamber rising, a water seal is forced and surplus gas escapes to the air. The patentees also claim a duplex generating apparatus provided with cocks and by-passes on the water supply for the purpose of automatically diverting the flow of water from one generating chamber to the other as soon as the carbide in the first is exhausted. The first chamber may then be recharged with carbide, and the water will be automatically diverted back thereto as soon as the carbide in the second chamber is exhausted.—J. A. B.

Acetylene and other Hydrocarbon Gases, An Improved Apparatus for Generating. J. F. Blakeley, Ravensthorpe, Yorks. Eng. Pat. 5150, March 7, 1896.

Carbide is placed upon horizontal or inclined trays in a chamber, at the bottom of which water enters from a supply pipe furnished with a tap, actuated, through levers, by a bell governor, which rises and falls with increase or decrease of the pressure of the gas in the chamber. When the gas is evolved more rapidly than it is consumed or stored, the pressure rises and the supply of water is automatically diminished and the evolution of the gas thereby decreased. In another form of the apparatus, the generating chamber has a water-jacket, through which the gas-outlet pipe passes on its way to the holder. The latter controls the water supply in the same manner as does the governor in the first form. In a smaller apparatus, the carbide is put on a grate and water is admitted at the bottom of the chamber from an annular cistern at the top by means of a siphon. When gas is evolved too freely, the increased pressure in the chamber forces the water back into the cistern. In the middle of the top of this apparatus is a special closed conical chamber for feeding the generating chamber with carbide without interruption of the process. Preference is given to a combination of two generators with one storage gasholder.—J. A. B.

Acetylene Gas or other very rich Hydrocarbon Gas, A Method of Burning. J. R. Wigham, Dublin. Eng. Pat. 4285, Feb. 26, 1896.

The gas is forced to the point of combustion through tubes with fine needle-hole outlets (equal to No. 7 or smaller of English make) through which the gas issues in straight jets, in contradistinction to fishtail or batswing jets. Where powerful lights are required, the jets are arranged in groups closely side by side or concentrically, so that the whole somewhat resembles an Argand deprived of a chimney glass. Over each jet or group of jets is suspended a tube of mica or other material, to cause an upward current that renders the flame smokeless and steady. The burner tubes are surrounded with a conical-shaped perforated metal cover, to check the current of air and heat it somewhat, before it comes in contact with the flame. The claim is for the method of burning acetylene gas.—H. B.

Lighting by Acetylene or other Gases rich in Carbon, A New or Improved Burner for. S. D. Gillet, G. Forest, and J. E. O. Bocandé, Paris. Eng. Pat. 27,086. Under International Convention, May 30, 1896.

The gas is mixed with air, previous to combustion, on the Bunsen principle. The gas first passes through metallic gauze into an upright nozzle which is fixed within a chamber. In the wall of the chamber are air-inlet apertures at a convenient height in relation to the mouth of the nozzle. In one form of the apparatus a slide is arranged to regulate the inward current of air. The mixing chamber terminates in a small orifice at its upper part, above which is a tapped part into which any suitable ordinary burner, such as the batswing, may be screwed, at the mouth of which the mixture of gas and air is burned.—H. B.

Acetylene Gas, Impts. in the Production and Purification of, and in Apparatus therefor. A. Schemidt, Vitry sur Seine, and O. Kaufman, Paris. Eng. Pat. 16,432, July 24, 1896.

Acetylene is produced by adding carbide in small quantities to a relatively large bulk of water, and is purified by passage in turn through dilute acid, solutions of lead acetate, acidified copper sulphate, and caustic soda, which remove respectively ammonia, sulphuretted hydrogen, phosphoretted hydrogen, and traces of acid. It is then passed through a chamber containing hygroscopic material, and is subsequently compressed and liquefied. The purification and drying of the gas are said to render unnecessary the use of refrigerants during compression.

The improved generator has a reciprocating slide working gas-tight between its top and the mouth of a hopper. There is a hole in the slide which comes alternately beneath the hopper and above a hole in the generator-top, and thus conveys carbide from the hopper to the generator. The carbide drops on a grid just below the level of the water in the generator. In the generator is a rotating stirrer, and pipes are provided for maintaining the water-level and removing both gas and lime sludge. The stirrer prevents the waste from depositing.—J. A. B.

Acetylene Gas Generators, Impts. in. H. H. Lake, London. From V. Hanotier and G. Hostelet, both of Chimay, Belgium. Eng. Pat. 27,697, Dec. 4, 1896.

The apparatus consists of several superimposed vessels with appropriate connections. The lowest vessel receives calcium carbide, on which water from a higher compartment is sprayed. In the event of surplus production of gas, the flow of water is automatically diverted into an empty compartment. The surplus gas at the same time forces a water seal and passes into a spherical chamber and thence into a chamber containing sulphuric acid, by which it is absorbed. When the gas production is normal, the gas bubbles through water in another compartment, the upper part of which is furnished with a grid loaded with quicklime and two fine wire gauzes. The purified gas circulates around the carbide receptacle, and thus recovers a portion of the heat disengaged by the chemical action, and arrives at the places of consumption in a heated state.—J. A. B.

Acetylene Gas, Improved Generator, Purifier, and Holder. A. G. Smith, Aberdeen. Eng. Pat. 22,646, Oct. 13, 1896.

Calcium carbide is put into a perforated tube with projecting wires, which serve to support it on a pair of quadrants attached to the inner surface of a bell. A lower pair of quadrants act as guides for the tube. The bell is slipped into an outer vessel containing water. The gas-exit pipe passes downwards through the water, and water condensed from the gas is drawn off from a tap at the base of the upright tube fixed by the side of the generator, into which upright vertical tube this gas-exit pipe passes. This vertical tube is packed with pumice or other material, and

serves as a purifier. The claims are: (1) "For the means of holding the calcium carbide in the gas-holder"; and (2) "For the combined condenser and purifier provided by means of the upright pipe and the tube at the side."—J. A. B.

Acetylene Gas, Impts. in Apparatus for the Production and Combustion of. R. Turr, Paris. Eng. Pat. 24,271, Oct. 30, 1896.

The apparatus consists of two concentric vessels, the inner one of which is closed to the air, and has suspended within it a cage loaded with carbide of calcium. The vessels are in communication near the base, and are charged with water to a certain height. A layer of petroleum floats on the water in the inner vessel. When the evolution of acetylene occurs at a greater rate than its consumption, the pressure within the inner vessel forces down the liquid and leaves the carbide in contact with the petroleum, whereupon the evolution of gas at once ceases. The residue from the carbide falls through the meshes of the cage. A gas generator and a lamp embodying the improvements claimed, are described in detail.—J. A. B.

Electric Furnaces [Carbides], Impts. in and relating to. S. Regnoli, F. Lori, R. Pignotti, M. Pandaleoni, and M. Besso, Rome, Italy. Eng. Pat. 13,297, June 16, 1896.

Apparatus is illustrated for the collection and utilisation of combustible gases from electric furnace reactions for heating up the material previous to electric treatment, or maintaining the space around the furnace at a high temperature, or for other purposes; treating the material in an atmosphere formed by the same gases as are generated in the furnace, which is surrounded by an air chamber heated by burning the recovered gases, with the object of diminishing the dispersal of heat. The furnace has a bottom in the form of a sliding case easily changeable to avoid breaking up a portion of the furnace when changing the electrodes, and is lined with refractory material of feeble conductivity to avoid the formation of lateral arcs.—J. C. R.

Electric Furnace [Carbides], A New or Improved. F. H. Haviland, A. Holloway, J. B. Collier, and W. H. Murch, Bournemouth. Eng. Pat. 15,448, July 13, 1896.

The electric furnace or pot for containing the material to be treated, is set in a furnace for the purpose of heating it and its contents, and, when a high temperature is reached, setting up the electric arc. The pot (plumbago) is shown as one electrode.—J. C. R.

Charcoal Fuel Blocks, Impts. in the Manufacture of. S. Katz, Hamburg. Eng. Pat. 21,098, Sept. 23, 1896.

Solid briquettes or blocks of charcoal are made from waste wood, sawdust, or other disintegrated wood, and are then heated under pressure, whilst excluded from air, till reduced to charcoal.—R. S.

Purifying Gases or Vapours [Coal Gas, Furnace Gases &c.], and gaining By-Products therefrom, and subjecting Gases or Vapours to the Action of Solid or Liquid Substances, and the Converse, by Means of Centrifugal Action: Impts. in, and Apparatus therefor. E. Theisen, Baden-Baden, Germany. Eng. Pat. 23,595, Oct. 22, 1896.

The patentee employs the method of obtaining intimate frictional contact between gases and liquids described in the specification of his prior patent, Eng. Pat. 16,726, 1892 (this Journal, 1893, 742), to obtain the separation from gases or vapours of certain of their impurities or separable constituents, or to cause the gases or liquids to react upon a thin layer of liquid or solid matter, or the converse. The apparatus employed consists essentially of a chamber, or a revolving cylinder, or a series of such, against the inner surface or surfaces of which the gases or vapours are caused to impinge by the action of revolving vanes, various modifications of this form of apparatus being described. The inner surfaces of the chambers or cylinders may be supplied with a layer of liquid or solid material of such a nature that the required absorption of, or chemical reaction with, the gases will be obtained, or the absorbing layer may be deposited by centrifugal action from the gases themselves. As an example, the separation of tar, ammonia, sulphuretted hydrogen, and carbonic acid gas from coal gas is fully described in the specification.—R. A.

Electric Device [Arc Lamp] for Showing the Relation of Oxygen and Nitrogen in Mines and other Places, Improved. L. Cohn, Breslau, Germany. Eng. Pat. 28,609, Dec. 14, 1896.

"For controlling the state of the air in mines, chemical and other works," an open arc lamp is employed which is placed in circuit with a meter on the dial of which contact pins are fixed in connection with an alarm bell, which registers a "dangerous surplus of nitrogen" through variations in the burning of the lamp.—J. C. R.

IV.—COLOURING MATTERS AND DYES.

Curcumin [from Turmeric], A Review of the Composition of. Ciamician and Silber. Ber. 1897, **30**, 192.

The authors have repeated the experiments of Jackson and Menke (Beilstein's Handbuch II., Aufl. III., 414), who attribute to the body the empirical formula $C_{14}H_{14}O_4$ and the constitution $C_6H_3(OH)(OCH_3).C_6H_6.COOH$. The pure curcumin employed by the authors was recrystallised from methyl alcohol and benzene until a constant melting point of 183° was obtained; the crystals occurred in two forms—in brilliant red needles and as lighter coloured prisms; both forms melted sharply at 183°. The melting point of Jackson and Menke's curcumin was 178°. Ultimate analysis led the authors to adopt the empirical formula $C_{21}H_{20}O_6$ and the constitutional formula $C_{19}H_{14}O_4(OCH_3)_2$.

On acetylating this pure curcumin, diacetylcurcumin, which crystallised from alcohol in long yellow needles melting at 170°—171°, was obtained. The constitution of this product was found to be $C_{19}H_{12}O_2(OCH_3)_2(OC_2H_3O)_2$. On methylating curcumin, a small crop of crystalline golden needles melting at 135°, and having the constitution of dimethylcurcumin, $C_{19}H_{12}O_2(OCH_3)_4$, was obtained. In addition to these products, curcumin is found to form definite compounds with phenylhydrazin and with hydroxylamine. The hydroxylamine body, the formula of which is given provisionally as $C_{21}H_{19}NO_5$, occurs in clumps of fine needles, having a melting point of 173°. The composition of this and of the phenylhydrazine compound require further attention.—J. O. B.

Dinitronaphthalenes, the Formation of. C. Gassmann. Bull. Soc. Ind. Mulhouse, 1896, 381—382.

Nitric acid acting upon naphthalene produces at first α-mononitronaphthalene, and on further action in the presence of sulphuric acid, a mixture of dinitronaphthalene 1:5 and 1:8, results. At low temperatures and with very strong acids the 1:8 derivative is chiefly formed, while for a larger yield of 1:5 dinitronaphthalene, high temperatures and the ordinary concentrated acids should be employed. In order, for example, to obtain a considerable yield of the 1:8 dinitronaphthalene, 128 grms. of naphthalene are mixed with 110 grms. of nitric acid (61·7 per cent. HNO_3), when mononitronaphthalene is formed and floats as an oil on the surface. The mixture is cooled, and a previously cooled mixture of 300 grms. of ordinary concentrated sulphuric acid and 100 grms. of fuming acid (60 per cent. SO_3) is gradually introduced together with 150 grms. of nitric acid (61·7 per cent.), a low temperature being maintained. When all has been added, the mixture is heated for 12 hours on a water-bath and the liquid poured whilst still hot into 5 litres of cold water. The products are collected on a filter, well washed with water to remove adhering acid, and separated by Beilstein's method with acetone. With the quantities here given the yield of crystalline products is 79 grms. of 1:5 dinitronaphthalene and 115 grms. of the 1:8 derivative.—R. B. B.

Yellow Dyestuff derived from Dinitrofluorescein. F. Reverdin. Ber. **30**, 332—334.

The subject-matter of this paper has already been abstracted (Eng. Pat. 3966 of 1896; this Journal, 1897, 137). The sub-

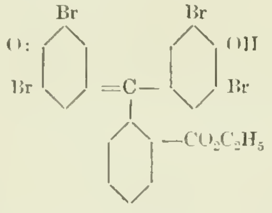
stance obtained by the action of ammonia on dinitrofluorescein has probably the formula—

$$C_6H_4 \begin{matrix} / COO \backslash \\ \end{matrix} C \begin{matrix} / C_6H_2.NO_2.OH \\ > NH \\ \backslash C_6H_2.NO_2\ OH \end{matrix}$$

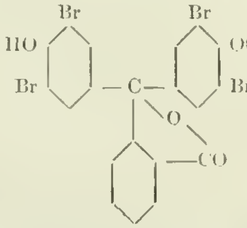
and dyes level shades on wool from an acid bath. The fastness to light of this dyestuff, as well as of its ethyl ether, surpasses that of the fluoresceïn derivatives, although inferior to that of tartrazine. The action of ethylamine on dinitrofluoresceïn gives an orange, whilst p-toluidine yields a yellowish-brown dyestuff for wool. By acting with ammonia on tetranitrofluoresceïn no decisive result was obtained, but by allowing 1 kilo. of tetranitrofluoresceïn, 1 kilo. of caustic potash, and $1\frac{1}{2}$ litres of water to stand for some days at the ordinary temperature, a yellow colouring matter is produced, which, however, is very fugitive to light.—T. A. L.

Phenolphthaleïn, Quinoid Derivatives of. R. Nietzki and E. Burckhardt. Ber. **30**, 175—180.

The oxidation of fluorescinether ester gives the quinoid-fluoresceïn ester, which further yields the quinonoid-diethyl ether and the monohydroxyl ether (Nietzki and Schröter, Ber **28**, 44). Phenolphthaleïn does not, however, behave in the same way. Its carboxyl ester has already been prepared (Herzig, Ber. **28**, 3258; **29**, 158), but this, on oxidation, is not converted into a quinonoid-phthalin ester. This reaction does, however, take place with the tetrabromophthalin ester, which dissolves to a colourless solution in alkalis. On treating the alkaline solution (containing 4KOH) with potassium ferricyanide, a deep blue solution is obtained, which, on standing, deposits the potassium salt of tetrabromphenylphthaleïn ether—



This substance is a pronounced dyestuff, and gives pure blue shades on wool and silk. If a dilute alkaline solution of the ether be carefully acidified with acetic acid, an almost colourless solution is obtained. On dipping a skein of wool or silk into the latter, and warming, the fibre is dyed blue, from which it follows that the animal fibre acts as an alkali towards the colour acid. This blue colour is also produced when the free ester comes into contact with the skin. On further ethylation, the yellow quinonoid-diethyl ether is obtained, and this, on saponification, yields the ethyl hydroxyl ether, which up to the present only exists in the lactoid form, and has the formula—



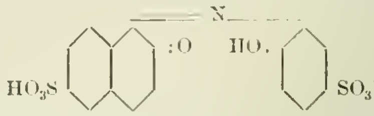
This gives an acetyl compound when treated with acetic anhydride, whilst tetrabromphenolphthaleïn, when heated with ethyl bromide and alkali, only gives the lactoid-diethyl ether.—T. A. L.

Azo Dyestuffs derived from α-Naphthol and α-Naphthylamine Sulphonic Acids, Constitution of. L. Gattermann and H. Schulze. Ber. **30**, 50—55.

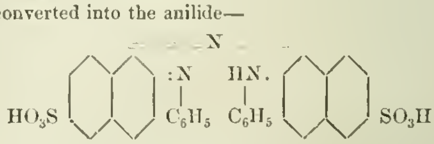
The formation of azo dyestuffs from the sulphonic acids of α-naphthol and α-naphthylamine has been held to be dependent on the two following laws:—1. The diazo group enters para to the hydroxyl or amido group if this position be free. 2. If the para position be substituted by a sulphonic acid group, but the ortho position be free, the diazo group enters there. Several cases are, however, known in which azo dyestuffs of the ortho series have been formed even though the para position is free. The authors have examined the azo dyestuffs obtained by combining diazobenzene chloride with 1·4′-naphthol sulphonic acid, 1·4′-naphthylamine sulphonic acid, and 1·3-naphthol sulphonic acid. The 1·3-naphthylamine sulphonic acid would not combine with diazobenzene chloride, but only with p-nitrodiazobenzene chloride. All these products were reduced with stannous chloride, the sulphonic acid group being subsequently removed by means of sodium amalgam. The resulting products were either 2·1-amidonaphthol or 1·2-naphthylene diamine. In the case of the dyestuff from diazobenzene chloride and 1·4′-naphthylamine sulphonic acid, a further proof that the azo group enters ortho to the amido group was given by the fact that the benzylidene derivative obtained by the action of benzaldehyde did not give off benzaldehyde when heated with acids, as Goldschmidt (Ber. **23**, 496, and **24**, 1000) has shown is the case with amido-azo dyestuffs of the para series. From the results obtained, the two following laws have been formulated:—1. If the position para to the hydroxyl or amido group be free, and the ortho and peri positions relative thereto are unsubstituted by sulphonic acid groups, dyestuffs of the para series are formed. 2. Azo dyestuffs of the ortho series are formed if the para position be occupied, or if it be free and the ortho or peri position relative to it contain a sulphonic acid group, providing, of course, that the ortho position is free. In further confirmation of the above laws, it is noteworthy that 1·2·4′-naphthol disulphonic acid does not combine with diazo compounds in spite of a free para position.—T. A. L.

2.3′-Naphtholsulphonic Acid, Action of Nitrous Acid on. R. Nietzki and T. Knapp. Ber. **30**, 187—190.

In the preparation of 1.2.3′-nitronaphthol sulphonic acid (used for the manufacture of eikonogen), the formation of a red by-product has been frequently observed. In order to obtain this product in larger yield, the authors stir 2.3′-naphtholsulphonic acid (Schaeffer acid) or the sodium salt, to a paste with nitric acid of sp. gr. 1·3, and run in fuming nitric acid saturated with nitrous acid at a temperature of 0° C. An equal volume of water is then added, the crystals are filtered off and washed with dilute nitric acid until the wash-water is a dark red. Analysis gives figures corresponding to the formula $C_{20}H_{13}NS_2O_8$. The analysis and reactions of the substance point to its being allied to indophenol, and probably possessing the constitutional formula—



When treated with an excess of aniline on the water-bath, it is converted into the anilide—



It yields a crystalline leuco compound with stannous chloride and hydrochloric acid, but on oxidation this gives a bluish-violet coloration, and not the original dyestuff.
—T. A. L.

Diquinoyltrioxime and Tetranitrophenol. R. Nietzki and F. Blumenthal. Ber. **30**, 181—186.

By the action of hydroxylamine hydrochloride ($1\frac{1}{3}$ mols.) on dinitrosoresorcinol (Fast green), an intermediate product is formed, which differs from the tetroxime obtained by Goldschmidt in being insoluble in alcohol, and from dinitrosoresorcinol, in giving blood-red instead of green salts

with iron. The substance is diquinoyltrioxime, having the constitution 2.4.3.4 or 1.2.3.4.$C_6H_2.O.(NOH)_3$. The action of acetic anhydride in the cold yields a diacetyl derivative, $C_6H_4N_3O_4(C_2H_3O)_2$, m.p. 142° C., which, on boiling with acetic anhydride forms an anhydride, $C_6H_4N_4O_6$, melting at 181° C. This latter substance gives with ferrous (but not with ferric) salts, a dark green iron salt which crystallises from acetic ether. On reducing the trioxime with stannous chloride and hydrochloric acid and adding alcohol and sulphuric acid, the sulphate of a new triamido-phenol is precipitated. It gives a blue coloration with ferric chloride and other oxidising agents, and also yields a picrate, a triacetyl derivative melting at 230° C., and a tetra-acetyl compound, m.p. 211° C. The oxidation of the trioxime did not yield any definite products, and the action of nitric acid, even dilute, gives a tetranitrophenol. This product, which probably has the constitution—

$$1.2.3.1.6.C_6H.OH(NO_2)_4$$

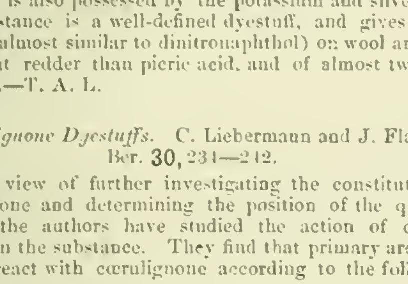
is very easily soluble in water, acetic acid, and acetic ether, but less easily in benzene and petroleum spirit. It melts at 130° C., and occasionally explodes violently. This property is also possessed by the potassium and silver salts. The substance is a well-defined dyestuff, and gives yellow shades (almost similar to dinitronaphthol) on wool and silk, somewhat redder than picric acid, and of almost twice the strength.—T. A. L.

Cœrulignone Dyestuffs. C. Liebermann and J. Flatau. Ber. **30**, 234—242.

With a view of further investigating the constitution of cœrulignone and determining the position of the quinone groups, the authors have studied the action of certain amines on the substance. They find that primary aromatic amines react with cœrulignone according to the following equation—

$$C_{16}H_{16}O_6 + 2NH_2X = 2CH_3OH + C_{14}H_{12}O_4(NX)_2$$

forming deep blue dyestuffs giving induline-like shades. Secondary amines like methylaniline, acetanilide, and diphenylamine do not, however, react, and the fact that benzylaniline after prolonged heating does give a colour, is ascribable to the circumstance that this product splits up during the process into benzaldehyde and aniline. Similarly, tertiary bases like dimethylaniline and quinoline are without action, and, in fact, this reaction may be made use of for detecting the presence of 1—2 per cent. of aniline in quinoline. The compounds obtained have probably one of the following formulæ—



that is to say, they are peculiar quinone anilides, giving however, dark blue shades. As compared, for instance, with naphthoquinone anilide, which is yellowish-red, these products bear the same relation to it that the phenyl do to diphenyl derivatives, the latter of which, in their azo compounds, give as a rule darker shades than the former. This may possibly be an explanation of the blue colour of the new compounds. Even sulphurous acid easily reduces the dyestuffs to leuco derivatives, and this fact renders difficult their employment in dyeing. They can be employed directly from a solution or else from the reducing vat, but are deficient in fastness. The first term of the series would be known as dianilidodimethoxydiphenylquinone, for which the trivial name lignone blue is suggested. The preparation of one of its homologues, p-dimethyl lignone blue, is as follows:—10 grms. of cœrulignone and 120 grms. of glacial acetic acid are heated together and treated with 8 grms. of p-toluidine. The reaction only lasts a short time, and on cooling, the dyestuff separates out (with a yield of 60—70 per cent.), and may be recrystallised from glacial acetic acid. Homologous and analogous derivatives are obtained from aniline, p-bromaniline, m-chloraniline, ψ-cumidine, o- and p-anisidine, anthranilic acid, m- and p-amidobenzoic acids, and sulphanilic acid.—T. A. L.

PATENTS.

Sulphonic Acids of Alkyl Derivatives of Meta-amido-phenolphthaleïne [Rhodamines], Manufacture of. O. Imray, London. From "The Farbwerke vormals Meister, Lucius, and Brüning," Hoechst a M., Germany. Eng. Pat. 2999, Feb. 10, 1896.

Certain alkylated m-amidophenolphthaleïns give salts which are only stable in presence of an excess of acid, and are decomposed in the dyeing process, becoming insoluble. The patentees avoid this difficulty by converting the rhodamines into sulphonic acids by treatment with fuming sulphuric acid at a low temperature. Thus, 5 kilos. of symmetrical di-ethyl-m-amidophenolphthaleïn or its hydrochloride are gradually stirred into 100 kilos. of fuming sulphuric acid (10 per cent. SO_3) at a temperature below 20° C. The melt is agitated until a sample dissolves to a clear solution in water containing an excess of alkali. After pouring into water, the precipitation of the sulphonic acid is completed by the addition of salt. The free sulphonic acid is then dissolved in sodium carbonate solution and evaporated to dryness. Wool completely exhausts the acid dye-bath and is dyed the same shade as the un-sulphonated di-ethyl rhodamine, with a brilliant fluorescence. The free sulphonic acid is sparingly soluble in water, more easily in alcohol with a bright yellowish-red colour and a greenish-yellow fluorescence, whilst it is insoluble in benzene, chloroform, ether, and acetic ether. The alkali salts are easily soluble in water to yellowish-red solutions having a yellowish-green fluorescence.—T. A. L.

Safranine Azo Dyestuffs Soluble in Water, Manufacture of. O. Imray, London. From "The Farbwerke vormals Meister, Lucius, and Brüning," Hoechst a M., Germany. Eng. Pat. 5090, March 6, 1896.

The compounds obtained by combining diazotised Safranines with α- and β-naphthols and their sulphonic acids are insoluble in water. By allowing the combination, however, to take place in presence of ammonia, soluble compounds are obtained, and an especially valuable one is produced from β-naphthol. The same result is effected by acting with an ammonium salt on the insoluble compounds. The following quantities are employed:—7 kilos. of Safranine T (from tolylene diamine, o-toluidine, and aniline) are dissolved in 500 litres of water and 5·5 litres of hydrochloric acid (28·7 per cent.), and diazotised with a solution of 1·6 kilos. of sodium nitrite. The diazo solution is then run into a well-agitated solution of 200 litres of water containing 2·9 kilos. of β-naphthol and 8·5 kilos. of 20 per cent. ammonia. After stirring for two hours, 50 litres of brine are added, and the precipitated dyestuff is filter-pressed and dried at a low temperature, or used as a paste. It dissolves easily in cold or warm water with a bluish-violet colour, and dyes mordanted or unmordanted cotton indigo-blue shades.—T. A. L.

New Raw Materials [Benzidine Derivatives], and of Direct-dyeing Colouring Matters derived therefrom; Manufacture of. C. D. Abel, London. From "The Actien Gesellschaft für Anilin Fabrikation." Eng. Pat. 7313, April 4, 1896.

The new raw materials are derivatives of diphenylamine and benzidine, and are represented by the following general formula:—

$$H_2N.D.NH.C_6H_3NH_2.SO_3H,$$

where D is the radicle of a benzidine compound. The latter are prepared by reducing the nitro compounds

obtained by condensing benzidine with o-chloro-m-nitrobenzene sulphonic acid. These diamido compounds can be converted into tetrazo derivatives, which, after combination with various components, give dyestuffs dyeing unmordanted cotton from an alkaline or salt bath. The following is the preparation of the condensation product:—27·8 kilos. of the sodium salt of o-chloro-m-nitrobenzene sulphonic acid and 18·4 kilos. of benzidine are dissolved in alcohol containing 50 per cent. of water, the whole being then heated to 150° C. in a closed vessel. The sodium salt of p-nitrophenyl benzidine-o-sulphonic acid is then precipitated with salt. To effect the reduction, a solution of 40·8 kilos. of the sodium salt is boiled with 50 kilos. of sodium sulphite until a blackish-green solution is obtained, when, on cooling, most of the p-amidophenyl benzidine sulphonic acid separates out, the rest being precipitated by salt. The tetrazo compound, obtained in the usual manner, can be combined either with the same or with different components, or if such be taken that after combination, they can be further diazotised, trisazo dyestuffs are produced. Thus a dark black for cotton is obtained by combining the tetrazo compound from 37·8 kilos. of the sodium salt of p-amidophenyl benzidine sulphonic acid with 14·3 kilos. of α-naphthylamine, again diazotising with 7 kilos. of sodium nitrite and the necessary quantity of hydrochloric acid, and running the compound into a solution of 47·8 kilos. of γ-amidonaphthol sulphonic acid kept alkaline with sodium carbonate. The dyestuff, after separation in the usual way, dyes cotton a dark blackish-blue, and this product, on further diazotisation and combination with m-tolylene diamine, either in solution or on the fibre, gives dark black shades.—T. A. L.

Substantive Trisazo Colouring Matters [*Blues, Blacks*], *Production of.* W. E. Heys, Manchester. From The Chemical Works, formerly Sandoz, Basel, Switzerland. Eng. Pat. 28,810, Dec. 16, 1896.

By combining tetrazo derivatives with certain amines in equimolecular proportions, rediazotising the intermediate products, and combining these compounds with two molecular proportions of an azo compound, valuable trisazo dyestuffs have been obtained. The amines generally employed have been α-naphthylamine, Clève's α-naphthylamine sulphonic acids, and certain amidonaphthol sulphonic acids. It has not, however, been possible hitherto to employ amines of the benzene series, since experiment has shown that the compounds formed were not amido-azo derivatives, but diazo-amido compounds, and incapable of further diazotisation. According to the present specification, p-xylidine $[(CH_3)_2:NH_2=1:4:2]$ is an exception to this rule, and gives a readily diazotisable intermediate product. By combining this diazotised derivative, for example, with two molecular proportions of amidonaphthol sulphonic acid H, dyestuffs are obtained which dye unmordanted cotton indigo to greenish-blue. These products are diazotisable on the fibre, and, after development with β-naphthol, give deep black shades fast to washing. The following are the quantities employed:—A solution of 6·1 kilos. of p-xylidine and 6 kilos. of hydrochloric acid (21° B.) in 80 litres of water, is added to the clear tetrazo solution from 9·2 kilos. of benzidine at about 5° C. and stirred for about two hours. The intermediate product separates as a brown precipitate, and in order to complete the reaction, 26 kilos. of sodium acetate dissolved in water are added. After standing 10 hours at 5° C., 25 kilos. of hydrochloric acid are run in, and the mixture is diazotised by gradually adding a solution containing 3·5 kilos. of sodium nitrite. The brownish-black precipitate dissolves to a reddish-yellow solution, which is then run into 600 litres of water containing 34·5 kilos. of amidonaphthol disulphonic acid H, 4·5 kilos. of caustic soda, and 40 kilos. of sodium carbonate. After standing 12 hours, the mixture is heated to boiling and the dyestuff is salted out, filter-pressed, and dried. It dyes unmordanted cotton indigo-blue shades from a bath containing salt, and when diazotised on the fibre and combined with β-naphthol it gives a deep black, with m-phenylene or m-tolylene diamine a greenish-black, and with resorcinol a dark green.—T. A. L.

Violet and Blue Colouring Matters of the Triphenylmethane Group, Production of. W. E. Heys, Manchester. From The Chemical Works, formerly Sandoz, Basel, Switzerland. Eng. Pat. 30,015, Dec. 30, 1896.

These dyestuffs are obtained by condensing tertiary derivatives of β-naphthylamine—such as methyl- or ethyl-phenyl-β-naphthylamine, methyl- or ethyl-tolyl-β-naphthylamine, methyl- or ethyl-xylyl-β-naphthylamine; or secondary derivatives, such as α-naphthyl-β-naphthylamine or α-β-dinaphthylamine—with tetramethyl- or tetra-ethyl-diamidobenzophenone by means of phosphorus oxychloride. The products formed, since they contain a β-naphthyl group, can be readily sulphonated, and yield compounds which give pure shades fast to alkalis. The tertiary derivatives are obtained by alkylating phenyl-β-naphthylamine by the ordinary methods, and methyl-phenyl-β-naphthylamine forms large colourless prisms melting at 52° C., whilst ethyl-phenyl-β-naphthylamine melts at 55° C. and crystallises in white plates. The following example illustrates the method employed for the production of the dyestuffs:—A mixture of 19 kilos. of methyl-phenyl-β-naphthylamine, 20 kilos. of tetramethyldiamidobenzophenone, and 7 kilos. of toluene is agitated in a closed vessel on a water-bath and cooled to 30° C., when 13·5 kilos. of phosphorus oxychloride are added. In about 15 minutes a reaction sets in, and the temperature rises to about 120° C. After about an hour, the melt is cooled, powdered, and dissolved in 1,000 litres of hot water, and salted out. It can be used directly for sulphonation, or may be purified by washing with toluene or redissolving in slightly acid water and reprecipitating. It forms a greenish crystalline mass, which dyes wool a pure violet. In order to sulphonate it, 20 kilos. of the product are dissolved in 60 kilos. of 100 per cent. sulphuric acid, to which 40 kilos. of fuming sulphuric acid (54 per cent. SO_3) are gradually added at 20°—30° C. When a sample dissolves in a large quantity of hot dilute caustic soda, the melt is poured on to ice and the dyestuff is filtered off. In order to purify it, it is dissolved in 1,000 litres of hot water containing the necessary quantity of caustic soda, filtered, and precipitated with salt and acetic acid. The product dyes wool a pure violet similar in shade to "Acid Violet 7 B," but has the advantage in being fast to alkalis. By condensing tetramethyldiamidobenzophenone with α-β-dinaphthylamine and sulphonating the product, a dyestuff is obtained which gives blue shades on wool from an acid bath.—T. A. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

Silk: The Effects of Weighting on the Fibre. R. Gnehm and E. Bänziger. Färber Zeit. 8, [1], 1—2, and [2], 18—22.

By the tin phosphate-silicate process, the weight of silk may be increased 50—60, and even 100—120, per cent. without deteriorating the fibre, whilst feel and lustre are actually improved. It has been found, however, that silk thus treated has, in many instances, become tender after having been stored for some time, without any cause for this change being assignable. This circumstance has led the authors to study the effects on the fibre of the above method of weighting silk at different stages of the process.

In practice, this process is carried out as follows. The silk is steeped for one hour in a solution of stannic chloride of 25°—30° B., squeezed to remove surplus liquor, rinsed, and steeped for ½—1 hour in a warm solution of a soluble phosphate of 3°—5° B., and is again rinsed. The silk thus prepared is treated for ½—1 hour in a warm bath of a soluble silicate, and washed. The silicic acid is the chief constituent of the weighting, and the object of the process is to precipitate in the fibre a mixture of silicate and phosphate of tin along with free silicic acid, whilst by the former alkaline method, stannic oxide only was deposited. By five successive passages through the stannic chloride, phosphate, and silicate baths respectively, the silk may be increased in weight 100—120 per cent., as against 15—20 per cent. by the older process. The above order of treatment is sometimes varied. In some works the silk is not passed through the silicate bath after each phosphate bath, as this would give the silk a harsh feel, and tender the fibre.

The authors conducted their investigations as follows. After each treatment, the quantities of tin, phosphoric acid, and silica absorbed by the silk, were ascertained by an analysis of the ash, and the fibre was tested for strength and elasticity. The silks used in the experiments were organzine, Italian, 18/22 D, and tram, and Japanese, 40 D.

The results are given in the subjoined tables.

The "tensile strength" is expressed by the number of grms. required to break the thread. The "elasticity" is the increase in length before breaking, calculated for 1 metre of thread.

The numbers of "strength" and "elasticity" are the mean of 10 experiments. But as fibres which vary considerably (say, more than 15 per cent.) cannot be compared

A.—Italian Organzine.

Stage of Process.	Degree of Weighting in Per Cent. of Raw Silk.	Ash of Weighting, calculated on Silk with 6 per Cent. of Moisture.	Ash found for Silk with 6 per Cent. of Moisture.	Ash found, calculated for Dry Silk.	SnO_2 in Per Cent. of Dry Silk.	P_2O_5 in Per Cent. of Dry Silk.	SiO_2 in Per Cent. of Dry Silk.	Numbers of Strength per 1 Metre of Thread. Tensile Strength in Grms.	Elasticity in Mm.	Variation of Thread in Per Cent.
Raw	..	..	..	..	..	..	..	68	242	11·0
Ungummed	..	..	..	0·36	..	..	..	55	184	9·1
„ after 1 pinksalt	..	..	..	9·73	..	..	..	50	169	20·0
1. Sodium phosphate	..	..	..	11·52	8·35	2·02	..	63	188	8·0
2. Pinksalt	..	..	..	18·75	16·96	1·76	..	51	170	6·0
Silicate	10–15	25·4–28·77	22·3	23·65	14·45	1·66	6·75	69	195	8·7
2. Sodium phosphate	..	..	..	21·24	15·96	4·08	..	55	192	10·7
3. Pinksalt	..	..	..	28·18	23·72	3·00	..	48	169	16·7
Silicate	20–30	31·1–36·3	32·1	34·10	20·51	1·86	10·08	60	165	11·7
3P, 3Na, 1Si	20–30	31·1–36·3	32·8	35·45	19·24	6·72	5·68	63	178	14·3
From 4th pinksalt into silicate	30–50	36·3–40·7	40·1	42·54	25·90	5·80	8·18	56	166	10·7

Stage of Process.	After 40 Hours' Exposure to Heat at stated Temperatures. Tensile Strength. 40°	Tensile Strength. 55°–65°	Elasticity. 40°	Elasticity. 55°–65°	Variation of Thread. 40°	Variation of Thread. 55°–65°	After 8 Days' Exposure to Sunlight in June. Tensile Strength.	Elasticity.	Variation of Thread.
Raw	..	..	..	..	..	..	..	..	..
Ungummed	73	69	195	216	5·5	7·2	57	173	3·5
„ after 1 pinksalt	70	..	202	..	7·1	..	39	77	12·8
1. Sodium phosphate	70	..	190	..	8·6	..	62	137	8·1
2. Pinksalt	66	..	210	..	7·6	..	28	67	17·8
Silicate	81	64	203	127	5·0	12·5	38	75	10·5
2. Sodium phosphate	64	..	187	..	9·4	..	41	83	9·8
3. Pinksalt	65	..	172	..	7·7	..	33	65	15·1
Silicate	66	61	173	135	6·1	6·5	37	84	13·5
3P, 3Na, 1Si	59	68	177	187	8·4	14·7	24	47	16·6
From 4th pinksalt into silicate	63	54	210	130	11·1	7·4	31	48	22·6

B.—Japanese Filature Tram.

Stage of Process.	Degree of Weighting in Per Cent. of Raw Silk.	Ash of Weighting, calculated on Silk with 6 per Cent. of Moisture.	Ash found for Silk with 6 per Cent. of Moisture.	Ash found, calculated for Dry Silk.	SnO_2 in Per Cent. of Dry Silk.	P_2O_5 in Per Cent. of Dry Silk.	SiO_2 in Per Cent. of Dry Silk.	Numbers of Strength per 1 Metre of Thread. Tensile Strength in Grms.	Elasticity in Mm.	Variation of Thread in Per Cent.
Raw	..	..	..	..	..	..	..	142	255	7·0
Ungummed	..	..	..	0·73	..	..	..	134	213	5·2
„ after 1 pinksalt	..	..	..	9·70	..	..	..	124	219	9·0
1. Phosphate bath	..	..	..	10·84	8·07	1·81	..	138	217	4·3
2. Pinksalt	..	..	.	18·72	14·86	1·15	..	113	191	5·3
2. Phosphate	..	..	..	21·73	15·43	4·12	..	133	198	6·0
3. Pinksalt	..	..	..	27·57	24·42	2·67	..	124	183	9·7
Silicate bath	20–30 or 30–40.	27·3–36·9	33·00	35·00	19·56	2·16	11·06	132	204	2·5
3. Phosphate	..	..	..	31·23	23·22	5·10	..	124	195	3·2
4. Pinksalt	..	..	..	38·22	33·85	4·29	..	121	191	5·0
Silicate	50–60	40·7–43·9	40·40	42·84	29·44	3·12	7·12	139	202	5·7
4. Phosphate	..	..	..	38·50	28·53	6·23	..	142	209	2·1
Silicate	..	..	40·90	43·37	27·49	4·11	7·58	134	208	3·0
5. Pinksalt	..	..	..	43·15	36·62	4·94	..	126	179	7·1
Silicate	70–80	45·5–49·4	48·30	51·23	35·02	3·55	11·84	132	172	10·6
5. Phosphate	..	..	..	47·18	40·00	6·70	..	125	187	17·6
6. Pinksalt	..	..	..	50·07	43·37	6·78	..	136	193	5·1
Silicate after 5th phosphate (5P, 5Na, 1Si).*	80–100	49·4–53·5	45·70	42·44	..	..	1·66	134	187	6·4
Silicate after 6th pinksalt (6P, 5Na, 1Si).	100–130	53·5–58·2	51·29	54·33	..	..	..	109	151	6·7
From 2 silicate after 6 pinksalt (6P, 4Na, 2Si).†	120–150	57·3–61·6	51·12	54·33	..	..	..	118	147	5·1
A trial batch from Crefeld (4Na, 5P, 2Si).‡	100	53·5	48·00	50·81	..	..	..	110	146	8·2

* Instead of the usual course, silicate after pinksalt.
† Order of baths.—1P, 1Na, 2P, 2Na, 3P, 3Na, 4P, 4Na, 5P, 1Si, 6P, 2Si.
‡ Order of baths.—1P, 1Na, 2P, 2Na, 3P, 3Na, 4P, 1Si, 5P, 2Si.

B.—Japanese Filature Tram—continued.

Stage of Process.	After 10 Hours' Exposure to Heat at stated Temperatures.						After 8 Days' Exposure to Sunlight in June.		
	Tensile Strength.		Elasticity.		Variation of Thread.		Tensile Strength.	Elasticity.	Variation of Thread.
	40°	55°—65°	40°	55°—65°	40°	55°—65°			
Raw	..	..	..	..	..	..	..	..	..
Ungummed	123	138	245	205	4·1	6·5	127	184	13·4
„ after 1 pinksalt	132	..	211	..	6·8	..	99	114	11·1
1. Phosphate bath	127	..	210	..	3·1	..	110	154	12·7
2. Pinksalt	131	..	179	..	8·4	..	65	63	10·8
2. Phosphate	131	..	182	..	3·8	..	70	82	12·8
3. Pinksalt	118	..	167	..	7·6	..	69	68	15·9
Silicate bath	112	110	190	144	7·1	10·9	79	72	20·2
3. Phosphate	130	..	189	..	5·4	..	90	91	10·0
4. Pinksalt	131	..	195	..	13·7	..	44	49	40·9
Silicate	132	130	199	175	6·8	6·1	50	55	26·0
4. Phosphate	102	..	205	..	5·0	..	70	54	15·7
Silicate	136	131	230	186	7·3	4·6	43	61	16·3
5. Pinksalt	127	..	191	..	4·0	..	47	51	25·5
Silicate	135	110	186	152	11·1	4·5	40	43	42·5
5. Phosphate	134	..	163	..	3·7	..	59	42	20·2
6. Pinksalt	108	..	195	..	8·3	..	Too brittle for testing.		
Silicate after 5th phosphate (5P, 5Na, 1Si).*	121	129	165	165	12·4	3·9	43	41	32·6
Silicate after 6th pinksalt (6P, 5Na, 1Si).	105	100	163	116	9·5	12·0	28	51	21·4
From 2 silicate after 6 pinksalt (6P, 4Na, 2Si).†	128	116	117	100	3·9	5·2	52	44	36·5
A trial batch from Crefeld (4Na, 5P, 2Si).‡	142	121	176	118	3 5	8·3	33	45	27·3

* Instead of the usual course, silicate after pinksalt.
† Order of baths.—1P, 1Na, 2P, 2Na, 3P, 3Na, 4P, 4Na, 5P, 1Si, 6P, 2Si.
‡ Order of baths.—1P, 1Na, 2P, 2Na, 3P, 3Na, 4P, 1Si, 5P, 2Si.

with each other, the "variation of fibre" is given in a separate column. These numbers are the difference between the mean of 10 trials and the mean of the numbers below the first mean, expressed in per cent. of the former. Thus, let the mean of 10 trials be 134, and that of the lower numbers 127; then 134:(134—127) = 100:X; or = 5·2.

P, Na, and Si in the tables stand for "pinksalt" (stannic chloride), sodium phosphate, and silicate baths respectively; and the numerals before each, indicate the number of passages of the silk through each bath.

Conclusions:—1. The results obtained show a considerable excess of SnO_2 in the fibre—that is, more than would be required to form neutral salts. Thus, in organzine silk which has been passed once through a pinksalt and once through a sodium phosphate bath, a compound corresponding to the formula $4SnO_2.P_2O_5$ seems to have been formed.

2. As the weighting progresses, so the elasticity decreases; temporarily, however, especially after a passage through the silicate bath, there is a slight recovery.

The tensile strength is not materially affected.

3. The heating of even heavily weighted silk for 40 hours at a temperature of 40° C. has no appreciable influence on the strength of the fibre. But if this temperature be increased (say to 55°—65° for 40 hours), then the elasticity, and partly also the tensile strength, seem to suffer diminution.

4. Direct sunlight has a fatal influence. The strength of fibres exposed to light fell to one half and more below that of samples which had not been exposed; and in one case, where the silk had been passed six times through the pinksalt, the fibre was so brittle that its strength could not be ascertained at all. Under the microscope this silk exhibited sharp cross cuts, and on the fibre small nodules were deposited.

As it is impossible to expose hanks evenly to light, the effects of the latter on the different fibres must vary accordingly, and this is shown to have been the case by the numbers of "variation."

The observation is also of interest that the strength and elasticity of the fibre, which have suffered from a passage through the pinksalt solution, are again slightly recovered after a passage of the silk through a phosphate or a silicate bath.

The authors have examined several samples of dyed silk, weighted, and the results were confirmatory of the above conclusions, *viz.*, that heating to 40° C. has no injurious effect, but that light weakens the fibre considerably.

The following table shows the influence of light after an exposure of 18 days, chiefly during rainy weather, for 14 days without any direct sunlight, and a few hours of sunshine only on the remaining four days. The numbers in the first column indicate the degrees of weighting, whilst the letters *a* and *b* signify different modifications of the process.

Colour and Degree of Weighting.	Before Exposure.			After Exposure.		
	Tensile Strength.	Elasticity.	Variation.	Tensile Strength.	Elasticity.	Variation.
			Per Cent.			Per Cent.
Organzine, light green, *a* 30 50	59	177	16·8	45	140	18·6
„ brown, *a* 30 50	51	132	25·4	51	132	6·8
Tram, red, *b* 30 50	83	184	14·4	51	106	6·2
„ yellow, *b* 30 50	108	185	9·7	91	186	10·9
„ red, yellowish, *b* 60 80	95	111	15·7	70	85	14·2
„ „ bluish, *b* 60 80	76	135	10·5	62	69	12·9
„ white, *a* 80 100	181	138	13·2	145	91	13·1
„ yellowish white, *a* 80 100	150	133	11·4	140	122	11·6
„ brown, *b* 80 100	125	120	8·0	119	90	6·5
„ blue, *b* 80 100	107	126	7·4	122	85	8·5
„ orange, *a* 111 *p*	75	169	20·0	69	150	20·0
„ green, *a* 112 *p*	71	117	17·6	73	91	4·0
„ ponceau, *a* 118 *p*	88	151	17·6	95	90	13·1

—I. S.

PATENTS.

Fibrous Material [Eucalyptus Fibre], The Manufacture of an Improved. J. A. Maquaire, Paris. Eng. Pat. 3533, Feb. 17, 1896.

The material is obtained by suitable treatment of the fibres of the eucalyptus. These fibres are subjected to successive treatment with warm water, alkali, and acid, after which they are washed with resin soap and then with water. The fibres are then dried, scutched, and stripped, and carded to form "eucalyptus wool or wadding." The resins removed during the above treatment are restored to the wadding by slow fumigation in a cylinder.

The material is stated to be applicable to the manufacture of felt, and also capable of being spun either alone or together with other textile fibres of animal or vegetable origin.—R. B. B.

Dressing or Finishing Compound for Woven Fabrics, An Improved. E. R. L. Blumer, jun., Zwickau, Germany. Eng. Pat. 28,670, Dec. 15, 1896.

The compound consists of magnesium chloride, borax, starch, and rye flour. The borax is added in order to neutralise any hydrochloric acid liberated by decomposition of the magnesium chloride, and the rye flour prevents the absorption of moisture.—R. B. B.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Gum for Thickening, A New. Ch. Gassmann. Färber Zeit. **8**, 2—4.

The author has made trials with various new gums, and found a good substitute for tragacanth, &c. in the new thickening material termed "Slepah" (Labiche, Mulhouse). The advantages of this gum are said to be that it is easily removed from the fibre by washing, leaving the latter soft, and in not tinting wool and silk yellow during steaming to the same extent as other gums commonly used in printing. A quantity of from 50—60 grms. of slepah boiled with 500—600 grms. of water, and diluted to 1 litre, yields a mucilage suitable for printing.—I. S.

Manganese Brown, Sulphocyanide Discharges on. F. Reisz (with report by C. Schœn). Bull. Soc. Ind. Mulhouse, 1896, 378—380.

The use of sulphocyanides as discharges for Manganese Brown has been described by Brand (Bull. Soc. Ind. Mulhouse, 1896, 79; this Journal, 1896, 449). The present author recommends for a red discharge, a printing colour consisting of 50 parts of thickening, 50 parts of ammonium sulphocyanide (400 grms. per litre), 10 parts of acetic acid 6 B., and 24 parts of alizarin (20 per cent paste); after printing with this colour, the cloth is steamed. In the case of chromium mordants, the author finds that (1) pure chromium sulphocyanide does not discharge the brown; (2) a mixture of 800 c.c. of a thickened saturated chromium sulphocyanide solution and 200 c.c. of hydrosulphocyanic acid (23·6 grms. per litre) has little effect in the cold, but on steaming discharges the brown and fixes on the fibre a green oxide of chromium; (3) a mixture of 660 c.c. of chromium sulphocyanide and 40 grms. of hydrosulphocyanic acid discharges even in the cold, and on steaming fixes a greenish-yellow oxide; (4) 800 c.c. of chromium sulphocyanide, together with 140 c.c. of sodium sulphite 40° B. and 60—70 c.c. of acetic acid of 6° B., discharge feebly in the cold and readily on steaming, with fixation of a dirty grey oxide. For a blue discharge 750 c.c. of thickened chromium sulphocyanide, 100 c.c. of Alizarin Blue S, and 150 c.c. of sodium bisulphite of 40° B., may be used. The brown in this case becomes a little paler, probably owing to excess of sulphurous acid.

C. Schœn confirms the author's statements, and remarks that for a clear bright red some hydrosulphocyanic acid should be added to the above-described alizarin and alu-minium colour. In all cases the brown tends to be partially reduced on steaming, and this may easily lead to uneven colours.—R. B. B.

Turkey-Red, An Impt. in the Dyeing of. T. Baldensberger. Bull. Soc. Ind. Mulhouse, 1896, 382—384.

In order to avoid the chief defects of Turkey-red dyeing, *viz.*, the necessity of frequently cleaning the dye-baths to remove grease, uneven dyeing (when dyed on the winch), and the great length of time required (when dyed in the jigger), the author recommends the use of an alkaline alizarin solution, prepared by dissolving 10 kilos. of alizarin (20 per cent. paste) in 40 litres of water containing 1 kilo. of ammonia. The pieces are dyed in the jigger; they are first passed through cold water containing a calcium salt, more water is then added, the bath heated to 50° C., and the alizarin solution added in two, three, or four portions according to the length of the piece. The bath is raised to the boil in half an hour, and boiled for 15 minutes; after 10 passages the bath is almost completely exhausted; the pieces are then washed and cleared in the ordinary manner. It is stated that the red is superior in brilliancy to that obtained without the use of ammonia, that the colour is perfectly even, and that one hour suffices for the dyeing of three pieces, $1\frac{1}{8}$ metres in width, by this method, while three hours would be required for the dyeing in the ordinary winch machine.—R. B. B.

Aniline Black [Oxidation] from Aniline Fluoride. H. Lindner. Leipziger Färber u. Zeugdr. Zeit. 1897, **46**, [2].

Aniline fluoride, the author states, has the advantage over aniline chloride of not attacking the vegetable fibre to the same extent as the latter, whilst yielding a full and good black. Its only drawbacks are that the black dyed with it has not quite the desired lustre, and that its price is somewhat higher than that of the chloride.

Aniline fluoride being much more neutral than the chloride, it requires a somewhat larger proportion of oxidising agents, such as sodium chlorate and copper nitrate, and aluminium chloride is to be substituted for the corresponding acetate. If the right proportions of the several ingredients are taken, the oxidation proceeds as evenly as with aniline chloride.

The author has obtained good results by working as follows:—5,400 grms. of aniline fluoride are stirred up with 10 litres of tepid water. In another vessel are dissolved 900 grms. of sodium chlorate in 4 litres of water, and in a third vessel 450 grms. of ammonium chloride in 4 litres of water. When cold, these three solutions are mixed together, and to them are added 3 litres of copper nitrate and 100 grms. of aluminium chloride, and the whole is made up to 30 litres.

The copper nitrate is prepared as follows:—To a solution of 900 grms. of copper sulphate in 3 litres of boiling water are gradually added, whilst stirring, 900 grms. of soda crystals. After the precipitate has settled, the supernatant liquid is decanted, and 400—500 grms. of nitric acid are added to the residue until the latter is dissolved.

The aluminium chloride is prepared by dissolving 1,000 grms. of alum and 600 grms. of barium chloride in 4 litres of boiling water, and allowing the precipitate to settle. The clear solution is used.—I. S.

PATENTS.

Rapid Tanning of Hides, Process and Apparatus for the; applicable also for Dyeing and otherwise Treating Leather. H. L. J. Roy, Paris, France. Eng. Pat. 2572, Feb. 4, 1896.

See under XIV., *page* 250.

Dyeing, Bleaching, or Scouring Fibrous Materials, Impts. in Apparatus for. T. Halliwell, Eccles. Eng. Pat. 2840, Feb. 7, 1896.

The apparatus described, consists of two closed vats connected at the bottom by a pipe, and two condensing chambers placed above these vats. The condensing chambers are connected with an injector, by means of which, air or steam

may be exhausted from either of the vats. A vacuum is produced in each vat alternately, and the liquor thus caused to pass from one vat to the other, this movement of the liquor being automatically reversed, when a certain level is reached, by means of a float and slide valve. In this way the liquor is repeatedly drawn through the cops, or other material in the vats, in alternate directions.—R. B. B.

Cutch, Impts. in preparing, for Sale and for Use. J. R. Carruthers, Tradeston, Glasgow. Eng. Pat. 3178, Feb. 12, 1896.

Any ordinary form of cutch is subjected to the action of hot or dry air in a centrifugal machine or otherwise, and is then reduced to powder. The powder is next granulated by treatment with steam, water, or other suitable liquid, and by the action of mixing and drying apparatus.

In another modification of the patentee's process, the original cutch is made into a paste with water, the paste dried and granulated as before, or spread into sheets, dried, broken up, and sieved into powders of various grain.

By any of the above means, a product similar in colour and appearance to the original cutch is obtained, but one which, in that it is more easily handled, dissolved, and measured, is more useful, technically and commercially.
—E. R. B.

Fast Colours on Animal Fibre, The Production of, by the Aid of Oxide of Titanium. J. Barnes, Accrington. Eng. Pat. 5712, March 14, 1896.

Oxide of titanium (TiO_2) in a more or less hydrated condition may be fixed upon the wool fibre by boiling in a solution containing titanium tetrachloride ($TiCl_4$) and cream of tartar, the mordant containing these two substances being previously prepared. It is claimed that titanium mordant yields bright and fast colours intermediate in shade between the colours produced with chromium and with aluminium mordants.

A titanium mordant may also be prepared by dissolving hydrated titanium oxide in oxalic acid; in this case the mordanting bath should not be heated above 160° F.

Silk may be mordanted by steeping for several hours in a cold solution of titanium tetrachloride at 10° Tw.

For further details, see this Journal, 1896, 420.—R. B. B.

Crape-like Designs on Cotton, Linen, and other Fabrics or Yarns of Vegetable Origin; A Chemical Process for the Production of White or Coloured. J. Weiss, Heidenheim-on-Brenz, Germany. Eng. Pat. 8235, April 18, 1896.

In this modification of the mercerising process, the fabric is impregnated with caustic alkali and *immediately* printed with acids, salts, or metallic oxides soluble in alkali, which neutralise the caustic alkali in the printed parts and thereby prevent the contractile action from taking place in those portions of the fabric. By varying the constituents of the printing mixture, a great variety of coloured crépon effects may be obtained. For instance, chromic acetate produces green chromic oxide; salts of aluminium and tin are decomposed by the alkali, and the oxides of those metals, thus fixed upon the fibre, act as mordants for alizarin or other dyestuffs. Ferric chloride yields ferric oxide, which can be combined with potassium ferrocyanide to form Prussian blue. If the printing mixture be composed of tannin and acetic acid, the printed portions may be dyed with a basic colouring matter. or if the fabric be impregnated with β-naphthol, dried, mercerised, and printed at once with diazo solutions containing acetic acid, the printed portions are coloured with the resulting azo compound.

By analogous methods, white crape-like effects may be produced upon a coloured ground.—R. B. B.

VII.—ACIDS, ALKALIS, AND SALTS.

Sulphide of Sodium, Electrolytic Oxidation of. Scheurer-Kestner. Bull. Soc. Chim. **17**, [2], 1897, 99.

A paper has recently been published by Durkee on the oxidation of sulphide and sulphydrate of sodium by electrolysis. This author finds that, whereas oxidation by air or gaseous oxygen gives rise to hyposulphite (thiosulphate), oxidation by means of the electric current causes the formation of sulphate. He further states that during the process, sulphur is precipitated, and hyposulphite (thiosulphate) can be detected in the solution; that is, the oxidation of the sulphide to hyposulphite occurs as an intermediate step in the formation of the sulphate. Scheurer-Kestner points out that the electrolytic oxidation of sodium sulphide was patented in France in 1875, and that in 1881 he published a note on the reaction. Working with sulphide only, and not hydrosulphide, he failed to note at any time either deposition of sulphur or formation of thiosulphate; and from his observations that the electrolysis always occurred with an unavoidable evolution of oxygen, and that it required the same time to oxidise by electrolysis, solutions containing the same quantity of sulphur, whether the sulphur was present as sulphide or as thiosulphate, he comes to the conclusion that it is inadmissible to consider an intermediate oxidation to thiosulphate as occurring, and that the oxidation proceeds directly to sulphate, thus: $Na_2S + O_4 = Na_2SO_4$.
—J. T. C.

Sulphur in Pyrites, Determination of, by means of Sodium Peroxide. C. Glaser. Chem. Zeit. 1897, 40.

See under XXIII., *page* 262.

Caustic Alkali, Estimation of, in Presence of Alkaline Carbonates. G. Lunge. Zeits. f. angew. Chem. 1897, [2], 41.

See under XXIII., *page* 262.

Persulphates, Analysis of. F. Ulzer. Zeits. f. angew. Chem. 1897, [2], 61.

See under XXIII., *page* 262.

PATENTS.

Carbonic Acid, Impts. in means for Obtaining, from Gaseous Mixtures. W. Raydt, Stuttgart, Germany. Eng. Pat. 873, Jan. 13, 1896.

This is an improvement on Eng. Pat. 13,456, 1895 (this Journal, 1896, 594), in which a method is described of obtaining carbonic acid from furnace and other mixed gases by bringing these into contact with moistened alkali carbonate, and heating the resulting bicarbonate. According to the present improvements, instead of moistening the carbonate to be acted upon, the gases, heated to 70° C., are led through layers of asbestos, small coke, or other suitable absorbent material, kept moist by a spray of distilled water, ordinary water being unsuitable. The gases thus moistened then permeate layers of the carbonate ranged intermediately, within a chamber divided into compartments by a series of heating pipes communicating by external chambers. The apparatus may be arranged for passage of the entering gases either upwards or downwards, but in such manner that the gases only reach the layers of carbonate after they are moistened by traversing the wet absorbent. The apparatus used in the previous process, may, with certain modifications such as indicated, be made available.—E. S.

Ammonium Nitrate and other Salts, Impts. in the Manufacture and Purification of. T. Fairley, Leeds, Yorks. Eng. Pat. 1667, Jan. 23, 1896.

132 parts of ammonium sulphate and 170 parts of sodium nitrate, slightly moistened if necessary, are heated, with constant stirring, until a temperature of 108°—110° C. is attained, and the partially fused mass is maintained at this temperature for from 3 to 5 minutes. The product thus formed consists essentially of ammonium nitrate and sodium sulphate, but the reaction is not complete, and the patentee states that a similar product is obtained by heating 160 parts ammonium nitrate and 142 parts of anhydrous sodium sulphate to 110°.

The mass is extracted with boiling alcohol (methylated spirit containing 85 to 93 per cent. of real alcohol), to which is added ammonia corresponding to 0·5 per cent. of the alcohol. This ammonia, by preventing the formation of an acid liquid during extraction, enables a purer product to be obtained, and prevents corrosion of the metal portions of the extraction apparatus.

The alcoholic solution contains the ammonium nitrate admixed with about 10 per cent. of sodium nitrate, whilst the insoluble residue consists of sodium sulphate, together with about 8 per cent. of ammonium sulphate.

The mixed sodium and ammonium nitrates are, after evaporation and recovery of the alcohol, washed in an upright cylindrical vessel, with cold water or a cold saturated solution of ammonium nitrate, which dissolves out the more soluble sodium nitrate (see following abstract). The process may be made continuous; the moist purified ammonium nitrate discharged from the top of the percolator is, after drying, ready for use.

It contains less than 1 per cent. fixed or solid impurity. The liquid from the percolator is evaporated and the dry salts added to the next material treated; to these is also added the ammonium sulphate recovered from its admixture with sodium sulphate by treatment with boiling water.

—J. T. C.

Ammonium Nitrate, Impts. in the Manufacture of. T. Fairley, Leeds, Yorks. Eng. Pat. 1668, Jan. 23, 1896.

The object of the invention is to obtain a practically pure ammonium nitrate from the mixed nitrates of ammonium and sodium obtained when a solution of sodium nitrate is treated with ammonium bicarbonate or ammonia and carbon dioxide. The separation of the mixed nitrates depends on the fact that at low temperatures sodium nitrate is more soluble than the ammonium salt. The concentrated solution of the mixed nitrates is fed in at the bottom of a cylindrical vessel, externally cooled to about -15° C., and the ammonium nitrate delivered at the upper end, is purified by washing with cold water or with a cold saturated solution of ammonium nitrate. The liquid remaining from the cooling process is used to dissolve fresh quantities of sodium nitrate for treatment in the ammonia-soda process above mentioned, and the cooling of the solution of the mixed nitrates may be partially effected by saturating the solution obtained in one operation with the dried salts obtained on evaporating the solutions obtained in a previous operation. By this means much of the external refrigeration may be avoided.—J. T. C.

Silicates of Sodium and Potassium, Nitric and Sulphuric Acids, and Caustic Soda and Potash; Impts. in the Manufacture of. W. Garroway, Glasgow, N.B. Eng. Pat. 2489, Feb. 4, 1896.

From 1 to 2 parts of sand or ground flint is mixed with 1 part of sodium nitrate, or with an equivalent proportion of potassium nitrate, and the mixture is heated in an iron retort, which may be arranged over a pyrites burner when the process is worked in connection with a sulphuric acid plant. Superheated steam and hot air are admitted. The nitric acid evolved may be condensed, or taken to a Glover tower. The alkali silicate formed is lixiviated, and the liquid may be treated with lime to precipitate silica, leaving caustic soda or potash in solution. Any insoluble silicate resulting from the process may be applied in the manufacture of cement. The apparatus employed is shown in connection with a sulphuric acid plant.—E. S.

Carbonic Acid Gas passing from Lime Kilns and designed for Liquefaction, A New or Improved Process for the Purification of. A. D. Delsemme, Liège, Belgium. Eng. Pat. 3662, Feb. 18, 1896.

Lime-kiln gases are pumped into high towers, down which streams of water descend, and are then passed into close receivers containing potash lye, which, when saturated with carbonic acid, is heated to recover the gas. The moist carbonic acid is then taken through a refrigerating apparatus to a gasometer, and, before being liquefied in a compressor, is dried by contact with calcium chloride.—E. S.

Sulphate of Ammonia, Impts. in or relating to Saturators for Use in the Manufacture of. P. Duggan, Rosherville, Kent. Eng. Pat. 5011, March 5, 1896.

The upper portion of the saturating vessel is preferably cylindrical in shape, but the lower part is made conical or curved, so that the sulphate as it forms, falls towards an outlet situated at the vessel's lowest point, and from this passes into a discharge pipe.

The saturator is fitted with a cover made in parts, hinged together, so that workmen may readily get inside when necessary. Through this cover pass pipes for the admission of the ammonia gas and sulphuric acid, and an outlet tube is provided for gases or vapours generated in the saturator.

The ammonium sulphate formed, either falls directly into the discharge pipe, through which it is driven by means of an injector and delivered into the drainer, or is discharged by the action of gravity alone. In the latter case the quantity passing out is regulated by a valve or plug worked from the top of the vessel and fitting into the outlet at the bottom of the saturator.

If an injector be employed, the author claims the arrangement by which this injector is applied externally to the vessel forming the saturator.—J. T. C.

Cyanides, Improved Means for Recovering, from Waste Products in Gas, Chemical, and Iron Works. G. P. Lewis, Catford, Kent, and F. S. Cripps, London. Eng. Pat. 5184, March 7, 1896.

Gas liquor, from which ammonia has been distilled off after addition of lime or an alkali, is run into a cooling tank, and mixed with a solution of sulphurous acid or ferrous sulphate. The mixture is then caused to overflow through an interposed strainer into another tank, in which it is treated with a solution of a copper salt to precipitate cuprous sulphocyanide. To obtain ammonium sulphocyanide from this precipitate, it is treated with the crude ammonium sulphide liquor produced on cooling the waste gases arising from the distillation of the gas liquor, after absorption of the ammonia from them. The copper sulphide resulting, is dissolved to obtain a copper solution for re-use. Potassium or sodium sulphocyanide is produced by using, instead of the ammonium salt, an alkali sulphide obtained by passing the waste gases referred to, through a caustic alkali solution. Sulphurous acid for use in the process, is produced by burning the waste gases in suitable apparatus.

The process may be applied to a liquor obtained from sulphur-freed spent iron oxide, by boiling it with lime. The solution, which contains calcium ferrocyanide and sulphocyanide, is treated, first with sulphurous acid, and then with a mixture of ferrous and ferric salts. Prussian blue is thrown down. The decanted liquor is then treated to obtain cuprous sulphocyanide as in the process first described.—E. S.

Nitrate of Ammonium, Impts. in the Manufacture of, and in the Apparatus therefor. G. Craig, Glasgow, Scotland. Eng. Pat. 5815, March 16, 1896.

Mixtures of nitrate of potash or soda with ammonium sulphate are heated together with sufficient water to ensure thorough double decomposition.

The resulting mixture is dried, and the hot, pasty mass well agitated during cooling, so that a loose powder is obtained. From the cold powder, ammonium nitrate is obtained in pure condition by extraction with anhydrous or high-strength ammonia. Owing to the high pressure exerted by the ammonia, strong vessels must be employed, and, to avoid corrosive action by the ammoniacal ammonium nitrate solution, the vessels should be of enamelled iron, or tinned or aluminium-lined iron.

An apparatus is described consisting of extraction vessels and filter and a filtrate-receiving vessel for the ammoniacal nitrate solution. By means of a pump in connection with this latter vessel, the ammonia is recovered by suction, and after compression through a condenser, whereby it is liquified, it passes again to the extraction apparatus to do fresh work.

To obtain a pure product, the temperature must be low and the ammonia in such excess that it takes up the ammonium nitrate to form a solution containing from 50—60 per cent. by weight of this latter substance.

The cold produced in the filtrate vessel may be utilised in the condensation of the ammonia.

The nitrate of ammonium is removed as a white powder through a manhole in the bottom of the filtrate vessel.

—J. T. C

Aluminate of Barium and Aluminate of Strontium, Impts. in the Manufacture of; and the Production of Alumina, Sulphate of Barium, and of Strontium, and other Products from the same. D. A. Péniakoff, Huy, Belgium. Eng. Pat. 6096, March 18, 1896.

To obtain barium aluminate, a mixture of barium sulphate, alumina or bauxite, and a metallic sulphide or polysulphide (as barium sulphide or pyrites), is heated, and the sulphurous acid evolved is collected and utilised in lead chambers or otherwise. Formulæ are given to show the proportions of the materials to be used in each case. When carbon is employed in the production of the aluminate, the proportions should be so adjusted as to prevent the formation of carbonic oxide, as according to the equation—

$$2(4Al_2O_3 + Fe_2O_3) + 8BaSO_4 + 5C =$$
$$8Al_2O_3BaO + 4FeO + 5CO_2 + 8SO_2$$

in which $4Al_2O_3 + Fe_2O_3$ is taken as representing bauxite.

The crude barium aluminate obtained, is treated with a solution of an alkali sulphate or acid sulphate, to obtain a solution of caustic alkali and a residue of barium sulphate, for re-use. Or the aluminate may be lixiviated, and the solution precipitated by the alkali sulphate, but in this case, some alumina remains combined with the alkali. Or the barium aluminate solution may be converted into hydroxide by stirring in presence of "crystallised hydrate of alumina," or by vigorous agitation without admixture. All the reactions remain unaltered when barium compounds are replaced by those of strontium.—E. S.

Carbonic Acid Gas, Improved Process and Apparatus for the Manufacture of. B. T. L. Thomson, London. Eng. Pat. 6835, March 28, 1896.

Steam is passed through a heated cylinder, charged with carbon, and the mixture of carbonic oxide gas and hydrogen produced is, after passage through a cleansing vessel, sent into one of a pair of heated cylinders containing copper oxide, or other oxide, capable of acting similarly, where the carbonic oxide is converted into carbonic acid and the hydrogen into steam. The mixture of carbonic acid gas and vapour is then passed into a cooler or washer, whence the gas passes to a reservoir. A pair of oxidising cylinders is provided, so that the reduced oxide in one may be reoxidised by pumping air through it, while the other is being used in the reverse process. The upright cylinders set in fireplaces, with accessory apparatus of pumps, four-way cocks, entrance and exit tubes, &c., are shown.
—E. S.

Disinfecting, Deodorising, and Bleaching Agents [Hypochlorites produced by Electricity], Impts. in the Manufacture of, and in Apparatus therefor. M. Crawford, London. Eng. Pat. 14,852, July 4, 1896.

In electrolysing sea-water or other saline solution for the production of hypochlorites, it is proposed to obtain greater efficiency by separating the electrodes by porous divisions, agitating the electrolyte, and keeping down the temperature by cooling agents.—J. C. R.

Crude Acetic Acid, Process for Purifying. A. Schmidt, Cassel, Germany. Eng. Pat. 25,100, Nov. 9, 1896.

This process is for the removal of the empyreumatic constituents (tar, sulphurous acid, &c.) from crude acetic acid, and consists in treating the crude, warmed acid with oxygen under pressure.

The apparatus employed, consists of a high, strong-walled charcoal filter with acid-proof lining, fitted with the necessary pressure-regulating apparatus, and capable of being suitably heated. Near the top and bottom of the cylinder are perforated plates; the space between these is filled with charcoal or coke in pieces about the size of nuts.

The compressed oxygen is forced in at the bottom of the apparatus; the acetic acid is pumped in and distributed at the top. The tarry matter of the acid is oxidised to form insoluble resinous substances, which are retained by the charcoal in the filter, and the purified acid passes out at the bottom of the apparatus through a U tube sufficiently deep to correspond to the pressure employed. Any sulphurous acid present is oxidised to sulphuric acid, which passes out with the acetic acid. The latter acid is finally freed from this impurity by distillation over a suitable quantity of pure sodium acetate.—J. T. C.

Caustic Soda Solution, Impts. in the Preparation of, from Soda Residues. F. J. Seyfried, Mulheim-on-Rhine, Germany. Eng. Pat. 26,889, Nov. 26, 1896.

"Pure transparent solution of caustic soda" is obtained by treating the dirty-red solution of soda residues with hydrated lead oxide. But it is preferred to carry out the process by adding a lead solution, as of the nitrate, followed by addition of milk of lime. In practice, two opened drums of the soda residues are immersed in water boiled by steam, and a the decanted solution, brought to a strength of about 40 per cent., is forced by a steam jet into an elevated tank, and a solution of 1 kilo. of lead nitrate is added, and then a bucketful of milk of lime. After subsidence, the clear liquor is drawn off into one of two lower vessels.—E. S.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

The Acid Clays [Fireclays], Classification of. Kosmann. Thonind. Zeit. **20**, [68], 933.

The silicates of alumina (clays and fireclays) containing in the dry state not more than 36 per cent. of alumina—*e.g.*, $Al_2Si_3O_9$—belong to the acid class of clays, and, by reason of their fusibility, are of less value for firebrick than the silicates —*e.g.*, $Al_2O, 2SiO_3$, and Al_2O_2, SiO_3—richer in alumina. The last-named silicate is highly basic in character, and is on that account well adapted, either in its natural form as Andaluzite, or in the condition of hydrate (Allophane), for improving the composition of fireclay, in place of bauxite. All the silicates with more than 36 per cent. of alumina are classed as basic, but in practice a higher percentage than 47 is scarcely attainable.—C. S.

Fireproof Bricks, Manufacture of. Diamant, **18**, [35], 755—757.

So far as resistance to fusion—one of the most important properties of a fireproof brick—is concerned, it is noteworthy that combinations and mechanical admixtures of alumina and silica, such as occur in natural clay, follow the general rule that compounds fuse at a lower temperature than the arithmetical mean of the fusing points of their components. Further, that although clays rich in silica take longer to sinter at the fusing temperature of cast steel than those poorer in this constituent, they fuse to a cake much sooner than the latter at higher temperatures. This, taken together with the behaviour of the clay in presence of fluxes, indicates that alumina is the chief factor in the production of a refractory material. That the composition of natural fireclays varies considerably is apparent from the analysis of Saarau and Niederpleiss clays, classified by Bischof as 100 and 10 respectively:—

Clay.	Al_2O_3	SiO_2 In Chemical Combination.	SiO_2 Free.	CaO.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Saarau..........	36·30	38·94	4·90	0·19
Niederpleiss....	28·05	30·71	27·61	0·72

Clay.	Na_2O.	MgO.	Fe_2O_3.	Loss on Calcination.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Saarau..........	0·42	0·19	0·16	17·78
Niederpleiss....	1·39	0·75	1·89	8·66

Firebrick powder consists of burnt fireclay or fragments of fireproof bricks, &c., ground to powder. Fireclays of

low plasticity (such as that obtained at Rakonitz, in Bohemia) are burnt and mixed with raw plastic clays for working up into a good fireproof material. By this process excessive shrinkage is counteracted, the particles of burnt clay forming nuclei of contraction for the surrounding raw clay, and also giving up to the latter in drying the moisture they have previously absorbed, thereby preventing the formation of cracks and fissures in the mass.

The fineness of division of the burnt fireclay is regulated according to the effect desired, coarse angular granules imparting the properties of porosity and power of resistance to changes of temperature, whilst finer particles give cohesion and power of withstanding shock, as well as diminishing the conduction of heat.—C. S.

Slag Cement, Testing of, for Alteration of Volume, by the Austrian Method. Mitt. des k.k. Tech. Gewerbe-Museums in Wien, 1896, 317—320.

The tests usually applied to Portland cement in respect of fineness and binding power are equally available for slag cement, but not so the test for constancy of volume, because the latter material shows a far greater tendency to form shrinkage cracks than does any other hydraulic binding medium. In applying the test to Portland cement, the cake of cement is allowed to remain in moist air for 24 hours after its preparation, and is then heated. In this way much of the uncombined water contained in the mass, and necessary to the final hardening, is expelled; and this favours shrinkage. At the same time the cake tends to develop cracks at the edges, owing to expansion under the action of heat; these cracks are wrongly termed shrinkage-cracks, which latter would always be either in the form of concentric circles or of superficial hair-cracks, and never at the edges. The distortion and crumbling that usually accompany the formation of edge-cracks in the case of Portland cement are absent when the slag material is tested; and this fact renders it impossible to rely upon the evidence of the heat test alone in determining the constancy of volume of the latter. Hence the Austrian method of conducting this test in air is not admissible for slag cements. The steam-heating test is to be recommended for the material in question, owing to the presence of vapour throughout the operation. In testing a Portland cement that is to be used for mortar, the ordinary test, without admixture with sand, gives a satisfactory indication of fitness. But in slag cement, lime is mixed with the finely-divided slag, and if the lime be not sufficiently slaked first, or if it be over-burnt, so that it does not slake until the cement has begun to set, the expansion will cause cracks, owing to the smallness of the space between the fine particles of the material. But if sand were present (as in mortar), the interstitial spaces would be larger, and the expansion might take place without danger. A second test should therefore be applied, using a normal mortar mixture of 1:3 in order to obtain a fair reading.—W. G. M.

PATENTS.

Marble-like Stucco or Plaster-Work suitable for Walls, Ceilings, and the like; Impts. in the Production of, and in Cement Mixtures therefor. M. Hoeft, Berlin. Eng. Pat. 24,967, Dec. 30, 1895.

Stucco or plaster-work with a glossy surface is produced by smoothly spreading upon a plaster ground or backing, a cement consisting of an intimate mixture of pure albumin (albumin of eggs or of serum) in aqueous or alkaline solution, burnt lime, and a "lean" or "poor" substance, such as finely divided natural or unburnt gypsum, over-burnt gypsum, marble, limestone, or sand, or a mixture of two or more of these substances, and coating the surface of the cement so applied, with a thin aqueous or alkaline solution of the cement.

The cement powder obtained by mixing the albumin in the dry state instead of in solution, in the above mixture, is also claimed.—A. S.

Cement or Artificial Stone, Impts. in the Manufacture of. F. Turner, Carnarvon. Eng. Pat. 2223, Jan. 30, 1896.

Powdered limestone and waste slate dust or stone chippings are mixed with water to form a "slurry," which is spread upon coke in a kiln for absorption of the moisture and rapid drying within the kiln before burning. The mixture may, when in the "slurry" state, be poured into moulds and pressed, for the production of blocks or slabs.
—A. S.

Marble and Similar Stone [Colouring], Impts. in the Treatment of. D. J. J. Froment, Brussels. Eng. Pat. 2451, Feb. 3, 1896.

A process for treating marble or other similar stones, in order to give them any coloured shades, veins, and spots, consisting in leaving these stones in one or more baths composed of a solution in alcohol or ether of one or more colours of aniline or other organic colouring materials. The colouring materials are fixed by leaving the coloured stone in a bath of oil or of any other fat stuff, or by applying upon the stone, layers of the same stuff. The absorption of the organic colouring materials and of the fat stuff by the stone, may be accelerated by heating or boiling the bath which contains the stone to be treated.—A. S.

Artificial Stone, Impts. in the Manufacture of. J. Grunhut, Leicester. Eng. Pat. 20,432, Sept. 15, 1896.

The manufacture of artificial stone by (1) mixing together in a dry state, sand, lime, and finely powdered sulphur, placing the mixture while still in that state, in suitable moulds, and then subjecting the mixture to the action of steam, or of sulphur vapour in combination with steam. (2) Mixing together in a dry state, sand, lime, and finely powdered sulphate of iron, copper, aluminium, or ammonium, or sulphide of sodium, placing the mixture, while still in that state, in suitable moulds, and then subjecting the mixture to the action of steam, or of steam combined with sulphate of iron, copper, aluminium, or ammonium, or sulphide of sodium.—A. S.

X.—METALLURGY.

Gold Ores, Refractory Low-Grade, Treatment of, at the Ouro Preto Mine, Brazil. S. J. McCormick. Inst. Mining and Met. 1897, 1—10.

In this mine the gold is found associated with iron and arsenical iron pyrites, and with bismuth sulphide. As sent to the mills the ore contains 1—2 per cent. of the former, and assays about $9\frac{3}{4}$ dwt. of gold to the ton. The crushing is effected by 92 head of stamps of various types, 4,300 tons of stuff being crushed monthly and the total cost of treatment ranging from 17*s.* to 22*s.* per ton. The free gold, mixed with pyrites, is caught on blankets placed in front of the mortar boxes; and this auriferous pyrites is further concentrated by passing it twice over blankets placed on a series of strakes. The rich product from these is washed in batéas by six women at 1*s.* *per diem*. In one of the mills (a 20-stamp mill, crushing 1,400 tons a month) the pulp from the blankets before the mills, is passed over a series of Frue vanners, which collect about 30 tons per month of concentrates, averaging 1 oz. $18\frac{1}{2}$ dwt. to 2 oz. $17\frac{3}{4}$ dwt. of gold per ton. Beyond the vanners are blankets which catch a further quantity of gold that has hitherto escaped, and the final tailings assay about $1\frac{1}{2}$ dwt. per ton. The vanner concentrates are chlorinated. In the two other mills (40 and 32 stamps) the first blankets are placed at the head of a series of revolvers, and after these are placed canvas strips, 3 ft. long, that collect a concentrate assaying 3 oz. $4\frac{1}{2}$ dwt., which is then chlorinated. The revolver tailings assay $3\frac{1}{4}$ dwt. and are passed over canvas-covered strakes, which remove another $\frac{3}{4}$ dwt. Owing, however, to the success of the system first described, the 40-stamp mill is now being supplied with vanners also.

In the chlorination works are two reverberatory calciners used alternately, with a cooling-floor, revolving barrel, leaching and precipitating tanks, and a turbine. 100 tons of concentrates are roasted monthly by six men, working in three shifts of two each. Two tons of roasted material are charged into a barrel with $17\frac{1}{2}$ lb. of chloride of lime, $28\frac{1}{2}$ lb. of sulphuric acid, and sufficient water to moisten the sand, and the mixture is revolved for five hours. The excess of chlorine is expelled from the gold solution by means of a small air-blower, and the gold is then precipitated

by ferrous sulphate. Cuprous sulphide was formerly employed as a precipitant, but its use has been discontinued, because it was found impossible to displace the whole of the copper by gold in the first precipitating barrel, and the separation of the gold from the regulus formed on melting, was troublesome. The treatment of concentrates assaying 2 oz. 11½ dwt., on an average, gives an extraction efficiency of 92 per cent. About 3 tons a month of a rich sand, assaying 32 oz., are roasted and chlorinated, and the extraction in this case is 95 per cent.

The cyanide process has also been tried both by agitation and by percolation. In the former case 12 cwt. of the unroasted vanner-concentrates were agitated in a barrel for 40 hours with 1,000 lb. of a 1·25 per cent. cyanide solution. The solution was used for four batches of concentrates, and was then passed through the zinc precipitating-boxes to the storage tank, as it still retained 0·3 per cent. KCN and a little gold. About 11 lb. of cyanide were consumed per ton of concentrates. The labour item is small, requiring only the service of the foreman of the mill aided by a small boy; and the working cost is smaller than that of chlorination, but the extraction is only 80—85 per cent. Fresh moist concentrates gave most unsatisfactory results (owing to the absence of air), the best work being done with the dry porous material obtained after draining for three days. In the percolation process, the concentrates were saturated with lime in a cement-lined brick tank, washed until the reaction was just alkaline, and treated with one-third their weight of 0·3 per cent. KCN solution. After draining through, the solution was pumped over the sand again, five or six times a day, and was made up to the normal strength every morning. After a month's treatment, the extraction of gold amounted to 85—90 per cent., and the consumption of cyanide was 13¼ lb. per ton of sand. But in the cyanide process the gold recovered does not agree with the extraction shown by assay, the discrepancy amounting to 1—2 per cent., owing to the failure of the zinc to extract the last traces of gold from the dilute wash-solutions. The cyanide process was only tried because of a threatened dearth of wood for roasting purposes. Experience has now shown that if any royalty had to be paid for the use of the process, it would be more economical to retain the chlorination process even though coal had to be imported from Wales. The actual cost of extracting the gold by chlorination from 100 tons of pyritic concentrates assaying 3·225 oz., is (per ton):—for labour 12·56*s.*, wood 11·2*s.*, chemicals 5·36*s.*, and repairs, 0·544*s.*; making 29·664*s.* per ton in all, and the value of the gold extracted is 12*l.* 11*s.* By cyanide agitation, the total cost is estimated at 23·72*s.* (labour = 4·88*s.*, and chemicals and zinc = 18·84*s.*), and by percolation it is estimated to be 24·368*s.* (labour = 1·92*s.*, chemicals and zinc = 22·448), the value of the gold extracted being in each case 11*l.* 12*s.* It is supposed for the purposes of the last-named calculation that the extraction by chlorination is 92 per cent., and by cyanide 85 per cent.—W. G. M.

Gold and Platinum Nuggets and Gold Ingots, On the Crystalline Structure of. A. Liversidge. Proc. Chem. Soc. 1897, [174], 22.

In view of the theory that gold nuggets are built up of concentric layers deposited round a central nucleus, the author has examined a large number of specimens from various sources. The nuggets were ground down, or sliced through, to obtain sections, which were polished and etched by suitable solvents. They all possess a well-marked crystalline structure, and usually enclose foreign substances. The crystalline structure is not incompatible with an aqueous origin; and the author suggests that the gold has been slowly deposited from solution, either at ordinary or at high temperatures, the nuggets being more or less rolled masses of gold which have been set free from disintegrated veins.

Gold, On the Presence of, in Natural Saline Deposits and Marine Plants. A. Liversidge. Proc. Chem. Soc. 1897, [174], 22—23.

The author gives a preliminary account of the results of the examination for gold of rock salt, sylvine, and other similar saline deposits, bittern, seaweed, kelp, oyster shells, &c. The process of determination used was to add from 0·5 to 5 grms. of ferrous sulphate to the unfiltered solution of 100 to 1,000 grms. of the salt in water. The resulting precipitate was then scorified with lead and cupelled. The natural salts contained from 1 to 2 grains of gold per ton, whilst kelp and bittern furnished in some cases as much as from 14 to 20 grains.

Tellurium [in Gold Ores] Occurrence of, in Montana. R. Pearce, Eng. and Mining J., **63**, (5), 117.

The author has examined for tellurium in an oxidised form, some rich gold ores which show no outward indication of precious metals, but which by assay yielded 21·33 oz. of gold and 93·67 oz. of silver per ton. On treating the ore with hydrochloric acid the whole of the ferric oxide was dissolved, and a residue of white silicious material remained. The solution contained tellurium, indicating a quantity equal to 87·49 oz. per ton. The insoluble silicious portion contained gold and some crystals of sylvanite. These results would indicate that tellurides of gold and silver might occur below the zone of oxidation, and that in this specimen, which was mined near the surface, the tellurides were not completely oxidised.—A. W.

Tellurium [in Gold Ores] from the Griffith Lode, Colorado. R. Pearce. Eng. and Mining J. **63**, (6), 139.

An analysis of a mineral recently found in the above mine gave the following result:—Silver, 50·65; tellurium, 18·80; lead, 9·34; copper, 4·65; iron, 4·00; bismuth, 1·16; sulphur, 8·06; magnesium carbonate, 1·95; alloy of gold and silver, 0·48; and insoluble residue, 1·07 per cent.; total, 100·16. It was believed that the gold was combined with tellurium, but on treating the mineral with nitric acid there was no indication of brown sponge gold, which would have been the result if these two elements had been combined. The residue obtained was a bright pale yellow alloy, which proved to be 0·725 fine gold, the remainder being silver. The mineral is considered to consist of a mixture of telluride and sulphide of silver, associated with other minerals, and with the alloy of gold and silver in the quantity mentioned in the above analysis.—A. W.

Uranium from Residues, Recovering. A. Gawalovski. Zeits. Anal. Chem. 1896, **35**, 678.

The uranium phosphate residues are dissolved in concentrated sodium carbonate solution, the whole filtered, then ferric chloride added in sufficient quantity to precipitate the phosphoric acid, and again filtered, from the solution magnesia mixture, added in small quantities at a time, separates the rest of the phosphoric acid. After standing 24 hours and filtering, the solution may be at once acidified with acetic acid, or acidified with hydrochloric acid, precipitated by ammonia, and the precipitate, after washing, dissolved in the desired acid.—A. C. W.

Lead Blast Furnaces, Wall Accretions on. M. W. Iles. School of Mines Quarterly, Nov. 1896, 18—23.

The author has investigated the "hangings" on the walls of blast furnaces and has been led to the following conclusions:—Zinc in its various forms and combinations is one of the chief causes of these accretions. Generally speaking an increase in the amount of zinc increases the trouble. Certain salts of zinc are more troublesome than others, the carbonate is the least, whilst the sulphide, especially when associated with galena in a fine state of division, is particularly so.

The finer the particles the more rapid the accumulation. Zinc is found, nearly always, as sulphide in the hangings, though in the lower portions of the furnace where the blast impinges small quantities of zinc oxide occur. The amount of zinc found by the author in these hangings during a period of 17 years averages 18·42 per cent., but the determinations, ranging over a great variety of conditions, varied from 5·3 per cent. to 63·8 per cent.

Experience has shown that it is not advisable to use scrap-iron upon heavy zinc charges, since heated metallic iron decomposes zinc sulphide, though good results have been obtained by the formation of slags containing iron in the

form of a silicate of protoxide. The amount of calcium oxide should be diminished with increasing amounts of zinc, great care being taken to avoid excess of the former, as the hangings accumulate very rapidly in presence of an excess of an alkaline earth.

The author gives the following percentage averages of the various determinations he made in his investigations:—17·26 of SiO_2, 13·26 of Fe, 1·60 of Mn, 3·30 of CaO, 10·90 of S, 18·42 of Zn, and 0·80 of Cu.

The fire assay gave as a general average of 17 years:—29·74 oz. per ton (2,000 lb.), silver; 21·06 per cent., lead; and 0·197 oz. per ton (2,000 lb.) of gold.

The silica was found to exist both as free silicic acid, mechanically lodged in these hangings, and as silicates. The iron existed as silicate, oxide, and sulphide. The author adds, however, that "the sulphide enters into the hangings as a true constituent, and is a potent factor in the formation of these accretions, whilst the silicate and oxide are merely accidental or incidental."

Manganese existed as silicate, from ore which had become entangled by entering from the top ledge of the hanging. The lime existed generally as such, free and uncombined; as sulphide there was the merest trace.

The sulphur was present chiefly as a sulphide of zinc and lead, which formed the main mass of the hangings. There was always a little sulphate present. The author has from time to time observed many curious and interesting chemical compounds in these accretions; the most interesting being the chloro-bromide of lead.

He recommends the old-fashioned way of taking the gases from the centre of the furnace instead of from the back end wall. Whilst he thinks the ore-fluxes and fuel should be uniformly distributed over the entire top, yet the coarse pieces should be thrown as nearly into the centre as possible; the furnace drives faster, and there is not so much heated gas following the side walls, and hence the tendency towards hangings is much diminished.—O. H.

Matte, Granulating. S. E. Bretherton. Eng. and Mining J., 1897, **63**, 43.

The matte, after being tapped from the crucible or settler into an ordinary slag pot, is wheeled while hot, directly over a square wooden tank nearly full of water. Just below the top of the tank, and about 10 or 12 in. above the water, is inserted a 2-in. water-pipe with the end flattened so as to throw a thinner and wider stream. This stream of water (a pressure of 25 to 30 lb. was found to be sufficient) strikes the stream of hot matte and scatters it just before falling into the water, where it settles. The shell should not be allowed to fall into the tank, but in case one should slide out, iron bars are placed across the tank to catch it, these bars not interfering in any way with the stream of matte passing down between them.

By granulating the matte, the expense of breaking, crushing, rolling, sizing through screens, and preparing for roasting or sampling, is done away with, and a product is obtained that will roast to much better advantage than when the matte has to be crushed and rolled.—A. S.

Spectrographic Analysis of some Commercial Samples of Metals, of Chemical Preparations, and of Minerals from Potash Beds, Stassfurt. W. N. Hartley and Hugh Ramage. Proc. Chem. Soc. 1897, [175], 46—47.

In continuation of the work already published (Roy. Soc. Proc., 1896, **60**, 393; and Proc., 1897, **13**, 11), samples were examined of steel made at Middlesbrough from the blast-furnace metal smelted from Cleveland clay ironstone, and rolled into rails; of alumina and "red mud" separated from bauxite at the British Aluminium Co.'s Works at Larne, and of the aluminium prepared from the alumina at Foyers and of various commercial alums.

It is shown that of the constituents of the blast-furnace metal, the alkali metals, calcium, copper, silver, gallium, manganese, and lead are present also in the steel, but the chromium and nickel have been removed.

Of the constituents of bauxite, traces of sodium, potassium, calcium, copper, silver, gallium, iron, manganese, and lead are found in the metallic aluminium. These elements are also present in larger quantities in the "red mud," and in addition, nickel and chromium are present.

The Alums.—Examined directly, by heating 0·5 grm. of the dried sample in the oxhydrogen flame, sodium, potassium, rubidium, calcium, and thallium are found as common constituents, and copper, gallium, iron, and nickel as occasional constituents. On examining the precipitates produced by potassium ferrocyanide in solutions, containing 50 grms. of the alum, strongly acidified with hydrochloric acid, these precipitates were found to contain the elements sodium, potassium, rubidium, cæsium, copper, silver, calcium, gallium, thallium, nickel, manganese, besides iron, which was also present in the acid radical. A sample of "aluminoferric" from Messrs. Spence and Sons, Manchester, contained all the elements found in the alums, but in much larger quantities. Of these elements, the pyrites furnished the thallium and also a trace of indium found in a by-product of the manufacture of alum, whilst the other elements were traced to the aluminous minerals, bauxite and shale. The shale was richer in alkalis and gallium than the bauxite, but a sample of French bauxite was richer in silver and lithium than either Irish bauxite or shale.

Samples of Stassfurt minerals were examined in the course of the investigation, and were found to yield spectra containing no lines of rubidium, cæsium, gallium, or thallium.

The systematic examination of railway metal by such an analytical method as is here employed, might lead to results of practical importance. The method reveals the presence of small quantities of metals such as copper, silver, gallium, and lead, which have not been considered in dealing with commercial irons, and the influences of which upon the physical properties of these have not been studied.

Steel, The Bertrand-Thiel Process. P. C. Gilchrist. Cleveland Inst. of Engineers, Dec. 1896.

The process consists in doing in two, or in two series of open-hearth furnaces, what has hitherto been done in one furnace, and in employing a temperature sufficiently high to cause the metal flowing from the first series of furnaces to emit a copious reddish-brown fume indicating a combustion of iron. The results are that a quality of steel equal to that from Swedish iron may be made from any non-sulphurous pig; the yield from pig iron may be more than cent. per cent. of the metal charged; and the speed of working is greater per furnace than heretofore. Possibly the speed may amount to 24 charges in as many hours from five 20-ton furnaces, or 480 tons of steel per day of 24 hours. An account of the process has been given by Hartshorne before the American Institute of Mining Engineers; and this was abstracted into the *Iron and Coal Trade Review* for October 27, 1896. The author has studied the working of the system in detail, and has followed several charges through, by sampling and analysing the products at different stages, and the results of the investigation are given in full. It is essential that the temperature should be sufficiently high in the primary furnaces to cause the evolution of brown smoke as the tapped-metal runs down the launders; this smoke may be due to the metal being composed of carbide of iron dissolved in fluid iron, and to the latter being so hot that it volatilises freely. At present only one primary and one secondary furnace are being used in the plant at Kladno. The author considers that not less than three, but preferably six furnaces should be used. In the latter case, four would be used to superheat molten metal from the blast furnace, 1 to desiliconise and dephosphorise down to 0·1 per cent., and the sixth to reduce this to 0·01 per cent. and to make ready for casting. The metal from the superheating furnace is conveyed to the third furnace, the slag goes into a bogey. In the finishing furnace the remaining phosphorus and carbon are removed.

At present in the primary furnace the ore and lime are added with the charge (in the middle); in the finishing furnace some of the ore and limestone is added with the charge, the balance being introduced if necessary, just before it receives the metal from the primary furnace. In the heats recorded, the charge of the furnace consisted of pig and scrap to the amount of 11 or 12 tons in the

primary and 8 tons in the secondary furnace. The proportion of scrap varied from 0 to 64 per cent., and the loss of metal usually ranged from about 2 to nearly 10 per cent. The time required, varied from $3\frac{1}{2}$ to $4\frac{1}{2}$ hours in furnace No. 1, and from 1 to 2 hours in No. 2. In the primary hearth the carbon was commonly reduced by over 1 per cent. (from 2·5 to 3·5 per cent.), and the phosphorus by 0·7 per cent. (out of 1·7 per cent.) In one instance, the phosphorus was reduced in the first furnace from 0·584 to 0·078 per cent. while the carbon remained at 2·57 per cent. Hence it is considered that such a furnace might be used as a feeder to the basic Bessemer converter and that no after-blow would be required. The limits of phosphorus and silicon permissible, are those obtaining in blast-furnace practice, but it would appear to be advantageous to use up tap-cinder and mill-cinder (if made on an oxide or basic bottom) in the Bertrand-Thiel- rather than in the blast-furnace. The loss of metal is found to be least when least scrap is used; in one instance a total charge of 20·06 tons of metal (including 30 per cent. of scrap) yielded 20·13 tons of steel. Some of the scrap must necessarily be oxidised in melting, but the oxide is partially or entirely reduced again by the metalloids in the charge. If the oxidation be effected by atmospheric oxygen, the loss must of necessity be considerable not only on account of the removal of metalloids, but because the silicon and phosphorus form acids which combine with lime and some oxide of iron. But when iron ore is used for oxidation, the removal of the metalloids entails the reduction of a greater weight of iron which is thus added to the charge. Thus the more silicon, phosphorus, and carbon there is in the pig iron, the greater will be the weight of steel obtained. It is possible that these good results may be due to the intense heat employed permitting the oxidation of the metalloids and manganese to take place with much more ease and rapidity than formerly; also to the transfer from one furnace to another getting rid of the voluminous slag and enabling the steel to be got ready for casting.—W. G. M.

Aluminium, Action of Carbon Monoxide and Dioxide on. Guntz and Masson. Comptes rend. 1897, **124**, 187—190.

In preparing aluminium iodide by acting on aluminium with iodine in a stream of carbon dioxide, there is always incandescence, accompanied by absorption of carbon dioxide. The authors find that aluminium alone heated in a stream of the gas absorbs it very slightly, but when two boats, containing respectively aluminium chloride or iodide and aluminium, are heated in the same tube in a stream of either carbon monoxide or carbon dioxide, there is incandescence as soon as the vapour of the iodide reaches the aluminium. An increase of weight amounting to 45·47 per cent. of the weight of the metal took place, and a dirty yellow, not quite homogeneous, substance resulted. This body, decomposed by water, gave off a gas which proved to be methane (but which had an unpleasant odour like that of the gas obtained by dissolving cast iron in acids), and no appreciable residue of carbon remained. The authors give the following equation in the case of carbon monoxide. $Al_6 + 3CO = Al_2O_3 + C_3Al_4$.
—J. T. D.

Aluminium Alloys. J. Franklin Institute, **143**, 145—147.

This paper is a report taken from the *Aluminium World* on the alloys of aluminium with other metals. The useful alloys are in two groups, one with not more than 15 per cent. of aluminium, and the other with not more than 15 per cent. of the other metal in the aluminium. The compound alloys are not generally dealt with. Tin improves strength and rigidity in heavy castings of aluminium; it gives also sharpness of outline with a decrease in the shrinkage of the metal. With from 7 to 10 per cent. of nickel, the best aluminium is produced for casting, where toughness combined with hardness is desired. Two new alloys for jewellery work consist of (1) 20 parts of nickel and 8 parts of aluminium; and (2) 40 parts of nickel, 10 of silver, 30 of aluminium, and 20 of tin. Manganese is one of the best hardeners, and is added as a rich ferro-manganese for castings, and as a pure oxide of manganese to the electrolytic bath in which aluminium is produced for rolling purposes. Tungsten alloys are specially used for rolled sheets and plates, to be afterwards "spun," and generally in conjunction with other metals, such as copper, iron, and manganese. Aluminium hardened with chromium, retains its hardness after annealing or heating better than almost any other of the alloys. Titanium increases the resilience, and with chromium, copper, and aluminium produces some very hard and tough light alloys. Alloys containing zinc up to 30 per cent. are successfully used. Cadmium does not harden aluminium, but with it produces a solder. Bismuth produces fusible and brittle alloys, which oxidise rapidly when melted.—A. W.

Nickel and Copper, Separation of, in Matte, and the Recovery of the Contained Precious Metals. T. Ulke. Eng. and Mining J. **63**, [5], 113.

See under XXIII., *page* 262.

PATENTS.

Iron and Steel, An Improved Manufacture of, and Method of Extracting Gold and Silver from Ores. A. J. Boult, London. From C. J. L. Otto, Dresden. Eng. Pat. 1016, Jan. 18, 1896.

The iron or steel is produced by heating spongy iron to a suitable temperature, in a neutral atmosphere, and under high pressure, in a reduction vessel or crucible heated from the outside. The crucible is so shaped that the iron produced may accumulate where the temperature is highest, and it should be arranged so that it may be swung or inclined so as to bring the molten metal more quickly in contact with the spongy iron. When gold or silver is present, the highest temperature is to be kept up till the precious metal is deposited by liquation on the bottom of the crucible. Compressed air is led into the crucible by means of a pipe, and the escaping compressed gases may be utilised for the preliminary heating or calcination of ores, &c.—J. H. C.

Metallic Alloys, Impts. in and connected with. J. P. Fullerton and A. B. King-Church, London. Eng. Pat. 2858, Feb. 7, 1896.

The alloy is composed of lead, 91 per cent.; copper, 1; bismuth, 1; and antimony, 7 per cent.; but the proportions may be varied. It is fusible at a low temperature, expands as it solidifies, and is less subject to corrosion than brass.

In preparing the alloy, the copper is reduced to small particles, which are sprinkled over the surface of a small part of the lead, which is heated to about 1,990°, and well stirred in. The antimony is heated in another vessel, and the mixture of lead and copper added; the remainder of the lead is then added, and finally the bismuth. The articles to be made from this alloy (gas and water connections) are cast either in moulds or chills.—J. H. C.

Metals from Refractory Ores, Improved Process for Obtaining. J. S. Wallace, Belfast, and J. Castell-Evans, Hornsey. Eng. Pat. 2931, Feb. 8, 1896.

The process is intended for smelting complex ores containing lead and zinc, and it is proposed, *inter alia*, to extract zinc from blende by the direct action of carbon, with the formation of carbon bisulphide.—W. G. M.

Zinc, Recovery of, from Blende Ores; Process for. The Exploring and Gold Mining Assoc., Ltd. From S. H. Emmens, Amador City, California, U.S.A. Eng. Pat. 4432, Feb. 27, 1896.

The process is stated to be particularly applicable to zinc-lead-sulphide ores, such as those of Broken Hill. The crushed ore is so roasted that the zinc sulphide is converted as completely as possible into sulphate, and is then transferred to a series of vats placed in cascade form, each on a slightly lower level than that preceding it. By means of overflow pipes, the excess liquor from the highest vat passes to the bottom

of the next, and, ascending through the ore, overflows to the bottom of the next vat, and so through the series to a reservoir at the end. At the bottom of each vat is a lead anode, and at the top a zinc cathode. In use, alternate vats are connected with an electrolysing current from a dynamo. Thus, at first, water flowing over the ore in No. 1 vat extracts zinc sulphate, and this solution, passing to the second vat, is electrolysed, zinc being deposited and sulphuric acid liberated; the acid liquid then passes through the ore in the third vat (without electrolysis), and, becoming neutralised by means of zinc oxide, is ready to be decomposed by the current in the next tank, and so on. When all the zinc in the ore has been extracted, the sulphate remaining in the solution is crystallised out, and the leached ore is run into settling tanks to await further treatment.—W. G. M.

Blast Furnaces, The Construction of a Chamber or Receiver to be coupled to Existing, for Smelting Iron from the Ore, and also to New Furnaces. W. Hutchinson, Manchester. Eng. Pat. 5225, March 9, 1896.

The chamber is built in front of the furnace and furnished with a chimney for the escape of "sulphurous" gases.
—J. H. C.

Briquettes of Iron Ore or other Iron Compounds, and Process of Manufacturing Briquettes thus made. L. W. A. Jacobi and G. W. Petersson, of Stockholm. Eng. Pat. 23,958, Oct. 27, 1896.

The crushed iron ores are partly reduced to spongy iron and then formed into briquettes by pressure or percussion, moisture or acids being added when necessary. Any form of furnace may be used for the partial reduction.—J. H. C.

[*Gold.*] *Slimes, Extracting Precious Metals from, or from other Finely Divided Material containing the same. Improved Method and Apparatus for.* D. White and T. M. Simpson, both of Stawell, Victoria. Eng. Pat. 26,066, Nov. 18, 1896.

The slimes (or ore) are treated with cyanide liquors in closed vessels, preferably conical or pear-shaped. The vessels are placed in series, each being provided with pressure-and level-gauges, and with gratings forming a false bottom, having discharge cocks or valves below. "Gas" is blown, under sufficient pressure, into each vat beneath the grating, and agitates the charge, escaping by a pipe at the top, through which it is led downwards to the bottom of the next vat. The gas is finally forced into another reservoir. Air is usually employed, and rapidly becomes charged with hydrocyanic acid, which is not lost for practical purposes, owing to the repeated use of the same "gas" for agitation. Any vat may be cut out of the series temporarily by means of control cocks, so that the charging or discharging of one vessel does not check the continuity of the whole operation. The agitation permits even the finest slimes to be rapidly treated, and the continued use of the same air for mixing the charges reduces the loss of cyanide below that which is met with in ordinary percolation practice.—W. G. M.

Gold Saving and Amalgamating Machine, Improved. G. Dillberg and W. Walker, both of Sydney, N.S.W. Eng. Pat. 26,490, Nov. 23, 1896.

The finely divided ore or material containing free gold is run through a fixed hopper on to the centre of the floor of a very shallow iron pan, rapidly rotated by suitable mechanism. The sides of the pan are somewhat incurved at the top, and the pan is caused to revolve at such a rate that mercury previously placed in it is driven by centrifugal force against the sides. At a short distance above the bottom is a movable cover extending nearly to the outer rim. As soon as the ore is discharged upon the rotating floor, it is driven beneath this cover, centrifugally, to the circumference, and, impinging upon the mercury, passes up the surface of the latter and escapes over the side into a circular apron. In this way gold is amalgamated, whilst, as it is claimed, the mercury is not permanently "split" by the action, owing to its tendency to reunite under the centrifugal influence of rotation. Platinum or other heavy metal, incapable of amalgamation, remains with the mercury.—W. G. M.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(A.)—ELECTRO-CHEMISTRY.

Voltaic Cell, Two New Types of. H. Paulig. Zeits. f. Elektrochem. **3**, [15], 332—333.

(1.) The first of these is a double-fluid cell with two prismatic electrodes of retort-carbon. One of the electrodes stands in a cylindrical jar 20 cm. high, the other in a porous cell standing inside the jar. The electrolyte inside the porous cell is strong chlorine-water; outside it, concentrated sodium thiosulphate. The initial E.M.F. is 0·64 volt. After short-circuiting for about 50 minutes, it sinks to 0·47, at which it remains for about 5 hours, giving a current of 0·7 ampère. The external electrolyte gradually becomes turbid, owing to separation of sulphur (mainly in the neighbourhood of the electrode immersed in it, which is the anode).

(2.) The second is a modification of the ferric chloride cell. It is made up in a peculiar way so that the iron forms part of the containing vessel. A sheet of iron, 20 cm. long, 7 cm. broad, and 3 mm. thick, is bent twice so as to form three sides of a narrow oblong; the fourth (narrow) side is to be the open top of the cell. On a strong wooden board are placed several layers of sheet-rubber. The bent iron is placed edgeways on these, and inside (but not touching the iron) is placed a plate of retort-carbon of the same breadth and of suitable length. On these are placed more layers of rubber and then another board. The two boards are pressed together by iron clamps or screws. When a battery of several cells is to be made, it is not necessary to have separate pairs of boards for each; one pair of long boards will do for several cells. The iron forms the anode and the carbon the cathode. The electrolyte is concentrated ferric chloride. The E.M.F. is 0·9 volt. When the cell is in action, the ferric chloride splits up into the ions chlorine and ferrous chloride; the chlorine unites with more iron to form fresh ferric chloride:— $3FeCl_3 = 3FeCl_2 + 3Cl$; $Fe + 3Cl = FeCl_3$. The spent liquid can be regenerated by passing chlorine into it and so converting the ferrous chloride into ferric chloride. If we include this in the cycle, it will be seen that the ferric chloride acts as a carrier of the depolarisator chlorine, and that the only substances used are iron and chlorine, both of which are cheap. As there is no diaphragm, the internal resistance is very small. A very constant current can be obtained from a battery of such cells arranged as follows, so that the electrolyte slowly flows through them in series. The cells are placed side by side (but not in metallic contact) and nearly filled with solution. Each is then connected with the next by a siphon, which is also filled with ferric chloride solution. From a siphon at the end, the spent liquid drops into an open vessel. The fresh electrolyte drops into the first cell from a 15-litre bottle provided with a stop-cock at the bottom. In the mouth of the bottle is a rubber stopper with an open glass tube reaching nearly to the bottom of the bottle. By sliding this up and down, the rate at which air bubbles enter, and therefore the rate at which the solution drops out, can be regulated. The battery does not emit any fumes or smell. The author states that the battery is much cheaper than any other, and that 900 watt-hours can be produced at a cost of only 25 pf. Nothing is said as to the effect of the ferric chloride upon the rubber, or as to troubles which may arise as the iron of the cells is used up.—D. E. J.

PATENTS.

Battery Cells or Vessels to contain Acids or other Chemicals, Impts. in the Manufacture of. [*Asbestos.*] D. M. Hawes, London. Eng. Pat. 1228, Jan. 17, 1896.

The cells or boxes are preferably made of woven asbestos coated or impregnated with liquid celluloid, as described in Annison's patent, 19,710, 1891 (this Journal, 1892, 904).

The asbestos fabric so treated is cut into suitable pieces to form the desired vessel, and they are cemented together with liquid celluloid. Powdered asbestos or fibre may also be worked into a dough-like mass by mixing two parts by weight with one of celluloid, and the pulp so formed is moulded by dies into the desired shape.—G. H. R.

Electrolytic Apparatus, Impts. in. [Covers for Anode Compartments.] Col. T. J. Holland, Tunbridge Wells. Eng. Pat. 12,149, June 3, 1896.

In tanks used for decomposing solutions of chloride of sodium, &c., so as to produce caustic soda and chlorine gas, to obviate the difficulty of making a gas-tight joint in the covers to the anode compartments, the electrolytic vessel consists of a shallow, open tank, formed of slate or other suitable material, and provided internally with longitudinally extending ledges or narrow shelves, on which rest inverted boxes of earthenware or suitable material, which form removable air-tight covers to the anode compartments. These covers are pierced to receive pipes for the supply of the solution and the withdrawal of the gas, and their sides form porous partitions between the electrodes. The anodes consist of blocks of carbon attached to a rod or bar, bolted to the bottom of the inverted box, and the cathodes are grids of iron or copper-wire basket-work, preferably double, and of U-shape.—G. H. R.

Accumulators or Secondary Batteries, Impts. in Plates for. [Improved Casting.] C. Pollak, Frankfort-on-the-Main, Germany. Eng. Pat. 13,520, June 18, 1896.

The improved plates are cast under pressure in a mould, which is lined with common salt or other suitable material which will stand the temperature of molten lead, and which can subsequently be easily dissolved away. The salt is packed round a pattern or core, which is removed before the casting; or instead of the core, a lead plate of the required shape may be employed, which melts during the casting, and runs into the salt. The plates so produced consist of a core and a porous portion forming a continuous mass of metal.—G. H. R.

Disinfecting, Deodorising, and Bleaching Agents [Hypochlorites produced by Electricity], Impts. in the Manufacture of, and in Apparatus therefor. M. Crawford, London. Eng. Pat. 14,852, July 4, 1896.

See under VII., *page* 240.

Electric Batteries, Impts. in. [Gas-tight Primary Battery.] W. Rowbotham, Birmingham. Eng. Pat. 16,268 July 22, 1896.

The battery is contained in a hermetically sealed outer vessel, which is divided into two compartments, one of which is further subdivided to form a liquid expansion chamber. The battery chamber proper, which contains the zinc and iron elements and the exciting fluid, is at one end, and in this section the carbons are enclosed in porous tubes closed at one end and having an intervening annular space communicating with the middle chamber, which contains the depolarising fluid. The carbons, which are tubular, are closed at their other end, and, passing through this chamber, enter the partition, and communicate with the expansion chamber, into which the depolarising fluid is forced by the pressure of the gases generated during working. The depolariser enters the carbon tubes, where it is utilised until thoroughly exhausted. Valves or other means are provided so that the compartment may be filled or emptied in succession from one of them, while, when full, electrical communication between them is cut off. The exciting fluid is preferably a strong solution of sal-ammoniac or nitrate of soda, while the depolariser consists of a mixture of 1 part of sulphuric acid, sp. gr. 1·790, to from 1 to 6 parts of nitric acid, sp. gr. 1·48.—G. H. R.

Electrodes [Secondary Leclanché Batteries], Impts. in Peroxide of Manganese and Carbon, and in the Method of Producing the same. A. Heil, Fräukisch Crumbach, Germany. Eng. Pat. 29,307, Dec. 21, 1896.

Electrodes composed of graphite and peroxide of manganese are placed in a chloride solution, free from nitrogen (magnesium or sodium chloride), and reduced electrolytically; the current is then reversed, "and thereby converts the said electrode into a compound of chlorine." This electrode is for use in secondary (Leclanché) batteries; "no ammonia, and therefore no hydrated oxide of zinc, will be produced in the cell . . . the solution of sal-ammoniac is thereby maintained in its initial proper condition or composition."—J. C. R.

(B.)—ELECTRO-METALLURGY.

Carborundum [Silicon and Carbon], Manufacture and Development of, at Niagara Falls. F. A. Fitzgerald. J. Franklin Institute, **143**, 81—96.

(See also this Journal, 1895, 755, and 1893, 847.) The furnaces are built of brick, the internal dimensions being about 16 ft. in length, 5 ft. in width, and 5 ft. in depth. The ends are built solidly about 2 ft. thick, through the centre of which pass the terminals, consisting of 60 carbon rods, 30 in. long and 3 ft. in diameter. These two ends are the only permanent parts of the furnace, the sides being built for each operation. The furnace is about half filled with the mixture, care being taken to keep the latter free from contact with the terminals. A semicircular trench of 21 in. in diameter is formed from end to end, the bottom of the trench being a little above the level of the lowest row of carbons. In this trench the "core" is formed, by placing coke kernels of a certain size so as to form a long, cylindrical centre, which is then covered over with further quantities of the mixture to a total height of 8 ft. The current is supplied by the Niagara Falls Power Co., at 2,200 volts, which is transformed into 185 volts before use.

Carborundum is apparently infusible, for after a certain temperature has been reached it is decomposed, without fusion, into carbon and silicon. It is insoluble in water or any acid. Its hardness is between 9 and 10, but nearer the latter, and its specific gravity is 3·23. In making wheels for abrasive purposes, the carborundum is mixed with kaolin and felspar, placed in a mould, and submitted to hydraulic pressure. The wheel is then vitrified by heating in a kiln for 7 days and cooling slowly. With very thin wheels the binding material is shellac. The cost is twice to five times that of emery, but carborundum will do more work, it is said, and do it faster and better than either emery or corundum. At present it has been found unsuitable for grinding and polishing glass, owing to its extreme hardness producing scratches. Carborundum is soluble in molten steel, and Mr. F. Luermann suggests its use as a source of silicon in the manufacture of that metal.—A. W.

Manganese, Electrolytic Determination of. C. Engels. Zeits. f. Elektrochem. 1897, **3**, [14], 286 and 305.

See under XXIII., *page* 262.

PATENTS.

Electrolytical Apparatus [Deposition of Zinc], Impts. in. R. Heathfield and W. S. Rawson, London. Eng. Pat. 19,828, Oct. 22, 1895.

In Eng. Pat. 5537 of 1894 (this Journal, 1895, 756), the inventors describe apparatus for depositing zinc or other metal. Instead of employing a perforated barrel of non-conducting material partly immersed in the electrolyte, they now describe and illustrate a hermetically closed barrel containing the electrolyte, preferably more than half filled, and caused to revolve by resting flanges at its ends on rotating rollers. The anode is placed so as to be centrally insulated from the barrel. The cathode is formed of conducting bars arranged at intervals around the interior of the barrel, but insulated therefrom. Electrode contacts are made by metal discs dipping in mercury, but they may be made by brushes bearing on insulated rings or segments.
—J. C. R.

Electro-Plating, An Improved Process of Preparing Non-Metallic Substances for. [Coating Non-Metallic Articles.] T. M. Ash and H. N. Weldon, Birmingham. Eng. Pat. 2327, Feb. 1, 1896.

If the non-metallic articles to be coated—such as paper, china, glass, or textile fabrics, &c.—are impervious, they are

first dipped into or sprayed with a solution which will give them a coating of silver, and they are then ready to receive an electrolytic deposit of metal by any of the ordinary processes. If the objects to be plated would be injured by the silver-depositing solution, they first receive a coating of copal or other suitable varnish. The silver bath is formed of a mixture of two solutions, formed respectively of 24 grs. of sodium potassium tartrate; and of 35 grs. of nitrate of silver dissolved in water, to which solution pure ammonia is added slowly until it becomes clear, when a further proportion of about 38 grs. of nitrate of silver is added. It is generally preferable to use only just sufficient water to dissolve the salts, but as much as 18 grs. may be added.
—G. H. R.

Electrodes for Accumulators [Zinc-Plated Copper], Impts. in and in the Construction of Soluble Metal. W. P. Thompson, London. From J. Julien, Brussels, Belgium. Eng. Pat. 7259, April 2, 1896.

The copper plates which are to form the supports of the soluble electrodes, are electro-plated with copper and then amalgamated with mercury, on which zinc, cadmium, or the like is electro-deposited. It is claimed that the mercury prevents these soluble electrodes from being attacked by the electrolyte, and to render the amalgamation more durable, the base of the electrodes may rest in a layer of mercury. In the case of a tubular copper electrode, its base may be closed by a block of insulating material forming a narrow rim round the tube to contain the mercury.—G. H. R.

XII.—FATS, OILS, AND SOAP.

Animal Fats, Contributions to the Chemistry of. C. Amthor and J. Zink. Zeits. Anal. Chem. **36**, 1—17.

The authors give the tabulated results of a number of analyses of the fats obtained from 26 animals of different species, which are not frequently met with. The figures are the averages of several analyses. In most cases the fats from several animals of the same species were mixed together, the analysis being made on the mixture. As may be seen in the table given in the original, the specific gravity of the fatty acids is sometimes higher and in other cases lower than that of the corresponding fat. The melting and solidifying points of the fatty acids are higher than those of the fats themselves, whilst the iodine figure is generally lower. With many of the fats the acidity does not appreciably increase as the fat grows old; in other instances—fox, cat, and hare fats—it increases to a considerable extent. The iodine numbers and, in most cases, the acetyl numbers of the domestic animals are lower than the corresponding figures of the wild animals of the same species. A comparison of the results obtained with the fat of a wild goose and that of a wild goose kept in captivity is interesting. It was found that hare, wild rabbit, wild boar, and black-cock fats have drying properties, and that they contain linolic and linolenic acids. The fat of a wild boar is in this respect very different from that of the ordinary pig.
—W. P. S.

Japan Wax, Adulterated. C. H. La Wall. Amer. J. Pharm. **69**, 18, and Pharm. J. 1897, **58**, 83.

Of 59 cases containing 205—225 lb. each, 25 were found to be adulterated with starchy material to the extent of 20 to 25 per cent. The specific gravity of the adulterated product was slightly higher than that of the genuine fat (or Japan wax), and it was as a rule, free from the peculiar network of minute cracks which usually characterise the surface of cakes of pure Japan wax.—A. S.

Fatty Acids, Note on Wechsler's Method for the Separation of. A. W. Crossley. Proc. Chem. Soc. 1897, [174], 21—22.

See under XXIII., *page* 263.

PATENTS.

Oil from Fish, Fish Refuse, or other Oil Producing Animal or Vegetable Matters; Impts. in and Relating to the Extracting and Recovering of. J. Jack and M. Blake, Greenock. Eng. Pat. 2511, Feb. 1, 1896.

A method of flotation and precipitation, in which use is made of the different degrees of density of liquids or semi-liquids, as applied to the extraction of oil from fish, fish refuse, or other oil-yielding animal or vegetable matters. The process is carried out in a series of vessels, or compartments of a single large vessel, to which heat is applied by means of steam or hot water, either circulating through coils, or introduced into a jacketed space surrounding the vessels, &c. In working the apparatus, the flow of the steam or hot water is so regulated by valves, &c., connected with the vessels, &c., that any desired temperature can be maintained in any or all of these. However, for the object directly in view, the greatest heat is to be applied to the vessel or compartment forming the last of the series, in which the process within the vessels terminates, the temperature in each preceding vessel, &c. gradually decreasing until the first is reached. The tops of these vessels are set level with each other, and there are sight-glasses to indicate the levels of the various constituents in each vessel, as well as run-off pipes, siphon-pipes, and cocks for each. There is finally a suction-pump, which serves the different vessels, and is so arranged that all or any part of the contents in any one tank can be transferred to all or any one of the others in the series if required.—J. J. K.

Oil from Seeds or other Oleaginous Substances, Impts. in the Method of and Apparatus for Extracting. W. R. Harrison and E. Stephenson, Hull. Eng. Pat. 7379, April 7, 1896.

"An apparatus or plant," is claimed "for extracting oil from seeds or other suitable oleaginous substances by means of solvents," such as benzoline, naphtha, &c., "and for recovering and re-distilling the solvent concurrently with the extraction of the oil, and also for converting the residuum into meal, the employment of gas generators, oil extracting kettles (with internal stirrers and sweepers) condensers, air extractors, and air receivers, oil separators, store tanks, bubble tanks, and water pressure tanks together with connecting pipes, receivers, cocks and valves, worm and band or other conveyers and hoppers, the whole apparatus driven by any suitable gearing."
—J. J. K.

Fatty Matters from Certain Animal Tissues, Impts. in the Manufacture of. J. Davidson, Sydney, New South Wales. Eng. Pat. 25,472, Nov. 12, 1896.

Fatty tissues of cattle are digested with steam under a pressure of 30—100 lb. per sq. in., until the cellular tissue is broken up, and the fat liberated. Whilst under pressure, the "soup" and fat in a liquid state are blown out, and sent through a centrifugal separator so as to ensure the separation of all the fat.

The claim is for "An improved manufacture of fatty matter from fatty tissues of cattle, consisting in treating the crude fatty products, obtained by boiling these tissues, with hot water and steam, and straining them in a basket-centrifugal, and treating the fats obtained from the product in a centrifugal separator."—J. J. K.

XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

(A.)—PIGMENTS, PAINTS.

PATENTS.

White Lead Pigments, Impts. in the Manufacture of, and in the Separation of Metallic Lead [with Recovery of Silver], from Lead Sulphide. A. Macdonald, Notting Hill. Eng. Pat. 25,133, Nov. 9, 1896.

This is a modification of Eng. Pats. 2883 and 11,972, 1893 (this Journal, 1894, 401 and 893). The fused galena has a current of air and steam, either together or separately,

blown over its surface, whereby a basic sulphate of lead, $2\,PbSO_4.PbO$, is sublimed, a portion of the metal is reduced, and some SO_2 is evolved and can be collected for further use. The basic sulphate forms an excellent pigment, and the reduced metal retains all the silver existing in the original ore.—F. H. L.

Distempers or Colour Washes, Impts. in. W. A. Hall, Bellows Falls, Vermont, U.S.A. Eng. Pat. 28,233, Dec. 10, 1896.

This patent describes the preparation of a distemper or colour wash, which becomes "insoluble," waterproof, and very hard on drying. It is kept in the form of a powder, and needs only suspension in water to be ready for use. The pigment consists of talc or about equal quantities of talc and whiting; the adhesive portion is composed of 60 per cent. of dry casein and 40 per cent. of slaked lime; and the ingredients are finally mixed together in the proportion of 90 per cent. of the pigment and 10 per cent. of the casein-lime cement.—F. H. L.

(B.)—RESINS, VARNISHES.

Gamboge, Composition of. G. Sassinari. Pharm. Centralh. 1896, **36**, 768.

The following constituents were found in gamboge: a gum analogous to acacia gum; a volatile oil boiling between 160° and 210° C., and containing a terpene and a camphor (this oil appears to be noted for the first time); isovitinic and acetic acids; a phenolic ester; a resin; methylic alcohol and other higher homologues, and a liquid with a fruity odour having a high boiling point and presenting the characters of an aldehyde or of an acetone. Phloroglucinol, previously recorded as a constituent, is considered to be a decomposition product.—J. O. B.

PATENT.

Turpentine from Wood and Wood Waste, Improved Process for Obtaining. A. Schmidt, Cassel, Germany. Eng. Pat. 26,015, Nov. 18, 1896.

The dried sawdust of wood, rich in resin, is formed into briquettes, heated in retorts to 120° C., and subjected to a current of dry steam under slight pressure so long as turpentine continues to be evolved. The steam is then shut off, and the distillation finished in the usual manner.

—F. H. L.

(C.)—INDIA-RUBBER, &c.

Isoprene [From Destructive Distillation of India-rubber], Constitution of. W. Ipatiew and N. Wittorf. J. Prakt. Chem. 1897, **55**, 1.

In the dry distillation of india-rubber the hydrocarbon isoprene, C_5H_8, is obtained. Isoprene polymerises to dipentene and other products similar to india-rubber; the authors find that the hydrocarbon unites with hydrobromic acid to form a dihydrobromide identical with β-dimethyltrimethylenedibromide $(CH_3)_2:CBr.CH_2.CH_2Br$. On the assumption that the carbon chain is an open one, isoprene must thus be unsymmetrical methyldivinyl, $CH_2:C(CH_3).CH:CH_2$, the addition of the second hydrobromic acid molecule taking place in a manner contrary to general rule.—A. C. W.

Isoprene, Constitution and Syntheses of. W. Ipatiew. J. Prakt. Chem. 1897, **55**, 4.

By the action of alcoholic potash on β-dimethyltrimethylenedibromide (prepared by addition of hydrobromic acid to isoprene or dimethylallene), a hydrocarbon is formed which has no action on ammoniacal cuprous chloride or silver nitrate, and thus cannot be a mono-substituted acetylene. It cannot be a di-substituted acetylene, because its boiling point is lower than that of any of the hydrocarbons, C_5H_8, of that series. The product should be dimethylallene $(CH_3)_2C:C:CH_2$, but boils at 33°—34°, whilst the former body boils at 40°—41° C. The substance is isoprene, which thus must have the constitution given in the preceding abstract.—A. C. W.

PATENT.

Solving and Re-integrating Vulcanised India-rubber, Process for. E. Sefton, Christchurch, New Zealand. Eng. Pat. 27,579, Dec. 3, 1896.

Vulcanised rubber may be dissolved in naphtha, petroleum spirit, or carbon bisulphide by heating it in a closed vessel with the liquid for seven days at a temperature of 60° C.

—F. H. L.

XIV.—TANNING, LEATHER, GLUE, SIZE.

Glue, Viscosity of; A New Process for Determining the. J. Fels. Chem. Zeit. 1897, **21**, 56 and 70.

See under XXIII., *page* 264.

Fermentation Phenomena in Tan Liquors. F. Andreasch. Imp. Research Laboratory, Vienna. Der Gerber, **22**, [513], [514], [524], and [525].

(See this Journal, 1896, 910; 1897, 52.) After the previous general consideration of the fermentation phenomena in tan liquors, the author gives results of the examination of some typical liquors to show the connection between the organisms present and the acids found, and the progress of the fermentation in sole leather colouring pits worked on the old and new systems. For the first examples, characteristic liquors were chosen, the preparation, age, and character of which were known.

Sour oak liquors from a Rhenish tannery:—

I. Sour liquor of the third layer, five months old, contained per 100 c.c. 0·63 grm. of acetic acid; traces of lactic acid; CO_2 found qualitatively to be present; alcohol found in the distillate.

Bacteriological result: Saccharomycetes, Bact. aceti, Mycoderma vini, two milk-coagulating bacteria, a mould, sarcina, and micrococcus plumosus.

II. Sour liquor nine months old:—Acetic acid 0·79 grm. per 100 c.c., lactic acid 0·41 grm., traces of CO_2 and alcohol.

Bacteriological result: Alcohol yeasts, B. aceti (in small numbers), lactic bacteria and yeasts in large numbers, mycoderma, forming a thick pellicle.

Sour oak-bark liquors:—

I. Eight months sour bark, third layer:—Acetic acid 0·34 grm. per 100 c.c., lactic acid 0·26 grm., tannin 0·51 grm., non-tannin 1·08 grm.

Bacteriological result: Yeasts in enormous number, few acetic bacteria, lactic acid bacteria and yeasts, the latter predominating, isolated cells of mycoderma.

Similar liquor, first layer:—Acetic acid 0·52 grm. per 100 c.c., lactic acid 0·28, traces of CO_2 alcohol found qualitatively, tannin 0·18 grm., non-tannin 1·72 grm.

Bacteriological result: Alcohol yeasts, mycoderma, acetic bacteria in large quantity and growing strongly, lactic organisms in small quantity.

The two following tables show the regular increase of acids during the use of the liquors and the simultaneous decrease of the soluble non-tannin. The decrease of tannin is proportional to the progress of the tanning process.

The influence of the mode of preparation is easily seen. [The old method of preparing liquors and tannin having been entirely discontinued in this country, the author's remarks on the subject are omitted.]

These analyses illustrate the conclusions previously given (see this Journal, 1896, 910). The next part of the research was to examine the action of the most frequently occurring typical species of bacteria on pure tan liquors, to ascertain the special conditions required to bring them to proper development, and finally to employ the results in practice.

It is of the highest importance to know whether the tannin is decomposed by bacteria and thus lost as a leather producing agent. It has been assumed, and in a few cases shown that certain tannins are not decomposed when the liquors undergo fermentation, but the question cannot be considered as settled. With the object of deciding this, the author examined the following tanning materials: oak, pine, willow and hemlock barks, quebracho and oak wood,

Oak-Pine Bark Liquors, prepared by Cold-Leaching (Battery).

—	Acetic Acid.	Lactic Acid.	Tannin.	Non-Tannin.	Remarks.	Bacteriological Result.
	Grms. per 100 c.c. of Liquor.					
I. (Strongest)	0·39	0·45	1·83	2·88	Strong evolution of gas. Alcohol and CO_2 found.	Bottom ferm. yeasts, B. aceti, no mycoderma, few lactic acid organisms.
II.	0·40	0·45	1·50	2·48	Slight evolution of gas ...	Yeasts, B. aceti, and lactic organisms, principally yeasts.
III.	0·42	0·51	1·27	2·31	.. ..	Increase of acetic and lactic bacteria.
IV.	0·48	0·58	1·08	2·14	.. ..	" " "
V.	0·56	0·58	0·92	1·98	.. ..	Acetic bacteria and lactic acid yeasts ceased to develop.
VI.	0·56	0·63	0·78	1·82	Pellicle of mycoderma begun to form.	Increase of lactic bacteria and mycoderma.
VII.	0·44	0·65	0·64	1·67	Thick pellicle of mycoderma.	Lactic acid bacteria sarcina and mycoderma in large numbers.
VIII. (first colouring pit).	0·47	0·48	0·58	1·53	Mycoderma pellicle, mould growth beginning.	Progressive increase of lactic organisms, moulds, diminution of acid owing to the fresh hides taking it up.

The relatively high acidity of the first colouring liquor (five days old) is caused by the use of sour liquors and sour material in the leaches.

Pine-bark Liquor from Sole Leather Colouring Pits (prepared by the old System).

—	Acetic Acid.	Lactic Acid.	Tannin.	Non-Tannin.	Bacteriological Result.
	Grms. per 100 c.c. of Liquor.				
I. (Strongest)	0·41	0·54	0·86	2·68	No characteristic ferments present, all the liquors much contaminated with putrefactive bacteria, owing to long use and consequent accumulation of nitrogenous nutrient material.
II.	0·43	0·65	0·74	2·58	
III.	0·43	0·70	0·70	2·40	
IV.	0·51	0·75	0·54	2·13	

sumac, gall-nuts (knoppern), and aleppo galls, valonia, myrabolans, and divi-divi. Infusions of these were used of two concentrations, one containing double the amount of tannin of the other.

The non-tannin was separated by precipitating the tannin (*a*) by hide powder, (*b*) by magnesia. The tannins of galls, myrabolans, sumac, and pine and oak barks were prepared by the methods of Böttinger, Etti, and Löwe, and their purity tested by the gravimetric method and precipitation with ignited magnesia. Löwenthal's method appeared to be unsuitable for estimating the non-tannin as a control. The infusions were inoculated with the following organisms. *Fungi:* Alcohol yeasts, Torula, Mycoderma, Penicillium glaucum. *Bacteria:* Various lactic acid bacteria and air germs. A few inoculations were made with B. aceti, which confirmed the conclusion that these ferments do not decompose the tannin. Putrefactive ferments as specific consumers of nitrogen were excluded from the research. Changes of composition were supposed to take place in the liquors in the absence of bacteria, therefore in each experiment, sterile infusions exactly similar to those inoculated were put aside for comparison in order to eliminate any such influence from the result. The solutions, after sterilising in the steam steriliser (both inoculated and uninoculated), were kept in the incubator at 30° C. for 50 days. All the materials prepared as above gave negative results with regard to the fermentability of the tannin, by any of the organisms.

The solutions were examined microscopically to ascertain the growth of the organisms.

The fermentation products were estimated by comparison of the amount of substance not precipitated by magnesia before and after the fermentation. The sugars were estimated by Fehling's solution after treating with magnesia and inverting. Alcohol was shown qualitatively by the usual iodoform reaction and quantitatively by the capillary method of Vallon.

CO_2 was weighed after absorption by soda lime. Acetic acid was distilled off, and the distillate titrated with NaOH. It was identified by concentrating the neutralised distillate and adding ferric chloride (blood red colour). Lactic acid was estimated by treating the residue of the fermented solution after distillation, with freshly ignited magnesia to form magnesium lactate, and estimating the Mg as pyrophosphate.

The result of the researches shows that of the organisms employed, only *Penicillium Glaucum* decomposed tannin, and that the whole of the fermentation products are produced from the non-tannin present in the solutions. Infusions of sumac, myrabolans, and divi-divi produced large amounts of alcohol. Infusions of double strength produced twice the amount of alcohol, the increase of tannin having no effect on the fermentation.

A table, giving the principal figures obtained during the researches will be given in the April number of this Journal.—J. T. W.

PATENTS.

Cutch, Impts. in Preparing for Sale and for Use. J. R. Carruthers, Tradeston, Glasgow. Eng. Pat. 3178, Feb. 12, 1896.

See under VI., *page* 238.

Leathers, for their Better Preservation and Surface Preparation; Impts. in the Treatment of. F. B. O. Hawes, Weybridge. Eng. Pat. 3901, Feb. 20, 1896.

The leather is impregnated and surfaced with a solution of celluloid instead of the usual varnishes, &c. The resulting material is claimed to be water- and acid-proof, and also very durable.—F. H. L.

Tanning Hides, Skins, or the like: Impts. in the Process of. [*Rapid Tanning.*] J. Blunck, Warsaw, Poland. Eng. Pat. 16,764, July 28, 1896.

In this rapid tanning process, the skins or hides are unhaired and limed as usual, and are then "mordanted by the use of common or rock salt, and thus rendered suitable for the reception of the tan liquor."

The tanning liquor is made up from extracts of the usual barks, myrabolans, valonia, divi-divi, gambier, quebracho wood, &c., until it reaches a certain specific

gravity, for instance, 4° B. To it is then added common salt, until another desired specific gravity, *e.g.*, 8° B. is reached.

It is stated that, in such liquor, the skins or hides may be tanned in from 1–8 days without any agitation at all, whether of skins or vats, being necessary; the quantity of salt solution which is to be added to the tan liquor being "regulated according to requirements," and the duration of the resulting tannage being thereby "shortened at will." —E. R. B.

Rapid Tanning of Hides, Process and Apparatus for the, applicable also for Dyeing and otherwise treating Leather. H. L. J. Roy, Paris, France. Eng. Pat. 2572, Feb. 4, 1896 (date claimed under sect. 103 of Act, July 4, 1895).

The plant described consists of a broad endless band, carried upon and moved by, a pair of revolving drums, and since the band has to sustain great weight, suitably placed rollers assist in its support. The band is made up of rods, disposed transversely to its travel and connected together at their ends by links. Upon the rods are threaded loops, which, in their turn, are attached to suitable parts of the hides.

The whole plant is arranged above a pit containing the tanning liquor, and, when the apparatus is in motion, the hides follow the travel of the band; depending from it into the tanning liquor, from its lower limb, lying upon its upper limb, and descending and ascending at the two ends respectively. When the band descends at the front end of the plant, the hides would slip down heavily towards the pit with injurious consequences, were it not that, here, spring rollers keep the hides against the descending band

To control the temperature of the tanning liquor and to ensure its circulation in the pit, the patentee employs a "thermo-siphon." This is a U-tube, one of the limbs of which receives liquor from the bottom of the pit, while the other returns it to the top of the pit; the latter limb is heated.

The tannage may also be assisted by electric action. In this case the rollers of the lower limb of the band are in contact with copper strips attached to the framework of the plant, and the rods and loops constructed of materials which are conductors, the anode being placed in the pit.

As to his process and its *rationale*, the inventor remarks that, while securing rapidity of tanning, he has succeeded in preserving, as far as possible, the beneficial effects of the ordinary hand operations of tanning, *viz.*, the lifting of the hides from the pits, the laying of them one upon the other, and the replacing of them in the pits. He objects to the use of revolving drums and barrels conjointly with strong tanning liquors, as in other rapid tanning processes, stating that though leather of great suppleness may be obtained by these processes, yet that such leather is porous and spongy, in consequence of the irregular movements and violent shocks which attend such processes, tending to break or to injure the fibres of the hides. With most heavy leathers, it is necessary to preserve the initial stiffness and strength of the fibres, and it has been found necessary therefore to still adhere to the old costly, laborious, and slow method; no assistance, in the case of these leathers, having hitherto, in the patentee's opinion, been rendered by modern rapid tanning processes.

As to the rapidity of his process, the patentee states that, in ordinary tanning, it is usually considered necessary to lift and replace a hide 100 times during 30—45 days, while, with his machine, using suitable tanning liquor made up from extracts to strengths of 0°—6° B., with the assistance of heat, and in some cases with that of electricity, an ox hide of average weight can be readily tanned in 48 hours; hides in the crust, split hides, and horse hides in 24 hours; and calf skins in six hours. When working with a linear speed of 18 metres per hour, 120 hides, it is said, may be treated at once; so that one machine, working only by day, could readily tan 1,000 hides per month.

The inventor also describes another form of machine with only one drum and no endless band. Here the hides are attached to wood strips, which form the periphery of the revolving drum.

The inventor also claims the use of the machines for treating skins, hides, or leathers with other liquors, *e.g.*, in dyeing and other processes.—E. R. B.

XV.—MANURES, Etc.

PATENTS.

Apatite and other Mineral Phosphates, Impts. in the Treatment of, and Manufacture therefrom of Products suitable for Fertilising Purposes. J. G. Wiborgh, Stockholm, Sweden. Eng. Pat. 2678, Feb. 5, 1896.

The apatite, or other phosphate, is pulverised and mixed with a sufficient quantity of caustic alkali or alkaline carbonate, and the mixture subsequently heated to a red or yellow heat in a suitable oven in order to get a tetracalcium-sodium (or potassium) phosphate produced according to the following formula:—

$$\underbrace{(9CaO.3P_2O_5 + CaFl_2)}_{\text{Apatite.}} + 3Na_2CO_3 =$$
$$(10CaO.2Na_2O.3P_2O_5) + 2NaFl + 3CO_2.$$

About 20 per cent. of carbonate of sodium is required for a pure apatite; should felspar (or silicic acid) be present, more alkali, up to 30 per cent., must be added.

The tetracalcium-sodium phosphate is insoluble in water, but is easily soluble in Wagner's ammonium citrate solution.—J. T. C.

Superphosphates, Process and Apparatus for the Drying and Disintegration of. O. Heymann and A. Nitsch, Breslau, Germany. Eng. Pat. 24,776, Nov. 5, 1896.

The large moist lumps produced by the action of sulphuric acid on raw phosphates are, as soon as formed and while still hot (about 96° C.), lifted by means of an elevator and transferred through a shoot into a revolving disintegrating cylinder, the shell of which is formed of wire netting of suitable mesh.

Hot air may be introduced into the cylinders, and the angle at which the cylinders are placed during the operation is adjusted according to the nature of the material treated.

The dried and disintegrated material passes through the meshes of the cylinder wall and is conveyed away through a second shoot, or, if necessary, into a second rotating cylinder, in which it is reduced to any desired degree of fineness.—J. T. C.

XVI.—SUGAR, STARCH, GUM, Etc.

Cane Juice, Nature of the Reducing Substances of. H. Pellet. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 562—568.

In a recent paper (this Journal, 1896, 730), Prinsen Geerligs maintains that for the study of the reducing substances present in the sugar cane, the fresh juice and not the molasses, which may contain sugar inverted during the manufacture, should be employed. He concludes that in the juice of unripe canes containing little saccharose the reducing bodies are composed of dextrose and levulose in approximately equal quantities, but that the quantity of levulose diminishes with maturity, so that in juice from ripe canes there are only traces of levulose. The present author explains that there are molasses in which the proportion of glucose arising from the transformation of sugar during working corresponds to only 16 or 18 per cent. of the total glucose, and at times much less; that such molasses give, by Pellet's method, notable quantities of levulose, which quantities correspond, moreover, to those necessary to form invert sugar with the dextrose present. The use of basic acetate of lead in all his experiments has vitiated the results of Prinsen Geerligs, in spite of subsequent treatment with sulphate of soda; for solutions containing invert sugar show higher dextro-rotations in proportion as more acetate of lead is used. But by using normal acetate of lead for the clarification of cane juice, and taking account of the action of raffinose, the reducing bodies are found to have the same effect on polarised light as invert sugar.—L. J. de W.

Sugar Liquors, Solubility of Barium Sulphite and Sulphate in. J. Weisberg. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 560—561.

The author having studied the solubility and stability of sulphite of lime from the point of view of the sugar industry, has been led to examine the solubility of barium sulphite and sulphate. He concludes that:—

(1.) Barium sulphite is almost insoluble in water and sugar liquors (10 and 30 per cent.), both hot and cold.

(2.) Barium sulphate is even less soluble than the sulphite.—L. J. de W.

Soluble Starch and Starch Solution, Preparation of. O. Foerster. Chem. Zeit. 1897, 41.

To prepare soluble starch, boil 200—300 c.c. of water with addition of 5 c.c. of hydrochloric acid of sp. gr. 1·124, remove the flame, and add, in a thin stream, 20—25 grms. of starch rubbed to a paste with water, stirring continually. Heat with stirring, until the liquid is clear, let cool, filter, precipitate and wash with alcohol until the chlorine reaction disappears; then replace the alcohol by ether, dry in the air, and finally over sulphuric acid.

Starch solution is similarly made, using a measured quantity of acid, the neutralised and filtered solution being made up to one litre with glycerin. The stability of the solution may be increased by evaporation of excess of water after the addition of glycerin.—A. C. W.

"Osmose Water"; A Condensed Acid Water obtained by the Evaporation of. K. Anderlik. Zeits. Zuckerind. Böhmen. 1897, **21**, 287—292.

Under normal conditions, the condense waters of sugar factories show an alkaline reaction, which is due to the presence of ammonia partly combined with carbonic acid. In seasons when the meteorological conditions are unfavourable to the growth of the beetroot, the phenomenon called "loss of alkalinity" has been observed, the saturated juice, on concentration, yielding neutral, or even acid liquors, unless corrected by the addition of sufficient soda. Jesser sought to explain this loss by the presence of a larger amount of nitrogenous matter, which, acted on by lime, gave off ammonia during concentration, and the natural alkalinity being less than usual, was totally used up by the decomposition of the invert sugar. These researches led to the distinction between permanent and temporary alkalinity. Under these circumstances, the sugar manufacturer, in order to avoid acidity of his syrups, must seek to maintain, immediately on saturation, a permanent alkalinity. This may be effected either by the action of sufficient lime or by addition of sodium carbonate.

According to the statement of experts, a similar change occurs in concentrating osmose waters, which give water of condensation of an acid reaction. This acid condense water is recognised by a turbid, brownish appearance, from separation of basic iron salts of fatty acids. 100 c.c. of the water required for neutralisation 14·8 c.c. of decinormal KOH. 20 litres were neutralised with soda, concentrated to 500 c.c., and filtered. The residue was found to be essentially hydroxide of iron with a little formate, the other salts having been decomposed during the evaporation with soda. Of the acids in the filtrate, 91·24 per cent. was acetic acid, 3·23 formic acid, and 5·33 higher fatty acids. The acetic acid in one litre of the original condense water was 1·01 grm.—L. J. de W.

Certain Gums, On the Origin of the Coloration of. E. Bourquelot. J. Pharm. Chim. 1897, **5**, [6], 164.

The varying brown colour seen in a greater or less degree in most gums, is considered by the author to be due to the action of an oxidising ferment which they all contain, and which, in the presence of moisture, acts on the astringent matter in disintegrating portions of the bark they traverse; forming a soluble colouring matter which tints the exuding gums. This theory explains the fact that the whiter gums are always produced from dryer regions, or are exuded during the dry season, whilst the darker gums come from moister localities or may be obtained in the wet season. Further, in any lot of natural gum, great variation in depth of colour will be observed. The author has previously demonstrated that Senegal gum is rich in an oxidising ferment, reacting with such bodies as guaiacol and acetyl-guaiacol, creosol, and other phenols. The same or a similar ferment is found in Soudan, Cape, and Brazil gums.

It is found that solutions of these gums produce a deep coloration with astringent extracts, such as that of cinchona, and further, that small pieces of solid white gum placed in solutions of such extracts become brown and lose their transparency, resembling the brown pieces of natural dark gums. Many of these natural coloured gums give evidence of a trace of a tannin, and if extracted with alcohol, the solution will give the familiar reaction with ferric chloride. White gums give no such reaction.—J. O. B.

Optically Active Substances in Beetroots, Presence and Estimation of. F. Herles. Zeits. Zuckerind. Böhmen, 1897, **21**, 302.

See under XXIII., *page* 264.

Reducing Sugars, Determination of, in Terms of Cupric Oxide. G. Defrew. J. Amer. Chem. Soc. 1896, **18**, 749.

See under XXIII., *page* 264.

Starch, Conditions affecting the Volumetric Estimation of, by Means of a Solution of Iodine. F. T. Littleton. Amer. Chem. J. 1897, **19**, 44—49.

See under XXIII., *page* 264.

Gums for Thickening, New. Ch. Gassmann. Färber Zeit. **8**, 2—4.

See under VI., *page* 237.

PATENTS.

Sugar and other Syrups, Impts. in and Relating to Boiling of. E. Shaw, Bristol. Eng. Pat. 1960, Jan. 28, 1896.

This invention relates to apparatus for boiling sugar, glucose, and like syrups through the agency of steam. It is well known that it is more advantageous in sugar boiling to apply heat more rapidly at particular stages in the boiling process than at others, and, further, to vary the degree of heat according to the class of syrup being boiled. These ends are facilitated and more effectually performed by the provision in the steam supply-pipe to the jacket of the pan and to the coil, or either, of a reducing valve whereby the pressure, and consequently the temperature, of the steam is capable of being regulated and varied at will from the full working pressure down to any desired degree. —L. J. de W.

Molasses and other Saccharine Liquors, Process for the Extraction of Sugar from, by Means of Barium Hydroxy-Sulphide, and the Regeneration of the Latter from the By-products formed. W. Feld, Linz am Rhein, Germany. Eng. Pat. 6508, March 24, 1896.

When caustic baryta is used for extracting sugar from molasses, there is a difficulty in regenerating it from the carbonate obtained. The present invention offers an agent capable of being easily regenerated.

Barium hydroxysulphide added to molasses at 60° to 100° C. precipitates barium saccharate, $BaO.C_{12}H_{22}O_{11}$, leaving barium hydrosulphide in solution. After filtration, the saccharate is decomposed by sulphurous or sulphuric acid, or if carbonic acid is used, the carbonate is afterwards converted into the sulphite or sulphate. The lye separated from the saccharate is treated with carbonic acid, yielding sulphuretted hydrogen, which is burnt to sulphurous or sulphuric acid. If the available carbonic acid is so weak that the sulphuretted hydrogen would not burn, the hot lye is treated with magnesium sulphite or sulphate. The precipitate of barium salt and magnesium hydrate is treated with carbonic acid, as the magnesium carbonate filters more easily. It is then converted into sulphite or sulphate, and thus separated from the barium sulphite or sulphate for use again.

The barium sulphite or sulphate obtained in any stage of the above process is heated to redness with charcoal. The melt obtained, on treatment with water, yields again barium hydroxysulphide.—L. J. de W.

Molasses of Raw Beet-Sugar, Process for the Production of a Food of a High Nutritive Value from. J. Hensel, Hermsdorf-Kynast, Germany. Eng. Pat. 24,038, Oct. 28, 1896.

This new process for preparing from the molasses of raw beet-sugar, a foodstuff of high nutritive value applicable for men and animals, consists principally in treating the molasses with phosphoric acid or phosphates, and at the same time neutralising the excess of free phosphoric acid by the addition of bicarbonate of soda, in order to transform into nutritive salts the mineral stuffs, especially the carbonates, which are contained in the molasses. For this purpose, to 100 parts of the molasses, 10 parts of phosphoric acid are added, having the sp. gr. 1,200. The phosphated molasses can be mixed with paste and baked as a nourishing bread, or dissolved in hot water as a beverage to take the place of coffee.—L. J. de W.

Sugar, Impts. in or relating to the Production of. L. Janssens and Co., Brussels. Eng. Pat. 28,188, Dec. 9, 1896.

Ground and sifted raw sugar is covered with 35 to 30 per cent. of concentrated and decolorised liquor produced at a temperature of from 30° to 70° C., both being heated, and then centrifugalised, when a beautiful crystalline sugar of fine grain, is obtained, containing 2 to 4 per cent. of water. To this porous and loose sugar (according to the amount of water in it), 20 to 30 per cent. of ground sugar is added, filling up the pores and giving a beautiful white appearance, though a little duller than the crystalline sugar. This product is pressed in moulds having interchangeable wooden supports on which are placed metallic plates by which the cracking of the sugar loaf is avoided. "That part of the process, which relates to the further treatment of centrifugal sugar by mixing it with ground sugar, is not new in itself, but the pressing process for moulding sugar is totally different from processes used hitherto."—L. J. de W.

XVII.—BREWING, WINES, SPIRITS, Etc.

Barley Plant, The Chemistry of the, with reference to its Carbohydrate Constituents. C. F. Cross and E. J. Bevan. J. Fed. Inst. Brewing, 1897, **3**, 2—18.

The authors give a brief account of their studies on special points in the chemical life-history of the barley plant, the purpose of which was chiefly to solve the constitution of a particular group of carbohydrates present in large quantities in the cereal straws, and which, from the fact that they yield furfural under the action of acids, may be called furfuroids. These carbohydrates constitute about one quarter of the substance of the mature straw. They are not acted on by alkalis, but when digested with 1 per cent. sulphuric acid under a pressure of three atmospheres, they are selectively hydrolysed, yielding principally formal pentoses. The acid solution containing the furfuroids is neutralised and filtered, and it is found that under suitable conditions it ferments freely with yeast. After the action is complete, it will be noticed, however, that only a certain proportion of the carbohydrates have been destroyed. This partial resemblance to dextrose will, nevertheless, establish for the group a certain claim upon the attention of practical men, which will be increased when it is borne in mind that the seed envelopes of the barley have much the same composition as straw, and that they contain a proportion of easily hydrolysable furfuroids, which find their way into the wort during the mashing process. By far the greater proportion resist this treatment, and are present in the "spent grains." Satisfactory results are said to have been obtained by treating such grains with acid, according to the instructions given in the Eng. Pat. 1895, 15,531. Some of these results are given in the paper.—J. L. B.

Diastase, Hypothesis of a Saccharogenic, in the Beetroot. E. Barbet. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896, **14**, 582—587.

The difficulty of fermenting beetroot juice when the roots are not ripe, led the author to examine the hypothesis of a saccharogenic diastase in the root in direct conflict with the invertase of yeast, which is saccharophagic, and without the agency of which the fermentation of saccharose is impossible (Bull. Assoc. Chim. **14**, 223).

As the leaves of the beetroot turn yellow and fall at the first touch of frost, and the root from this moment ceases to grow, its richness in sugar having reached a maximum, it is to be supposed that the saccharogenic diastase, if such there be, is very impressionable to cold, and ceases to act in frost. The author found that samples of juice after being frozen, or juice extracted from frozen beetroots, fermented as well as the same juice which had been boiled and cooled before the addition of sulphuric acid, and the commencement of the fermentation was more rapid, juice not thus treated being left far behind.—L. J. de W.

Malt and Wort, Acids of. H. Johnson. Petit Journ. du Brasseur, 1896, 328.

The author has made a long series of experiments to ascertain whether lactic acid is formed during the mashing of malt or not. The acidity produced during mashing increases with the temperature of mashing from 20° C. (68° F.) to 50° C. (122° F.), but decreases above 60° C. (140° F.). Thus, in a mash carried out at 70° C. (168° F.) less acid is produced than in a mash of the same materials and proportions at 40°, 50°, or 60° C. When a small quantity of acid wort or sour milk was added to the mash, the acid formation was about doubled at 40° C., but was not increased at all above the normal at 50° or 60° C. This latter fact shows that the formation of lactic acid is active at 40° C., but ceases altogether at 50° C., and that therefore the normal formation of acid during mashing, which increases up to 50° C. cannot be due to a lactic fermentation. The addition of alcohol (10 per cent.) and of chloroform (1 per cent.) to the mash completely stops the extra formation of acid by the addition of sour milk or sour wort, but has little or no influence on the normal acid formation. The fact that the acid formation reaches its maximum at 50° C. shows that it cannot be due to simple extraction from the malt. The author believes that the acidity produced during mashing is due to a chemical or diastatic action (ceasing above about 70° C.) on some of the albuminoid or nitrogenous matters in the malt, with the formation of acid compounds more or less of the type of asparagine $COOH.CH(NH_2).CH_2.CO(NH_2)$, or asparagic acid—

$$COOH.CH(NH_2).CH_2.COOH.$$

Experiments with asparagine and asparagic acid showed that these compounds behaved with alkalis and with various indicators very similarly to the behaviour observed in malt worts with those reagents.—L. T. T.

Yeasts, Difference between Low and High Fermentation. P. Petit. Comptes Rend. 1897, 93—94.

The author grows the two varieties of yeast in a dextrose solution containing both ammonium phosphate and asparagine, and determines what amount of these constituents is removed by each yeast. He finds that the high fermentation yeast removed double as much asparagine as the low fermentation yeast.—A. L. S.

Wine, Isolation by Steam Distillation and subsequent Estimation of the Glycerin in. F. Bordas and S. de Raczkowski. Comptes Rend. 1897, **124**, 240—242.

50 c.c. of wine, previously neutralised with potash, are placed in a bulb-flask (300 c.c.), joined to two Woulff's bottles, which serve as receivers. The flask is submerged in a bath of saturated salt solution, the latter heated to 110° C., and a current of air aspirated through the apparatus until the wine has been evaporated to dryness. Subsequently, the air supply is somewhat reduced, and steam blown

through the flask for about three hours, after which time the whole of the glycerin will have passed into the receivers, whilst the alcohol and other more volatile constituents of the wine are expelled from the distillate, by reason of its high temperature. The combined distillates are diluted to 500 c.c. and the glycerol estimated by titration with a standard solution of potassium bichromate (24 grms. per litre) in the manner previously described (Comptes Rend., Dec. 1896). As compared with Pasteur's process, the new method yields somewhat higher results.—H. T. P.

Mashing Thicker. W. Windisch. Wochenschr. f. Brauerei, 1897, 9.

The author recommends German brewers to mash thicker for the following reasons:—

(1.) Less first wort is obtained and consequently more sparging water can be used and a better extract obtained.

(2.) The amount of mash is less and consequently there is less to heat in the mash copper, and the temperature can be raised quicker.

(3.) In concentrated mashes the diastase is better able to resist high temperatures.

(4.) Less of the undesirable constituents of the malt are dissolved in thick mashes.

(5.) Less wort is obtained and consequently less coal is consumed in the copper, and the boiling is sooner finished.
—A. L. S.

Beer, Relation between the Degree of Fermentation and the Keeping Properties of. W. Windisch. Wochenschr. f. Brauerei, 1896, **13**, 1362.

In a brewery working without an ice-machine, the fermentation was habitually carried from 12·6 per cent. to 3·4 per cent., *i.e.*, to the high fermentation degree of 73 per cent. The beer, however, did not taste thin. After 18 days there was a slight deposit of yeast cells and the supernatant liquor kept in good condition for four months. An ice apparatus was then put in, and the temperature of the fermentation room thereby so reduced that it was only found practicable to carry the fermentation from 12·6 per cent. to 4·4 per cent. (corresponding to 65 per cent.). The beer was now found to become slightly cloudy at the end of eight days, and did not subsequently become clear. The cloud consisted of wild yeasts.—L. T. T.

Brandies, Results of Analysis of, and Conditions of Ageing. F. Lusson. Bull. de l'Assoc. des Chim. de Suer. et de Dist. 1896, **14**, 587—591.

Following the method given by Girard, Dupré, and Saglier, in the *Encyclopédie chimique*, published in 1894, and adopted by the Municipal Laboratory of Paris, for the determination of the various impurities in alcohol, the author has analysed a number of samples of brandy of known origin and age so as to obtain average values; the results promising to be of interest both from the commercial and the hygienic point of view.

In these analyses nitrogenous matters in the form of ammonia and pyridine bases, are neglected as of only secondary interest, the tables showing acids (calculated to acetic acid), aldehydes (as acetic aldehyde), also furfural, ethers (as ethyl acetate), and higher alcohols (as isobutylic alcohol), and giving the number of mgrms. of each per 100 c.c. of absolute alcohol in order to avoid taking account, in each case, of the strength of the brandy. The sum of these impurities gives the *coefficient of impurity*. As it is not sufficient to consider the coefficient of impurity to know the value of a brandy, the percentage of the products of simple oxidation, acids, and aldehydes, on the total impurities, is given as the *coefficient of oxidation*. It was found that all the samples of pure brandy, Aunis, Charente, and Algeria, show—a coefficient of impurity higher than 340—a total of ethers and alcohols over 300—a coefficient of oxidation varying from 11 to 36 for products of 1 to 40 years without being exactly proportional to age. Other products (cognacs, eaux-de-vie, &c.) of good quality, the purity of which was not guaranteed, but which may be considered as good, if not absolutely pure, gave coefficients of impurity ranging from 340 to 300, and a total of ethers and alcohols between 300 and 250.

The ordinary neuter alcohols of commerce of good flavour are 10 times purer than true brandies. Commercial products obtained from industrial alcohol rendered aromatic by brandy from natural wines are therefore less harmful than brandy itself, and as it is difficult to maintain that among good old cognacs the most impure are not beneficial, the conclusion cannot be avoided that the ravages of alcoholism are simply due to the abuse of alcohol itself.—L. J. de W.

Fusel Oil, Bases contained in. E. Bamberger and A. Einhorn. Ber. 30, 224—229.

The authors call attention to the danger of pyridine bases being contained in fusel oil. They have examined the amyl alcohol with which they were working, and purchased as pure, and they have separated in a pure condition the two bases, pyridine and 2, 5-dimethylpyridine, and have indications of the existence of others of the same series.—A. L. S.

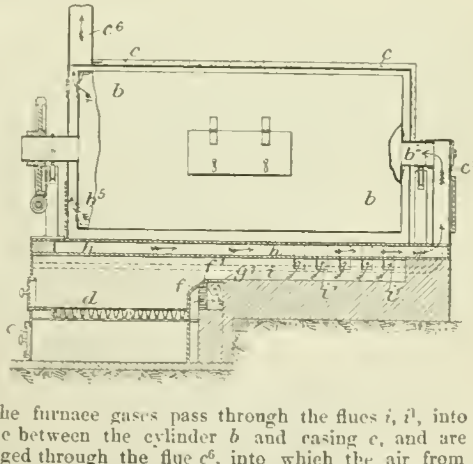
Large Spirit Vats, The Cleaning of: A Warning. Zeits. für Spiritusind. **22**, 27.

See under XVIII. B., *page* 254.

PATENTS.

Brewers' Refuse and other Materials or Substances Requiring Similar Treatment, Impts. in and connected with Machines for Drying. R. Cunliffe, Weaste, Lancs. Eng. Pat. 2150, Jan. 30, 1896.

The machines are of the type wherein the material or substance is dried in a cylinder adapted to rotate inside a casing over a furnace. Air is admitted, in regulated quantity, through doors c into two lateral chambers, and is heated in two separate volumes, one passing through the hollow fire-bars *d*, and the other through the hollow fire-bridge *f*, perforated partition f^1, and tube g^1. The heated air passes through the hot-air chamber *h*, lateral casing c^1, and hollow trunnion b^2, into the cylinder *b*, which is revolved by hand or power.



The furnace gases pass through the flues *i*, i^1, into the space between the cylinder *b* and casing *c*, and are discharged through the flue c^6, into which the air from the cylinder *b* also passes through the apertures b^5. In the form shown, the cylinder is charged and emptied through doors in the cylinder and casing. In a modification, the material is fed automatically from a hopper, which is placed against the chimney of the apparatus, so that the material is heated before entering the cylinder. The chimney is bifurcated, the branches communicating separately with the cylinder and its casing.—R. A.

XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

(A.)—FOODS.

Albuminoids in Substances Rich in Starch, A Modification of Stutzer's Method for the Estimation of. H. Tryller. Chem. Zeit. 1897, **21**, 54.

See under XXIII., *page* 264.

PATENTS.

Lactic Acid Bacteria, An Improved Process for Raising Permanent Pure Cultivations of. H. H. Lake, London. From L. v. Lorentz, Hamburg, Germany. Eng. Pat. 7898, April 14, 1896.

With a view of preserving cultivations of lactic acid bacteria such as are used for acidulating cream for the preparation of butter, the patentee mixes a pure cultivation with a solution of 4 grms. of milk sugar in 100 c.c. of water. Of this cultivation, called a "fluid cultivation," 200 grms. are mixed with 5 grms. of milk sugar and preserved in sterilised vessels, holding about 250 c.c., which are then sealed with sterilised stoppers or corks. In order to preserve the preparation in the form of powder, 200 grms. of potato starch, 50 grms. of milk sugar, and 75 grms. of the above fluid cultivation are mixed together and dried at 30° C., the powdered material being preserved in well sterilised glasses. The cultivation can also be kept in a solid compact form by preparing a porous cake from 60 grms. of gypsum, 20 grms. of milk sugar, and a sufficient quantity of distilled water. After drying and sterilising the cake by heating, it is cooled and impregnated in a sterilised space with 75 grms. of the fluid cultivation, dried at a temperature below 30° C., and preserved in sterilised filter paper.—T. A. L.

Molasses of Raw Beet Sugar, Process for the Production of a Food of a High Nutritive Value from. J. Hensel, Hermsdorf-Kynast, Germany. Eng. Pat. 24,038. Oct. 28, 1896.

See under XVI., *page* 252.

Vegetable Extracts, and the Method of Obtaining the same, Impts. in. A. J. Boult, London. From L. Fromm and R. Schmidt, Dresden, Germany. Eng. Pat. 25,786, Nov. 16, 1896.

This process is for the production of an extract containing both fatty and albuminous substances from vegetables, beans, nuts, &c., in a form easily soluble and assimilable, as food, containing about 50 per cent. of fat, and 25 per cent. of albumin. The vegetable substance, after being ground to a flour, is made into a paste with cold water (containing sodium chloride in the proportion of 5—10 per cent. of the flour). It is kneaded between rollers, by which treatment the fat becomes thoroughly incorporated with the albumin, which is also converted into a soluble form. The mass is then dissolved in hot water of 50°—60° C., converted into an emulsion by stirring, separated from impurities in a centrifugal machine, and finally dried by steam to a thick mass.—J. J. K.

(B.)—SANITATION; WATER PURIFICATION.

Large Spirit Vats, The Cleaning of: A Warning. Zeits. für Spiritusind. **22**, 27.

In a large distillery and cider manufactory in Saxony a serious explosion occurred in the autumn of last year under circumstances of some interest. A wooden fermenting vessel of a capacity of 18,000 litres had been standing full of 96 per cent. spirit for three months. It was then emptied and partly filled with water, and the workmen were preparing to enter it for the purpose of scrubbing the inside when a violent explosion occurred. Four workmen were burnt, two of them so severely, that death ensued in a short time.

An official inquiry showed that the timber of the vessel had absorbed a considerable quantity of spirit, of which the slight treatment with cold water had removed but little. The vapour arising from this spirit had formed an explosive mixture with the air, which had been ignited by a gas flame near at hand. The use of safety lamps in the vicinity of vessels of this description was recommended.—J. G. W.

PATENTS.

Filtration of Sewage, Impts. in Arrangements and Apparatus for. R. E. von Leugerke, London. Eng. Pat. 1038, Jan. 15, 1896.

The filtering area is divided into four equal separate filter beds, all of which have their supply and discharge pipes radiating from a central chamber in which the valves are also contained. The outlet and inlet valves of each bed are connected, so that when the outlet valve is opened by the pressure of liquid in the bed, the inlet valve closes, and the valves belonging to the different beds are also connected with each other in such a manner that the opening of the outlet valve and closing of the inlet valve of one bed, closes the outlet valve and opens the inlet valve of the next, and so on in rotation. In this way each filter bed successively becomes charged with liquid, remains charged for a time, then discharges, and finally remains empty for a time. —L. A.

Filtering and Purifying Liquids [Water, &c.], A New or Improved Method of and Apparatus for. J. G. Lorrain, Norfolk Street, Strand, W.C. Eng. Pat. 2859, Feb. 7, 1896.

The liquid is passed first through a finely perforated plate, which offers considerable resistance to the flow, then through a layer of purifying material (preferably platinised carbon, &c.) arranged so as to offer less resistance to the flow than the plate, and finally through a second perforated plate, which offers less resistance to the flow than the purifying material, air being at the same time supplied through a tube to the purifying material, in order that the oxygen of the air may combine with the oxidisable matters in the liquid. The apparatus may be provided with a straining device, through which the liquid is first passed to remove suspended matters.—R. A.

Settling Tanks or Lodges used in the Purification of Sewage and other Foul Waters, and in the Clarification of Manufacturers' Liquid Compositions; Impts. in the Construction of. J. Petrie, Rochdale, Lancaster. Eng. Pat. 3047, Feb. 11, 1896.

The tanks are constructed with smooth sloping sides, down which the suspended solids readily slide and are removed from time to time by opening a valve or valves at the bottom.—L. A.

Sewage, Impts. in Apparatus for the Treatment of. W. P. Thompson, Liverpool. From E. de Harven, Antwerp, Belgium. Eng. Pat. 5690, March 13, 1896.

The apparatus described is a cesspool composed of two separate pits, the first of which receives the whole of the liquid and solid substances, retains the solid ones, and delivers the liquid from or near its top to the second pit. The second pit is divided into four parts, by means of partitions of different height, and contains a siphon which automatically discharges a fixed proportion of the liquid into the sewer after it has circulated through all the subdivisions of the pit. At one end of this pit there is a receptacle for disinfectant, and a tilting ladle with a float, which, actuated by the rise and fall of the water level, automatically and gradually discharges a measured volume of the disinfectant into the pit while it is again filling up after the siphon has operated. This apparatus is intended to be used in connection with the water-closet system, the solid matters being retained by the first pit, and the liquid being disinfected in the second pit before passing into the sewers.—L. A.

(C.)—DISINFECTANTS.

PATENTS.

Disinfectants in Porous Cylinders, An Improved Method of using. T. Grimstone, London. Eng. Pat. 2424, Feb. 3, 1896.

Any suitable disinfectant or deodoriser is hermetically sealed within a porous pot which is placed in the w.c. flushing tank or similar receptacle. Solution of the disinfectant takes place by diffusion in a slow and regular manner. Permanganate crystals alone may be placed in the pot, but the

inventor prefers to employ "kresyline (or soluble creosote), eucalyptol, terpinol, or carbolic acid in liquid form, or any desired combination of these or their equivalents, to which is added as a basis, silica, to prevent volatile effusion," in proportions to form a thick paste.—L. A.

Sewage Matter, Composition for Disinfecting and Rendering it available for Agricultural or Fertilising Purposes. P. O'Dowd, Westport, Ireland. Eng. Pat. 5381, March 10, 1896.

The composition consists of a mixture of peat charcoal and sulphate of iron, suitable proportions being 90 to 95 parts of the former with from 10 to 5 parts of the latter.—L. A.

Insecticide, A New or Improved. J. N. Spence, Harrow, Middlesex. Eng. Pat. 5705, March 13, 1896.

This consists of a mixture of borax and sugar, preferably 3 to 1, with or without a trace of charcoal or lampblack, *e.g.* 8 oz. to 140 lb. of the borax and sugar mixture.—L. A.

XIX.—PAPER, PASTEBOARD, Etc.

Esparto Half-Stuff Manufactured in France. Papier Zeit. 1897, 214.

A factory has been started at Fos-sur-Mer (Bouches-du-Rhone) by a company trading as the "Société Anonyme des Pâtes de Cellulose." This factory is now regularly supplying half-stuff to paper-makers in France. It is noted that the importation of esparto pulp from this country to Paris alone, amounted last year to the value of 1,000,000 francs.

The following notes on the method of using it are supplied to purchasers:—The half-stuff should be soaked before adding to the beater, from 2 to 6 hours, according to its condition. In the beating the finish should be very thick, especially in papers containing a very high proportion of the esparto. In mixing with other half-stuffs it is recommended to work up the esparto separately. In bleaching it is important to avoid acidity.—C. F. C.

Parchment Paper. [*Waste Waters Treatment.*] Papier Zeit. 1896, **21**, 3390.

In the preparation of parchment papers the waste waters are very strongly acid, and are therefore not allowed to be run into streams and rivers in England. For this reason the industry, although originated in England, is no longer carried on in this country. J. S. Rigby, of Wavertree (Liverpool), has just taken out a patent by which the more strongly acid water pressed from the paper is passed through towers filled with magnesite, and the more dilute washings are treated with crude calcium chloride. In this way he hopes to obtain the marketable products, magnesium sulphate, gypsum, and pure carbonic acid, and at the same time purify the waste waters sufficiently for their discharge into streams, &c.—L. T. T.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

"Lunar Caustic," Separation of Nitrate of Copper from Nitrate of Silver in the Manufacture of. C. J. H. Warden. Pharm. J. 1897, **58**, [1387], 61.

In the manufacture of lunar caustic at the Calcutta Medical Depôt, the silver, which always contains a certain amount of copper, is dissolved in nitric acid, and, after separation of the gold, as much as possible of the nitrate of silver is crystallised out. The deep blue mother-liquor is evaporated to dryness and the residue powdered and placed in a glass funnel stopped with a plug of asbestos, where it is treated with nitric acid of sp. gr. 1·42. The nitric acid dissolves the whole of the nitrate of copper, while only a very small amount of silver nitrate is dissolved. The nitric acid can be recovered by distillation, and the small amount of silver separated from the nitrate of copper by precipitation with salt. By this method a salt is obtained practically free from copper.—A. S.

Mercury Pyroborate. Dupuoy. Bull. de la Soc. de Pharm. de Bord. **36**, 269; and Pharm. J. 1897, **58**, 82.

Tokayer (Pharm. Post. **25**,156) obtained, by precipitating an aqueous solution of mercuric chloride with borax, a brownish-red precipitate, which he regarded as mercury pyroborate, HgB_4O_7. This substance has recently been successfully employed as an antiseptic. The author, on examining the salt, found it contained no boric acid, but was a basic oxychloride of mercury having the formula $HgCl_2.3HgO$. This oxychloride may also be prepared by treating a boiling solution of mercuric chloride with sodium carbonate, keeping the mercuric chloride in excess, to avoid formation of other oxychlorides.—A. S.

Mercury Benzoate. Rebière. Bull. de la Soc. de Pharm. de Bord. **36**, 280; and Pharm. J. 1897, **58**, 82.

The author prepared this salt in a pure state by the following method:—A known quantity of pure mercuric chloride was precipitated by soda and the precipitate washed until free from chlorine. From the amount of mercuric chloride taken, the amount of oxide was calculated, and sufficient finely powdered benzoic acid mixed with it to leave a slight excess of HgO. The mixture, diluted with water, was left for 24 hours and then heated to boiling; a white amorphous powder resulted, which, when again heated with a large volume of boiling water, redissolved, and on cooling crystallised out in long silky needles, which were drained and dried at the ordinary temperature. These crystals had the definite composition $Hg(C_7H_5O_2)_2$.—A. S.

Bismuth Benzoate. Rebière. Bull. de la Soc. de Pharm. de Bord. **36**, 272; and Pharm. J. 1897, **58**, 82.

For the preparation of the basic benzoate of bismuth, $BiO.C_7H_5O_2$, which is the French official salt, the author uses the following method:—Freshly precipitated oxide of bismuth, thoroughly washed, is drained to a pasty consistence The amount of anhydrous Bi_2O_3 is determined in a portion of the paste, and to the rest is added the calculated quantity of finely powdered benzoic acid, to produce the salt $BiO.C_7H_5O_2$. The mixture is diluted with sufficient water to make it fluid, well mixed, and left in contact for 24 hours. The precipitate is then thrown on a cloth, drained, and dried in the air.—A. S.

[*Menthol and Menthone.*] *Researches in the Camphor Series.* E. Beckmann. J. Prakt. Chem. 1897, **55**, 14.

The menthol used had the melting point 43° C. and specific rotation $[\alpha]_D = -49·3°$ in 20 per cent. alcoholic solution.

Menthol sodium is obtained by heating the theoretical proportions of the components at 200°, in an atmosphere of hydrogen. The mass becomes solid, melts at a higher temperature with partial decomposition, and soon changes in the air, even when cold.

Benzoic acid ester may be made by heating with benzoic anhydride at 160°—170° for 2—3 hours. The ester melts at 54·5°, in 20 per cent. alcoholic solution $[\alpha]_D = -90·72°$. On the water-bath and with a current of steam the compound is very slowly volatile; menthol and menthone may thus be readily separated.

Stearic Acid Ester.—Stearic anhydride is obtained by the action of phosphorus oxychloride on dry sodium stearate (from commercial acid) suspended in benzene, and evaporation of the benzene after filtering. The stearic ester, prepared in a similar manner to the benzoic ester, may, on account of its difficult volatility, serve for the isolation of this alcohol (and others). Menthol stearate melts at 39° C.

Separation of Menthol and Menthone.—This may be accomplished by means of the oxime; the method may be used for the extraction of menthone from ethereal oils. After formation of the oxime by heating in alcoholic solution with hydroxylamine and sodium bicarbonate on the water-bath for 15 minutes, addition of water, extraction by ether, and evaporation of the solvent, a mixture of menthol and *l*-menthone oxime is obtained. From this mixture four extractions with dilute sulphuric acid (sp. gr. 1·17) extract the oxime and leave menthol of normal melting point and rotation. The oxime is obtained by saturation with soda

after addition of ice, and extraction by ether after passing carbon dioxide through the solution. The oxime is usually not quite solid, and melts at 47°—49°, instead of 58°; it probably contains a little menthol.—A. C. W.

Camphor Series, Researches in. [*Borneol and Reduction of Camphor.*] E. Beckmann. J. Prakt. Chem. 1897, **55**, 31.

In the reduction of camphor, borneol is formed in quantities equal to or greater than those of isoborneol. The amounts of each were calculated from the observed rotation of the mixture. In the case of reduction in alcoholic solution (ethyl or amyl alcohols, phenol) isoborneol forms 16—21 per cent. of the product. In indifferent solvents (toluene, petroleum ether, ether) 43—47 per cent. of isoborneol is formed. The mixtures may be separated by the process described for menthone and menthol in the preceding abstract, or more simply by passing in carbon dioxide, shaking with ice-cold water, and separation of dissolved borneol, by acidifying with sulphuric acid and addition of alkali, or by standing 8—14 days. All mixtures produced in non-alcoholic media contain about 5 per cent. camphor-pinacone, which is left behind on steam distillation.

—A. C. W.

Caffeïdine-Carbonic Acid. E. Fischer and O. Bromberg. Ber. 1897, **30**, 219.

By agitating caffeïne with dilute alkali at ordinary temperatures for three or four days in a mechanical agitator, the authors have been able to convert the alkaloid into caffeïdine-carbonic acid in a materially shorter time than that required by the original method of Maly and Andreasch (Monatsh. für Chem. **4**, 369). The acid so formed is separated as a copper salt, the metal removed with H_2S, and the acid solution evaporated *in vacuo* at 40°. In small quantities the acid separates readily in crystalline form, but in larger quantities it remains uncrystallised in a syrupy condition; if this syrup be dissolved in glacial acetic acid, and this be treated with four times its volume of benzene, large colourless crystals, which melt between 127°—130°, are obtained; these appear to consist of a compound of caffeïdine-carbonic acid and acetic acid. The pure acid is obtained from this body by dissolving in 30 parts of boiling acetone; on cooling, caffeïdine-carbonic acid separates in long colourless prismatic needles, which have, after drying *in vacuo*, the formula $C_8H_{12}N_4O_3$. When heated in sealed tubes with phosphorus oxychloride in an oil-bath for three hours, the resulting syrupy liquid obtained by concentration on the water-bath gave, when treated with an excess of potash, a crystalline mass; this, on extraction with chloroform, proved to be caffeïne.—J. O. B.

Sparteïne Derivatives. F. Ahrens. Ber. 1897, **30**, 195—200.

The fact that oxysparteïne and dihydroxysparteïne, the chief products of the oxidation of sparteïne, differ markedly in their physiological action on the heart—oxysparteïne imparting greater power to the pulsation, while the actual number of beats is decreased, whereas with dihydroxysparteïne the action appears to be exactly contrary,—has induced the author to investigate the subject. He finds that an aldehyde group exists in oxysparteïne and two hydroxyl groups in dihydroxysparteïne. The hydrochloride of this base had the composition $C_{15}H_{24}N_2.2HCl + 3\frac{1}{2}H_2O$, and occurred as large, well-formed, colourless crystals. The hydriodide, $C_{15}H_{24}N_2HI$, obtained by neutralising the alcoholic solution of the base with hydriodic acid, gave shining scales, which melted at 225°—226°; excess of acid gave the corresponding acid salt, $C_{15}H_{24}N_2.2HI$, in large, clear crystals, melting at 256°—258°.

The formula for oxysparteïne is $C_{14}H_{23}N_2CHO$.

When treated with a large excess of hydrogen peroxide and left for some weeks, the result was a slow oxidation of oxysparteïne and the formation of a crystalline acid, which melted at 287°—289°, and had the composition $C_{10}H_{16}NO_2 + 2H_2O$, giving crystalline soluble salts with alkalis and alkaline earths.—J. O. B.

Piperidine Guaiacolate. Schidrowitz. Pharm. J. 1897, **58**, 81; Brit. Med. J., No. 1881, 136.

This compound, $C_5H_{11}NC_7H_8O_2$, was prepared by the action of piperidine on guaiacol dissolved in benzene or petroleum ether. It crystallises in prismatic needles or plates, melts at 79·81° C., is soluble in water to the extent of 3·5 per cent., and also easily soluble in most organic solvents. Mineral acids and alkalis decompose it into its constituents. It is used in the treatment of phthisis.—A. S.

Eckart's Rhodinol, Properties of, and its Technical Production from Pelargonium Oils. R. Pfister. Chem. Zeit. 1897, 38.

Monnet and Barbier have shown that the alcohol, rhodinol, $C_{10}H_{17}.OH$, the odoriferous principle of otto of roses, is contained in the true geranium oils distilled from the pelargonium species. The name geraniol had already been given to an alcohol of unpleasant odour contained in palmarosa oil. According to Ger. Pat. 80,007, by the Société Chimique des Usines du Rhône, rhodinol is obtained from the essential oils of pelargonium (geranium) varieties. The pelargonium oil is carefully fractionated under as low a pressure as possible, when a rhodinol boiling within a few degrees, but not quite pure, is obtained. This is heated in an autoclave for eight hours at 140°—150° C. with acetic anhydride, by which quantitative esterification is brought about. By fractionation, the rhodinol acetate is now separated from the non-alcoholic impurities, which have the same boiling point as the alcohol. After saponification and further fractional distillation *in vacuo*, rhodinol is obtained as a colourless mobile oil of constant boiling point. For use, this rhodinol is diluted with citronellol, as in the natural rose oils, since in the pure state it rapidly alters and loses its pleasant odour. Mixed with citronellol, rhodinol may be distilled *in vacuo* and obtained free from other admixture, but if distilled alone, traces of decomposition products are formed, and render the product useless for perfumery.

For controversy as to the composition of rhodinol, geraniol, and réuniol, and the use of these names, see this Journal, 1896, 292, 373, 469, 827.—A. C. W.

Otto of Roses, Methods of Examining. R. Jedermann. Zeits. Anal. Chem. 1897, 96.

The author, in common with most modern authorities, regards the physical characters of otto of roses, as well as its chemical reactions, of considerable value in its critical examination. He finds the pharmacopœial tests as given in the official works of most countries to be quite valueless, and specially comments on the fallacious deductions which may be made from the modification of Hager's test, contained in the United States Pharmacopœia; the author finds that perfectly pure samples of otto will not respond to this test, the disturbing substance being a trace of resinous matter, derived from the green sepals of the flowers, traces of which are sometimes carried over by the steam in the process of distillation; when the roses expand quickly, as was the case last year, the process of distillation has to be carried on so rapidly that time is not available for the elimination of the green outer portions of the rose flowers, so that the otto produced contains a minute trace of this resinoid body.

The preference shown for ottos having a high congealing point is regarded as fallacious, since the amount of odourless stearoptene in the oil is dependent on the conditions under which the otto is distilled; moreover, this preference on the part of buyers for an oil rich in stearoptene induces the distillers to produce the same at the sacrifice of the aroma. The optical rotation is a useful factor: pure oil will give a rotation of 43°.30′ at 30° C. in a Wild's polaristrobometer. The specific gravity is also useful, lying between 0·8555 and 0·8645 at 30° C., the lighter oils being naturally rich in stearoptene.—J. O. B.

Commercial Civet. J. O. Braithwaite. Pharm. J. [4], **4**, 101.

The author has examined three samples of commercial civet, and finds them all to be grossly adulterated with either added fat, water, fæcal matter, or sugar, or other

foreign organic matter. No published data of any value being available, a sample of the natural secretion from the civet cats in the Zoological Society's Gardens was obtained. Except for accidental impurities, mainly hairs of the animal and sawdust from its cage, this product was almost wholly soluble when extracted in a Soxhlet apparatus with light petroleum ether. The insoluble residue was almost free from odour, and dry. The commercial samples gave from 70 to 50 per cent. of matter soluble in this solvent, and the residues in all cases were stercoraceous in odour. One—the worst—also contained much sugar. Another lost 30 per cent. on drying, showing at the same time water globules under the microscope. The total acid number of the standard sample was 140, and the volatile acid number 32·3; all the commercial samples deviated markedly from these figures, that for the volatile acid being more than 60 per cent. too low.—J. O. B.

Artificial Pulegone from Citronellal. F. Tiemann and R. Schmidt. Ber. **30**, 22—32.

In a previous paper (Ber. **29**, 903) the authors showed that citronellol—

$(CH_3)_2:C:CH.CH_2.CH_2.CH(CH_3).CH_2.CH_2OH$,

citronellal—

$(CH_3)_2:C:CH.CH_2.CH_2.CH(CH_3)CH_2.COH$,

and citronellic acid—

$(CH_3)_2:C:CH.CH_2.CH_2.CH(CH_3).CH_2.CO_2H$,

are compounds of the aliphatic series. They also pointed out that under the action of acetic anhydride, citronellal was converted into a secondary alcohol of the formula $C_{10}H_{18}O$, which they called isopulegol, and converted by oxidising agents into the corresponding ketone, $C_{10}H_{16}O$, designated isopulegone. The former compound is obtained by heating 150 grms. of citronellal with 100 grms. of acetic anhydride for 20 hours in an oil-bath to 160°—180° C. The melt is taken up with alcohol, treated with 150 grms. of caustic potash and steamed, when isopulegol passes over, giving a yield of 66 per cent. on the weight of the citronellal. The oxidation to isopulegone is effected by emulsifying 100 grms. of isopulegol in 1 litre of water and gradually adding a hot solution of 150 grms. of potassium bichromate, 200 grms. of concentrated sulphuric acid, and 600 c.c. of water during about half an hour, after which the crude isopulegone is at once driven off with steam. For purification, it is dissolved in dilute alcohol and treated with semicarbazide hydrochloride in sodium acetate solution, when, after a few hours, isopulegone semicarbazone crystallises out and is further crystallised from benzene. This compound is then dissolved in alcohol, decomposed with sulphuric acid, filtered from semicarbazide sulphate, and the filtrate extracted with ether, the residue therefrom being steamed, when pure isopulegone passes over, distilling under 12 mm. pressure at 90° C. The product does not give a crystalline compound with sodium bisulphite, thus differing from pulegone. It is, however, converted into this latter compound by agitating 1 part by weight of it with 20 parts of a 5 per cent. barium hydrate solution for 50—60 hours, when it boils at 99°—103° C. under 15 mm., and reacts in every way similar to natural pulegone obtained from oil of "Pennyroyal." Pulegone and isopulegone, as well as pulegol and isopulegol, possess a certain similarity in their physical and chemical properties, such as odour, specific gravity, and direction of rotation (the former rotating the plane of polarisation to the right, the latter to the left), and both are converted by boiling with formic acid, into acetone and methyl-1-cyclo-hexanone-5. Pulegone and isopulegone, however, give different oximes and semicarbazones, since a mixture of either of these two compounds has a lower melting-point than the pure compounds separately. Moreover, pulegone and pulegol are easily reduced by sodium and alcohol to menthol, but isopulegone and isopulegol have not been directly converted into this substance.—T. A. L.

Mercury in Ammonio-Mercuric Chloride, Estimation of. J. R. Thompson. Chem. and Druggist, **50**, 234.

See *under* XXIII., *page* 263.

Aldehyde in Alcohol, Estimation of. J. Paul. Zeits. anal. Chem. 1896, **35**, 647.

See under XXIII., *page* 265.

Alkaloids and their Salts, Note on the Drying of. D. B. Dott. Pharm. J. 1897, **58**, 21.

See under XXIII., *page* 265.

Morphine in Opium and its Preparations, Assay of. A. Grandval and H. Lajoux. J. Pharm. Chim. 1897, **5**, [6], 153.

See under XXIII., *page* 265.

Antipyrine, Chemical Estimation of. C. Kippenberger. Zeits. anal. Chem. 1896, **35**, 659.

See under XXIII., *page* 266.

Guaiacol and Creosotes, Rapid Method of Analysis. L. Adrian. Bull. de Therapeut. Sec. Pharmacol, 1897, **2**, 1.

See under XXIII., *page* 266.

Gamboge, Composition of. G. Sassinari. Pharm. Centralh. 1896, **36**, 768.

See under XIII. B., *page* 248.

Musk-Pouches, Identification of Adulterated, by Means of the Röntgen-Rays. Wolff. Pharm. Centr. H.N.F. 1896, **17**, 827.

See under XXIII., *page* 266.

PATENTS.

Remedial Serum [for Tuberculosis], The Manufacture of, Impts. in. G. B. Ellis, London. From F. Niemann, Basel. Eng. Pat. 2014, Jan. 28, 1896.

The object of this invention is the preparation of antitoxic serum, free from glycerin (the presence of glycerin being objectionable, because, as alleged, it prohibits, on account of its poisonous properties, the use of large injections of remedial serum), and possessed of greater curative powers than it has hitherto been possible to obtain. The process used, is described at length. Briefly, a healthy animal is injected (subcutaneously):—

(1.) 2—3 times with tuberculin, *i.e.*, with a sterilised and filtered culture of the tubercle bacillus.

(2.) At intervals of 3—4 days with gradually increasing doses of the active principle of tuberculin, free from glycerin, until a quantity of about 2 grms. of active material per kilo. of body weight has been reached. The active principle is prepared by precipitating tuberculin with alcohol, &c., and dissolving the precipitate in water.

(3.) The final treatment consists in injecting a quantity of a boiled, but not filtered, culture of the tubercle bacillus. 3—4 weeks, subsequently, blood is drawn from the jugular of the animal, allowed to coagulate, and the serum filtered off, treated with a trace of carbolic acid and stored in sterilised bottles.—H. T. P.

Artificial Musk, An Improved Production of. J. W. Mackenzie, London. From C. Schmid, Brussels. Eng. Pat. 2205, Jan. 30, 1896.

A new benzenesulphonic acid having the constitution $(CH_3)_2:C_6H_2.C(CH_3)_3(SO_3H)$, a trimethylmethanedimethyl benzene sulphonic acid, is prepared from the following two solutions and subsequently nitrated:—

(*a*.) 1050 grms. of ordinary sulphuric acid and 460 grms. of pure sulphuric acid (66° B.), are mixed in a vessel surrounded by ice and water and kept at from 8° to 10° C.

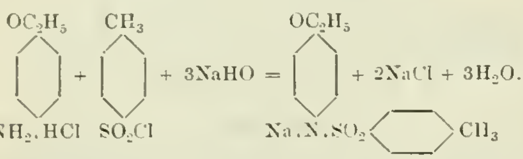
(*b*.) 130 grms. of trimethylmethane, 90 grms. of dimethyl benzene, 70 grms. of oil of turpentine, 20 grms. of ethyl benzene, and 10 grms. of dimethylethylmethane, are mixed, the mixture (*a*) is added in quantities of 10 grms. at a time, keeping the temperature below 10° C.

The whole is left in the cold bath for some hours; it is then poured cautiously in small quantities into a vessel containing 7·2 litres of water. After standing 12 hours, the floating oily layer is removed and the aqueous solution

poured into 29·5 litres of brine at 24° B., well stirred, and allowed to crystallise for 48 hours. The crystals are collected, washed, "heated in a water-bath to 125° C.," and filtered whilst hot. On cooling, the crystalline creamy mass of the new acid is formed. This is then nitrated in the usual manner, the resulting nitro product recrystallised from alcohol, neutralised with ammonia, pressed and dried. It smells intensely of musk.—J. O. B.

Phenetidine and its Homologues, New Compounds from [Sulpho-phenetidides], The Manufacture of. G. B. Ellis, London. From La Société Chimique des Usines du Rhône, anct. Gilliard P. Monnet and Cartier, Lyons. Eng. Pat. 3791, Feb. 19, 1896.

The production of aromatic sulpho-phenetidides, by the action of the corresponding sulphonic chlorides on *p*-phenetidine, is claimed. In general, the aromatic sulphonic chloride (1 mol.), dissolved in some indifferent liquid, is mixed with an aqueous solution of *p*-phenetidine hydrochloride (1 mol.) and the whole thoroughly agitated with caustic soda (3 mols.) solution, added very gradually. After some time the aqueous layer, which contains the sodium salt of the new body, is acidified with a mineral acid, whereupon the phenetidide is precipitated. The reaction that takes place is as follows, taking toluene-sulphonic chloride as example :—



Similar condensation products may be formed under the same conditions by the action of benzene, naphthalene, and other sulphonic chlorides (and their nitro- and other derivatives) not only upon phenetidine, but also on anisidine and higher homologues of phenetidine. The products formed are all more or less insoluble in water, but possess distinctly acid character and yield salts with alkalis, &c. The alkaline salts of toluene-sulpho-phenetidide and anisidide are soluble in water, and therefore suitable for medicinal and other purposes.—H. T. P.

Oxygen Gas [for Lecturers chiefly], Impts. in and relating to Apparatus for the Production of. F. J. Stedman, Strand. Eng. Pat. 4548, Feb. 29, 1896.

An apparatus is described for producing oxygen, in which the application of a heating agent to a retort is so controlled automatically as to proportion the generation of gas to the expenditure thereof.—R. S.

New Pharmaceutical Preparations [Tasteless Derivatives of Quinine, &c.], The Manufacture and Production of. J. Y. Johnson, London. From The Vereinigte Chininfabriken, Zimmer and Co., Frankfort-on-the-Main. Eng. Pat. 4991, March 5, 1896.

This claim relates to compounds produced from quinine and other cinchona alkaloids by the action of carbonyl chloride, or of chloro-carbonic ethers. See under "Eu-Quinine," this Journal, 1897, 159.—H. T. P.

Bismuthoxyiodide-methyl-gallate, Production of. W. E. Heys, Manchester, England. From The Chemische Fabrik vormals Sandoz, Basel, Switzerland. Eng. Pat. 15,295, July 10, 1896.

Ten kilos. of bismuth nitrate and five kilos. of sodium acetate are dissolved in a little water with the addition of 10 kilos. of acetic acid. A solution of $4\frac{1}{2}$ kilos. of the methyl ether of gallic acid and 3·5 kilos. of potassium iodide in about 100 litres of lukewarm water is added at once, and the mixture is heated, at a temperature of about 40° to 50° C., until the precipitate, which was at first brownish-red bismuth oxyiodide, has turned a dark grey colour. This precipitate, which is the new body, is then well washed, and dried below 100° C. It forms a light grey insoluble powder, and is introduced as a healing antiseptic dressing. It has the formula—

$$C_6H_2.CO_2CH_3(OH)_2OBi(OH)I.$$

—J. O. B.

Camphor and Isomers thereof, Impts. in the Manufacture of. J. C. Richardson, London. Eng. Pat. 3555, Feb. 17, 1896.

The reduction of turpentine hydrochlorides is effected by means of alkalis or alkaline earths, and the liberated camphene oxidised by means of hot air or oxygen to oxycamphor. If the reducing agent be a peroxide, the oxygen is supplied in the reaction. Or, turpentine is treated direct with oxides or peroxides of the alkaline earths, a current of steam or hot air passed through the mixture, and the products condensed in the usual way. The residual "uncrystallisable camphor" is used as a solvent for gums, resins, or, with sulphur, for vulcanisation of rubber. It is also emulsified with soap for a medicinal embrocation. Electrolytic reduction of the hydrochlorides is obtained either from the melted substance or from solutions, and a current of steam or air may be passed through at the same time.—J. O. B.

A Pharmaceutical Product [Menthol Solutions], the Production of; Impts. in. J. Y. Johnson, London. From The "Vereinigte Chininfabriken, Zimmer and Co.," Frankfort-on-the-Main. Eng. Pat. 3706, Feb. 18, 1896.

A fluid preparation of menthol for internal use, produced by dissolving 10—30 per cent. of solid menthol in one of its compound ethers. The valerianic ether of menthol is mentioned as being especially suitable for the purpose. In this way, it is claimed, the burning taste of the menthol is, to a great extent, masked, and at the same time its stimulating action on the system exalted.—H. T. P.

Iodo-oxybenzoic Acid, Manufacture of Esters of. O. Imray, London. From The Farbwerke vormals Meister, Lucius, and Brüning, Hoechst-on-the-Maine, Germany. Eng. Pat. 5490, March 11, 1896.

The ester of iodo-oxybenzoic acid is produced by iodising salicylic acid ester, or by esterification of di-iodosalicylic acid. The alkyl-ester of di-iodo-oxybenzoic acid is described as being a white crystalline compound readily soluble in alcohol. The di-iodosalicylic acid methyl ester melts at 110°, and crystallises in long brilliant needles. The ethyl ester melts at 132°, and crystallises in square tablets. These new esters are to be employed therapeutically.—J. O. B.

Musk, Process for the Manufacture of Substitutes for Natural. M. Dinesman, Paris. Eng. Pat. 22,139, Oct. 6, 1896.

The process comprises—

(1.) The conversion of any one of the isomeric butyl-toluidines, or homologues thereof (butyl-xylidine, &c.), into the corresponding mono-nitro-hydrocarbon, either by diazotising and treating with a cuprous salt, or by nitrating and treating the nitrated base obtained, with nitrous acid and alcohol.

(2.) Nitration of the mono-nitro-hydrocarbon thus obtained with fuming nitric and sulphuric acids. A nitro-(tri-) body is thus produced, possessing a strong musk-like odour.—H. T. P.

Acetyl and Lactyl Derivatives of Aniline and Phenetidine, Process for Preparation of. L. Lederer, Munich, Germany. Eng. Pat. 28,969, Dec. 17, 1896.

The patentee claims the cheap production of these lactyl and acetyl derivatives by heating the sulphates or chlorides of these bases, with or without pressure, with lactates or acetates, such as the lime salts of these acids. Double decomposition then results.—J. O. B.

XXI.—PHOTOGRAPHY.

Albumin, Action of Formaldehyde on. A. Bach. Monit. Scient. 1897, 157.

This investigation, begun in 1893, is avowedly incomplete, but is published in order to retain the author's rights of priority; Blum having recently taken up the same subject.

The latter has shown that egg albumin treated with formaldehyde, while retaining all its other properties unaltered, yet loses the power of coagulating with heat. At first sight this does not seem very peculiar, for a similar result may be obtained in other ways. Dried in thin layers in the sun, albumin leaves a residue completely soluble in water, and not altered by heating, except after the addition of acetic acid. Desiccated at low temperatures also, white of egg will not coagulate on heating until it has been treated with a current of carbonic acid. After addition of formaldehyde, too, albumin is less easily precipitated by alcohol, as the following experiment proves:—Two equal portions (10 c.c.) of an albumin solution were placed in test-tubes, and one was treated with 0·5 c.c. of formalin, and heated for several minutes in a water-bath. 95 per cent. alcohol was then run into both from a burette till the liquid became turbid. The normal sample required 2·2 c.c.; the other, 63·5 c.c.

A fairly pure specimen of albumin, containing only a minute quantity of mineral matter, was prepared from eggs by Würtz's process, which consists in a precipitation of the body with lead acetate, and decomposition of the albuminate with CO_2. The last traces of lead were removed by H_2S; but as the liquid would not filter, it was heated to incipient coagulation, and then allowed to settle. 750 c.c. of a solution containing about 6 per cent. of the purified albumin were mixed with 75 c.c. of 40 per cent. formalin, and the whole divided into different parts. It was found that these would bear a temperature of 92°, or even the boiling point, without decomposition; and they were apparently unaltered by exposure to sun and air for 14 months, when all odour had vanished. The liquid still gave all the reactions of albumin, and was found not to have been peptonised, as it seemed might possibly be the case. On the other hand, the formalin was not polymerised: for, on treatment with phenylhydrazine acetate in excess, no crystallisable osazone could be recovered.

With the view of isolating the compound of albumin and formaldehyde, Blum distilled the liquid till it no longer reduced ammoniacal silver nitrate and the distillate ceased to give Schiff's reaction. The present author has preferred to try repeated precipitation with alcohol and re-solution in hot water. In this way he obtained a colourless, opalescent fluid having a strong burnt sugar-like odour, and yielding, on evaporation *in vacuo*, pale yellow scales. They also possessed the ordinary properties of albumin; but on treatment with phenol or aniline, gave white precipitates soluble in alcohol. —F. H. L.

Aldehydes and Ketones, Development with, in Presence of Sodium Sulphite. L. Lumière and Seyewetz. Bull. Ass. Belge Phot. 1896, **23**, 770.

In 1889, Schwartz and Mercklin showed that the reducing power of a developer is considerably increased by the presence of formaldehyde. The authors confirm this observation, and find that the accelerated action does not depend on the formaldehyde alone, but also on the presence of sodium sulphite. No other sodium compound, such as the acetate or neutral phosphate, can take the place of the sulphite. It is also stated that most aldehydes and ketones behave like formaldehyde. Such bodies not only hasten the development, but enable the use of alkali to be dispensed with; the developer consisting of an aldehyde or ketone, a phenolic reducing agent, and sodium sulphite. The authors believe that the phenol forms a salt with a portion of the alkali of the sulphite; the sulphite being changed into bisulphite, which in its turn forms a bisulphite compound with the ketone or aldehyde. The reducing power of quinol probably depends on this liberation of alkali.

The authors append the following developer, which has given good results:—Quinol, 3 parts; sodium sulphite, 10 parts; acetone, 10 parts; water, 100 parts.—J. L. B.

Colour, The Production of, by Photographic Methods. H. Trueman Wood. J. Soc. of Arts, **45**, 278—287.

The problem of producing a naturally coloured photograph demands a substance capable of being modified by light of different colours, so as to show the same colours permanently; and this susceptibility must be capable of being arrested or destroyed by some means, so as to fix the pictures and prevent further change.

Scheele first discovered that silver chloride was darkened by exposure to light, and Senebier, in 1782—reported by Seebeck in 1810,—remarked that different parts of the spectrum affected it differently, producing colours roughly corresponding to their own. Herschel and Hunt, about 1840, exposed silver chloride paper first to diffused light and then under coloured glasses, reproducing thus approximately the colours of the glasses. Becquerel, in 1848, made copies of the spectrum by direct photography on Daguerreotype plates coated electrically with thin films of silver chloride, but did not succeed in fixing them Warren de la Rue, Niepce de St. Victer, and others, worked on Becquerel's lines, but did not succeed in making any considerable advance. More lately (first exhibited in 1891) Lippmann produced photographs by his now well-known process, which depends on the formation of a striated structure in the film, produced by interference between the direct light and that reflected from a mercury surface at the back of the film. The defect of this process arises from the fact that the colours are not pigments, but interference results, and hence only visible in certain directions. Another recent invention is the photochromoscope of Mr. Ives, in which three separate pictures of the same object are taken, through blue, red, and green glass respectively; positives made from these negatives are then illuminated, each by light of the corresponding colour, and the three images so obtained are superposed, either on the eye or on a screen. In a modification of this instrument, the three positives are dyed, each of a colour complementary to the light by which the negative was taken, then superposed and viewed by transmitted white light. A somewhat similar idea, needing, however, only one positive, was worked out by Cross, Bevan, and Green. This depends on the fact that certain diazo derivatives will take up as dyes various coal-tar colouring matters, but lose the power when acted on by light. The film, then, washed with the mixture for producing a blue dye, is exposed under the blue-printing negative, the process repeated with the red dye mixture and red-printing negative, and again with the yellow dye mixture and the yellow-printing negative. A positive in colours is thus obtained, but the colours fade in a strong light. Yet another modification of Mr. Ives' method consists in taking a picture through a variously coloured screen of fine lines, and having a similar screen behind the positive when viewing it by transmitted light. The latest process is that of M. Chassagne, an account of which has quite lately appeared (this Journal, 1897, 161), and very remarkable results of which have been shown. The nature of M. Chassagne's solutions is still a secret, and speculations as to their mode of action are therefore premature.

Other recent researches in this direction seem to be all based on the process of Becquerel. Wiener, examining the Becquerel colours at various angles of view, has concluded that on chlorinised silver plates the colours are chiefly interference effects, but that with flocculent chloride they are pigmentary; and he has attempted to sketch the elements of a theory of the action of coloured light in producing similar colour. Graby has tried to develop Becquerel's process by using silver chloride in contact with various reagents, but no very definite results have yet been obtained. Mr. Bennetto has also shown some coloured photographs, but has not yet published his process. —J. T. D.

PATENTS.

Painting and Colouring Photographs, Prints, Drawings, and the like, Impts. in and in Solutions therefor. M. Williams, Liverpool. Eng. Pat. 2711, Feb. 6, 1896.

The back of a photographic print is painted with a solution of a suitable "aniline" dye in alcohol containing a little glycerin, &c., the presence of water or saline matter being

avoided. The colour soaks into the paper, becoming visible from the front through the silver film, which softens the effect and provides the necessary light and shade.
—F. H. L.

Photographic Developers. J. Hauff, Feuerbach, Stuttgart, Germany. Eng. Pat. 27,931, Dec. 7, 1896.

The alkyl-o-amido substitution products of phenols, cresols, xylenols, and naphthols, also their derivatives, the carboxy-alkylamido substitution products of the same bodies, and their derivatives, and the molecular combinations of hydroquinone and pyrocatechol with these bodies, are claimed for the development of photographic negatives.—J. O. B.

Drawings, Impt. in the Photographic Reproduction of. E. Gay, Frankfort-on-Main, Germany. Eng. Pat. 28,087, Dec. 8, 1896.

Glazed drawing paper is treated with a mixture of gum arabic, 100 grms; bichromate of potash, 40 grms.; water, 300 centilitres: half a grm. of washing soda is preferably added. The mixture is spread on the paper and dried in the usual way. The drawing, upon tracing paper, is placed over this and exposed to the light for 5 to 15 minutes. The print is first washed very delicately, and again a second time with a sponge until the lines of the drawing appear hollow, owing to the solvent action of the water. It is dried first with blotting paper, then in the air. When dry, the print is coated with a sponge with a mixture of shellac, 75 grms.; lamp black, 70 grms.; spirits of wine, 750 centilitres. It is then plunged in a bath of hydrochloric acid for 20 minutes, when the paper whitens and the drawing comes out black; it is finally cleaned by brushing in water, and dried.—J. O. B.

XXII.—EXPLOSIVES, MATCHES, Etc.

PATENTS.

Explosives [Safety], Impts. in or relating to. W. Greaves and E. M. Hann, both of Aberdare. Eng. Pat. 4509, Feb. 28, 1896.

The object of this invention is to render ordinary gunpowder, or explosives of that class, safe for use in fiery or dusty mines, by adding one or more of the following salts: Oxalic, formic, tartaric, or acetic acids. The alums, carbonate of ammonium and soda, sulphates of ammonium, soda, or potash, chloride of ammonium, or, preferably, ammonium oxalate.—R. B. P.

Measuring the Power or the Volume of the Gases resulting from the Explosion of Explosives, An Improved Method of and Apparatus for. H. H. Lake, London. From the Sprengstoff Actien-Gesellschaft-Carbonit, Hamburg. Eng. Pat. 3023, Feb. 10, 1896.

See under XXIII., *page* 261.

Matches, Manufacture of; Igniting Compound for. G. Schwieniog, Beltenhausen, Cassel, Germany. Eng. Pat. 6052, March 18, 1896.

An igniting mixture for the heads of matches consisting of 15 parts of potassium chlorate, 0·5 part of sulphur, 4 parts of calcium plumbate, 1 parts of "terra sienna," 2 parts of glass, 1·5 parts of amorphous phosphorus, 1·5 part of gum or cementing matter, all by weight.

It is claimed that the above mixture ensures ignition of the match stem on being rubbed against any surface offering frictional resistance.—R. B. P.

Explosives [Safety], Impts. in or relating to. W. Greaves and E. M. Hann, both of Aberdare. Eng. Pat. 6937, March 30, 1896.

To produce an explosive that can be used with safety in fiery or dusty coal mines, these inventors propose the addition in suitable proportions of borax or boric acid, either alone or mixed with one or more of the following salts:—To carbonite, dynamite, the gelatine explosives, &c., or to "ordinary gunpowder" "and explosives of that class."

The salts named are:—Oxalic acid, ammonium, sodium, and potassium oxalates, or the double oxalates of these. The salts may be used either in the hydrated or the anhydrous state.

The employment of various alums for the same purpose is also described in the provisional specification.—R. B. P.

Gunpowder [Nitrate Mixture], Impts. in the Manufacture of. C. Mortier, Bilbao, and H. A. Saudon, Portugalete. Eng. Pat. 25,711, Nov. 14, 1896.

For sporting guns, pistols, and revolvers the powder consists of trinitrocresol, 25 parts; nitrate of potash, 65 parts; charcoal, 9 parts; and stearic acid, 1 part.

For military rifles ("Gras" system) and cannon of small calibre, it consists of trinitrocresol, 20 parts; nitrate of potash, 70 parts; charcoal, 8 parts; and stearic acid, 2 parts, all by weight, in both cases.

The three last-named ingredients are incorporated in a revolving iron cylinder containing bronze balls. The mixture is then transferred into a wooden revolving cylinder, with wood balls, and the trinitrocresol incorporated with it. The mixture is then pressed hydraulically into slabs, which are subsequently granulated by means of rollers. The granulated powder is then treated in a glazing drum in which the heat produced by friction is sufficient to melt the stearic acid, which then forms a waterproof coating on each grain.—R. B. P.

XXIII.—ANALYTICAL CHEMISTRY.

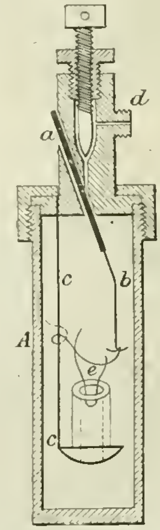
APPARATUS, ETC.

Ultimate Organic Analysis under Pressure in an Autoclave. Hempel. Ber. **30**, (1897), [2], 202—211.

The author has devised a rapid and easy mode of determining carbon, hydrogen, sulphur, and halogens in organic compounds by combustion in compressed oxygen in an autoclave similar to Berthelot's explosion-bomb, but smaller, so as to economise substance, save time, and admit of weighing the whole on an ordinary chemical balance.

The apparatus, shown half size in the figure, is bored and turned out of a block of iron or steel, is enamelled inside, is 2 mm. thick in the walls, will stand a pressure of 200 kilos. per

Fig. 1.



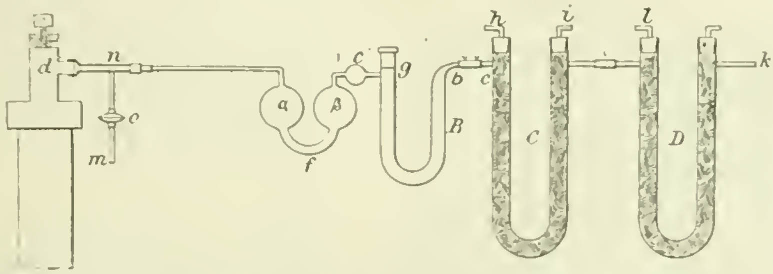
sq. cm., and holds 33 c.c., so that 800 c.c. of oxygen at atmospheric pressure will be contained in it at 25 kilos. per sq. cm. The cover is screwed on upon a lead ring packing, and the central screw for the valve is packed with vulcanised fibre. Through *a* passes (packed and insulated by india-

rubber) an iron rod, to which is attached the platinum wire *b*, while *c* is a platinum spoon attached to the cover, and in which stands the little open cylinder of fireclay.

Solids are compressed in a mould into little cylinders, a short length of cotton thread being first inserted, the ends of which, twisted round a fine platinum wire stretched between *b* and *c*, serve as a torch or wick. Liquids are dropped into the spoon from a small weighed pipette, a similar thread dipping into the liquid. The CO_2 and H_2O yielded by combustion of a known length of this thread are determined once for all, and allowance made in each analysis.

The substance is weighed, placed in the spoon, the autoclave closed, and the whole weighed. An adapter is then screwed on *d*, communicating with an oxygen cylinder and a pressure gauge, and oxygen allowed to enter till the pressure reaches 25 kilos. per sq. cm.; the valve is closed, and the oxygen bottle disconnected. The autoclave is then placed for safety in a strong outer cylinder of iron, the thin platinum wire heated by a current of electricity, and the combustion allowed to proceed. After half an hour (two hours if nitrogen acids, sulphur, or halogens be present) connection is made by the adapter *n* to the bulb-tube *f* containing a little strong sulphuric acid, the calcium chloride tube B, the soda-lime tube C, and the guard-tube D. The valve is opened, and the gases allowed to pass slowly out. When this ceases, *k* is connected to a water aspirator, and when exhaustion goes no further, purified air is admitted through *m* and the pump again set to work; the admission of air and exhaustion are repeated. The apparatus is taken apart, and autoclave, water-collector, and CO_2-collector separately weighed. The autoclave is now opened, rinsed with water, and the acidity determined by centinormal soda solution. Sulphur and halogens can now be determined in the usual way. Any carbon remaining in the spoon is burnt off in the air, the spoon being weighed before and after, and the weight of the carbon added to that calculated from the CO_2.

Fig. 2.

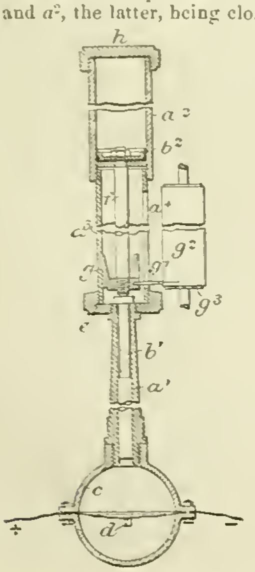


A specimen analysis and calculations are given in the paper. The autoclave used by the author weighs 250 grms. and costs about 50*s*.—J. T. D.

PATENTS.

Measuring the Power or the Volume of the Gases Resulting from the Explosion of Explosives, An Improved Method of and Apparatus for. H. H. Lake, London. From The Sprengstoff Actien-Gesellschaft-Carbonit, Hamburg. Eng. Pat. 3023, Feb. 10, 1896.

The apparatus consists of two pistons b^1 and b^2 moving in two cylinders a^1 and a^2, the latter, being closed at its upper end by a cap *h*. a^1 is in communication with a chamber *c*, in which the material to be experimented upon is exploded by means of an electric detonator *d*. The piston b^2 is attached to a rod *f*, the lower end of which rests upon the top of b^1, so that any movement of b^1 is communicated to b^2. The lower end of *f* is guided by the block *g*, which slides in the tube a^3. a^3 is attached by screwing to a^2 at one end and to a^1 at the other by means of *e*.



When the material being experimented on is exploded in *c*, pressure is set up and b^1 is forced outwards.

The motion of b^1 is communicated by *f* to b^2 and the air, or other gas, in a^2 is compressed until the total pressures acting on b^1 and b^2 are equal.

The dimensions of the various parts being accurately known, the pressure acting on b^2, and therefore the pressure acting on b^1, can be calculated from the distance moved through by the pistons. This distance can be recorded by attaching to the block *g*, a marking point g^1 (which is guided by a slot a^4 in the wall of a^3) and allowing its outer end to trace a curve on the surface g^2, of a revolving drum g^3.

In place of an elastic medium in the cylinder a^2, an inelastic one, such as glycerin, may be used.

The cylinder a^2 is then provided with an opening of suitable size through which the inelastic medium is forced out. The quantity forced out serves as a measure of the power of the explosive.—R. B. P.

Electric Device [Arc Lamp], for showing the Relation of Oxygen and Nitrogen in Mines and other places, Improved. L. Cohn, Breslau, Germany. Eng. Pat. 28,609, Dec. 14, 1896.

See under II., *page* 231.

INORGANIC CHEMISTRY.—QUALITATIVE.

Nitrous Acid, a Delicate Reaction for. E. Riegler. Zeits. Anal. Chem. 1896, **35**, 677.

In a small tube, place 0·02—0·03 grm. of crystallised naphthionic acid, add 5—6 c.c. of the liquid to be tested, shake well, add 2 or 3 drops of strong hydrochloric acid and shake thoroughly for a minute, now add 20 or 30 drops of ammonia, holding the tube in a slanting position. Traces of nitrous acid cause the appearance of a rose-coloured ring where the liquids meet; on shaking, the whole liquid becomes rose or dark red according to the quantity of nitrous acid present. Since very dilute solutions of naphthionic acid fluoresce, the colour should be examined by transmitted light. In rain water and tap water the presence of nitrous acid is well shown by this reaction. The coloration depends on the formation of diazonaphthalenesulphonic acid, which with ammonia and a second molecule of naphthionic acid produces a dyestuff.—A. C. W.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Phosphoric and Arsenic Acid, Titration of: An Exact Method of. A. Christensen. Zeits. Anal. Chem. 1897, **36**, 81.

The author finds that by titration of the free iodine liberated on mixing an excess of solution of potassium iodide and potassium bromate with free phosphoric acid, the reaction which takes place is in accordance with the equation—

$$KBrO_3 + 6KI + 6H_3PO_4 = 6KH_2PO_4 + 6I + KBr + 3H_2O.$$

In conducting the determination, the diluted phosphoric acid solution not exceeding the equivalent of 40 c.c. of decinormal thiosulphate solution (1 c.c. of which = 0·007 grm. P_2O_5), is placed in a flask with a well-ground stopper along with 3 grms. of potassium iodide and 10 c.c. of 5 per cent. potassium bromate solution diluted to 100 c.c. and either allowed to stand at normal temperature until the next day or warmed between 40° and 50° for half an hour, and the free iodine titrated. The end of the reaction is sharp, and far more delicate than in the methods of acidimetric titration with standard alkali. The method is applicable with modifications to bone meal, Thomas slag, and other commercial phosphates, the phosphoric acid being precipitated as silver or magnesium ammonium phosphate, from which it is liberated by means of sulphuric acid, and in the author's hands has given most satisfactory results. For details of the many experiments reference should be made to the original paper.

In the determination of arsenic acid by this method the results obtained were not quite so exact as in the case of phosphoric acid, but were sufficiently so to be of considerable value.—J. O. B.

Sulphur in Pyrites, Determination of, by Means of Sodium Peroxide. C. Glaser. Chem. Zeit. 1897, 40.

In the extraction of the melt (see this Journal, 1894, 1095) it sometimes occurs that the residue is more or less black, due to the formation of ferrous sulphide produced by interaction of iron oxide and still-present sodium sulphide. This is more likely to occur if the peroxide by frequent exposure has lost some of its oxidising power. The finely divided iron sulphide may without difficulty be oxidised whilst wet; let the liquid cool somewhat, and add sodium peroxide in small quantities until the colour of the residue becomes a pure brownish-red and a permanent foam forms, when this is the case all the sulphur is oxidised. The residue should be washed at the pump with boiling water. Since the iron oxide readily dissolves in a hydrochloric acid solution of bromine, it may readily be tested for sulphur, and if necessary any small amount of barium sulphate can be added to the main precipitate.

The difficulty mentioned by Asbóth (this Journal, 1895, 597), that in low grades of pyrites (30 per cent.), separation of silicic acid may increase the weight of the barium sulphate, has not been noticed by the author in analyses of 40—45 per cent. American pyrites, and moreover analyses made by his method agreed very well with results obtained by Fresenius's method. Possibly the explanation may be that the author only allows the precipitate to stand long enough for complete deposition, for which 15 minutes is ample when precipitating in hot solution with addition of the barium chloride in drops.—A. C. W.

Ferric Chloride, Volatility of. H. P. Talbot. Amer. Chem. J. 1897, **19**, 52—59.

The author states that no loss of iron, in the form of ferric chloride, is to be feared under the conditions which prevail in ordinary analytical processes, unless residues containing also ammonium chloride are ignited, or such residues have contained traces of aqua regia, when a small, but appreciable loss of iron appears to be possible.—A. S.

Caustic Alkali, Estimation of, in Presence of Alkaline Carbonates. G. Lunge. Zeits. f. angew. Chem. 1897, [2], 41—42.

An investigation by Küster into the volumetric methods of estimating caustic alkali in liquors containing carbonate shows that of six methods involving the use of barium chloride, only that of C. Winkler gives true results. The present paper gives a comparison of the results obtained on analysing various samples: (*a*) by the gas volumetric method; (*b*) by direct differential titrations using phenolphthalein and methyl orange as indicators; (*c*) by the Winkler method (direct titration with methyl orange as indicator, and in a second portion, titration, after the addition of barium chloride, using phenolphthalein), carefully following Küster's directions as to the very gradual addition of the ½ normal hydrochloric acid used for titration. When only small percentages of carbonate were present all three methods led to identical results, but when the quantity of alkali as carbonate became greater than that present as caustic alkali, whilst methods (*a*) and (*c*) gave correct, identical and true values, method (*b*) no longer yielded correct results.—J. T. C.

Persulphates, Analysis of. F. Ulzer. Zeits. f. angew. Chem. 1897, 61.

In the estimation of active oxygen, the author states that he obtained good results by heating a solution of about 0·3 grm. of the sample with an excess of ferrous ammonium sulphate solution (containing 1 to 1·5 grm. double salt solution) and dilute sulphuric acid in a current of carbonic anhydride, or using a Bunsen valve, for about half an hour, and titrating back the excess of iron salt with permanganate. To determine the total sulphuric acid, a solution, as concentrated as possible, of a weighed quantity of the sample, was heated for some time with hydrochloric acid to destroy the persulphuric acid, and after strong dilution, the sulphuric acid was precipitated as barium sulphate. A sample analysed gave 70·01 per cent. total sulphuric acid, 14·01 per cent. of ammonia, and a fixed residue of 0·61 per cent. The sample contained 45·61 per cent. of ammonium persulphate (3·2 per cent. active oxygen) and contained free acid, titrated by normal alkali, using methyl orange, equivalent to 18·93 per cent. of sulphuric acid. The composition of the sample is given as follows:—Ammonium persulphate, 45·61 per cent.; acid ammonium sulphate, 48·77 per cent.; residue, 0·61 per cent.; and water, 5·01 per cent.—J. T. C.

Manganese, Electrolytic Determination of. C. Engels. Zeits. f. Elektrochemie, 1897, **3**, [14], 286—289, and 305—308.

These are further communications on an electrolytic method for quantitatively estimating manganese described in a previous number of the same Journal (1895–6, Heft 19). An account of the method (abstracted from the *Berichte*) as applied to salts of manganese will be found in this Journal for 1896, 219. It is now stated that the method can be used for directly determining manganese in salts of manganic and permanganic acids. To a solution of the salt (say, potassium permanganate) add 5 c.c. of acetic acid (1:2), and enough hydrogen peroxide to decolorise the solution. The presence of even a small quantity of hydrogen peroxide prevents the formation of an adherent deposit. The excess must therefore be removed by adding a small amount of chromic acid: 0·2 to 0·4 grm. is generally sufficient. 2 grms. of chrome alum and 10 grms. of ammonium acetate are next added and the solution is made up to 150 c.c. The electrolysis is carried out with an E.M.F. of 3 to 3·4 volts and a current density of about 0·9 ampère per 100 sq. cm. The electrolysis is most satisfactorily performed at a temperature of 85°, the deposit then obtained being adherent. At lower temperatures it is brittle, and during the subsequent processes of washing with alcohol, drying, and heating, there is apt to be decrepitation and loss.—D. E. J.

Nickel and Copper, Separation of, in Matte, and the Recovery of the contained Precious Metals. T. Ulke. Eng. and Mining J. **63**, [5], 113—114.

In this article the author gives a short review of the various electrolytic methods proposed for the above separa-

tion. In referring to Hoepfner's process (this Journal, 1897, 49), he mentions the chief objection as being due to the fact that a cheap and durable diaphragm of low electrical resistance has not yet been discovered. Rickett's method (1893) of precipitating the nickel as basic sulphate and separating the copper by electrolysis, is also spoken of as not showing promise. In mentioning the difficulty of depositing a thick plate of pure nickel owing to its peeling off, in consequence probably of thick films of oxide, it is said that plates ⅜ in. thick are now produced at the Balbach Works, which are very tough and elastic, and will not break or crack under the hammer. In these works, anodes containing about 95 per cent. of nickel, 0·55 of copper, 0·75 of iron, 0·25 of silica, 0·45 of carbon. and 3 per cent. of sulphur—the latter added to facilitate the casting—are used; and over 1,000 lb. of metallic nickel, containing less than 0·03 per cent. of arsenic and 0·02 per cent. of sulphur, are produced daily. The chief drawback to electrolytic processes appears to be the falling to pieces of the metallic or matte anodes, and the formation of large quantities of anode scrap, which sometimes amounts to 50 per cent. of the charge, and which consequently must be remelted into anodes and re-treated.

The process for treating Bessemer matte and recovering the precious metals therefrom, proposed by the author, is then described. The granulated matte is dissolved in dilute sulphuric acid, so that the electrolyte will contain about 8 per cent. of free acid. To this is added one-half per cent. of ammonium sulphate to retard the electro-deposition of arsenic, and a little hydrochloric acid to retain all the silver in the slime. Copper and nickel dissolve at the anode, while copper deposits at the cathode, and the precious metals with the anode scrap are deposited and settle down as slime. The copper is deposited from the solution first until the percentage is very small as compared with the nickel present, when the solution is separated from the electrolytic tank, and the remainder of the copper thrown down by sodium sulphide. The nickel solution thus obtained is freed from the small amount of iron present, and the nickel electrolytically deposited therefrom. For this purpose the liquid is kept neutral or slightly ammoniacal, and insoluble anodes of carbon or lead, or soluble anodes consisting chiefly of nickel, are used. With the former anodes the nickel will be of great purity, whilst with the latter it will be practically pure until the other metals in the nickel anode accumulate to an appreciable extent in the bath. With insoluble anodes the solution gradually becomes acid, and with soluble anodes it becomes alkaline, so that it is suggested both anodes be used in the same bath to preserve its neutrality.

A method of determining copper and nickel in the same portion of matte is then described as follows:—5 grms. are treated with 2·5 c.c. of sulphuric acid (1 : 1) and 10 to 15 c.c. of hydrochloric acid. After 10 minutes digestion, 5 c.c. of nitric acid are added, and the solution is evaporated to dryness. The mass is dissolved in water with a few drops of nitric acid, diluted to 100 c.c., and connected with the battery to deposit the copper, allowing about ¼ ampère for each assay, the deposited metal being washed with alcohol and dried. To obtain the nickel, 0·5 to 1 grm. of sodium peroxide is then added to the solution, and the iron precipitated with ammonia, redissolved, and reprecipitated four times. The four solutions are mixed and evaporated to about 200 c.c., made strongly ammoniacal, and placed in connection with a Grove's battery. Two ampères per solution are allowed, and the temperature should be 70° C. In about 1 to 1½ hours the nickel is deposited, and is washed with water and alcohol, and weighed.
—A. W.

Molybdenum and Vanadium, Volumetric Estimation of. C. Friedheim. Ber. **29**, 2981—2985.

This paper is a reply to some criticism by Gooch and Fairbanks of a method for estimating molybdenum, previously described by Friedheim and Euler, depending on the reaction $2MoO_3 + 2HI = Mo_2O_5 + H_2O + I_2$ (See this Journal, 1896, 220).

The determination, as previously described, is carried out in a Bunsen apparatus, and one objection raised by Gooch and Fairbanks is that error is introduced through oxidation of distilled hydriodic acid by atmospheric oxygen present in the receiver. They accordingly recommend that the reaction be carried out in an atmosphere of carbonic acid; this the present author condemns as unnecessarily complicating the process, and he reasserts that correct results are readily obtained provided that the first heating in the distillation is a gradual one, and that the solution is not raised to boiling until the retort is filled with iodine vapour. The reaction is ended when the iodine colour has vanished and the solution has become light-green; and the author shows that a statement by Gooch and Fairbanks that these indications are not sufficient, is based on insufficient grounds, and that the iodine found by these authors in the distilling flask is due to oxidation by air of the contained hydriodic acid.

For the reduction of vanadium pentoxide to the tetroxide (or, in presence of syrupy phosphoric acid, to the trioxide) by means of hydriodic acid, to obviate the use of the Bunsen apparatus, Browning has proposed to reduce the vanadate with hydriodic and sulphuric acids in an open vessel, and, after boiling off the iodine and nearly neutralising with caustic alkali, to add sodium bicarbonate and tartaric acid, and then to estimate the vanadium tetroxide with excess of standard iodine solution, titrating back with arsenious acid.

The author regards the use of two standard solutions as undesirable, and states that the Browning process renders the estimation of bases (lead and barium) in the distillation residue exceedingly difficult. A similar objection holds for Gooch and Fairbank's proposal to apply Browning's process to the estimation of molybdenum.—J. T. C.

Mercury in Ammonio-Mercuric Chloride, Estimation of. J. R. Thompson. Chem. and Druggist, 1897, **50**, 231.

The author has reported the examination of five samples of ammonio-chloride of mercury obtained from different sources. He found the following percentages of Hg estimated as Hg_2Cl_2 in the five samples respectively:—A = 71·53, B = 71·56, C = 73·05, D = 75·01, E = 76·80. The B.P. standard is 77·5. He also found that the method of estimating the mercury as a metal was open to error on account of the loss in drying the metallic mercury. To reduce it to sub-chloride Hg_2Cl_2, he dissolved a weighed quantity of ammonio-mercuric chloride in hydrochloric acid in a beaker. To this an acidified solution of potassium hypophosphite was added, when a precipitate of Hg_2Cl_2 was almost immediately thrown down. This was set aside for 12 hours, then washed with hot water till free from chloride, pressed, dried at 100° C. till constant, and weighed.—A. S.

ORGANIC CHEMISTRY.—QUANTITATIVE.

Fatty Acids, Note on Wechsler's Method for the Separation of. A. W. Crossley. Proc. Chem. Soc. 1897, [174], 21—22.

Wechsler (Monatsh. 1893, **14**, 462) has described a method for the separation of fatty acids, the principle of which method is contained in the following statement:—

If to a mixture of two fatty acids four-fifths of the caustic soda necessary to neutralise them be added, and the whole steam-distilled, the distillate contains the pure higher boiling acid. From the residue of the distillation a further three-fifths of the acids are set free by addition of sulphuric acid, and the whole is distilled with steam. Finally, the remaining fifth of the acids is set free, and in this case the distillate contains the lower boiling acid in a pure condition.

The purity of the acids contained in the various distillates was proved, by converting them into silver salts and subsequent analysis of these salts.

After experimental trial, it is concluded that Wechsler's method does not give such good results as its author suggests, nor can it in any way be looked upon as a satisfactory method for separating mixtures of fatty acids.

Prussian Blue: Commercial Analysis. E. J. Parry and J. H. Coste. Analyst, 1896, **21**, [246], 225.

The authors adopted the following scheme of analysis:—The moisture was determined by drying at 100° C., and the

water of combination by combustion with lead chromate. The nitrogen was estimated by boiling with sulphuric acid and then determining the ammonia in the ammonium sulphate formed. For the estimation of the total iron, a weighed quantity was ignited, care being taken that the temperature was sufficiently high to decompose the last traces of the blue, but not high enough to render the oxide of iron difficult of solution. In all cases, on adding hydrochloric acid to the "ash," a marked effervescence showed that alkaline carbonate was present. The acid solution was filtered, if necessary, from barium sulphate or other insoluble matter, and the iron (and alumina) precipitated with ammonia. The iron in the (weighed) mixed oxides was determined by titration with potassium bichromate, after reduction with stannous chloride, the alumina being estimated by difference. A portion of the filtrate was evaporated to dryness, and, after volatilisation of the ammonium salts, the alkaline salts were weighed, and the chlorine therein determined by titration with silver nitrate. In another portion of the filtrate, the sulphuric acid was determined, and from these data—*viz.*, the weight of the alkaline salts, chlorine, and sulphuric acid—it was shown that in all cases the alkali-metal was almost entirely potassium or sodium, and in no case a mixture of the two metals. Another weighed quantity of the pigment was boiled for a few minutes with aqueous potash, and the resulting precipitate of oxide of iron weighed, after ensuring its complete conversion into Fe_2O_3. This, multiplied by 0·7, represents the "extra-radicle" iron.

Pigments sold as Prussian blue should contain at least 20 per cent. of nitrogen and 30 per cent. of iron, calculated on the dry matter, and, after burning, should be entirely dissolved by hydrochloric acid. The sulphuric acid used in the nitrogen determination should not be blackened; this, if it occurs, indicating the presence of organic adulterants.

—A. S.

Cœrulignone Dyestuffs. [Determination of Aniline in Quinoline.] C. Liebermann and J. Flatau. Ber. **30**, 234.

See under IV., *page* 233.

Glue, Viscosity of, A New Process for Determining the. J. Fels. Chem. Zeit. 1897, **21**, 56 and 70.

The author discusses the commercial valuation of glues, and offers a criticism and list of the principal chemical and physical methods. He adopts in principle Lipowitz's method (Neue Chem. techn. Unters. Berlin, 1861, 37—42), which is based on the fact that the consistency of a glue is equivalent to the quality—that is, to its adhesiveness. The glue under examination is powdered, and a portion dried at 100° C. for two hours, to determine the water. 100 grms. are treated with 400 c.c. of cold water for 24 hours, and finally dissolved by warming on a water-bath; it is then diluted so that 15 per cent. of dry substance is in solution. The viscosity determination is made with this solution at 30° C., using an Engler's viscosimeter; the viscosity of water at the same temperature being taken as unity. The viscosity period, divided by that of water, gives a number expressing the consistency of the glue in question.

—J. L. B.

Albuminoids in Substances Rich in Starch, A Modification of Stutzer's Method for the Estimation of. H. Tryller. Chem. Zeit. 1897, **21**, 54.

When albuminoids are estimated by Stutzer's method, dealing with substances rich in starch, considerable difficulty is experienced in filtering and washing the copper hydroxide compound. Stutzer himself has proposed to warm such substances on the water-bath for only 10 minutes, so as to prevent, as far as possible, the gelatinisation of the starch. The author employs malt extract to transform the starch, and carries out the determination in the following manner:—1 grm. of the substance is well incorporated with 100 c.c. of water, placed in a beaker, and heated on a boiling water-bath for 10 minutes; it is then cooled to 65° C. and 10 c.c. of malt extract (100 of malt to 500 c.c. of water) added, the above temperature being maintained for 20 minutes. The alum and copper hydroxide are next introduced, and the contents of the beaker heated for 10 minutes in a boiling water-bath. The precipitate is collected on a filter, well washed, and the nitrogen determined by Kjeldahl's method in the precipitate *plus* the filter. The result is corrected for the nitrogen present in the malt extract. A list of results show that the numbers obtained by this modification and by Stutzer's method agree closely.

—J. L. B.

Optically Active Substances in Beetroots, Presence and Estimation of. F. Herles. Zeits. Zuckerind. Böhmen, 1897, **21**, 302—305.

When the yield of sugar does not agree with the polarisation of the beetroots worked up, the task of the chemist is often to ascertain if, along with sugar, there are not other dextrorotatory substances present in the roots. In factories where the basis of control is the polarisation of the juice and not the direct polarisation, the discrepancy is to be sought in this circumstance, but it also occurs when hot-water digestion is used, and in this case the difference between the alcoholic and water digestion is regarded as a proof of the presence of optically active non-sugars.

The author had occasion to examine a sample of halved beetroots which had given a difference of 2·2 per cent. between the alcoholic and water digestion. By digestion with 96 per cent. alcohol, 90 per cent. alcohol, and with water, he obtained 13·30, 13·60, and 13·90 per cent. respectively. The normal weight of expressed juice, when made up to 100 c.c. with 93 per cent. alcohol and with water, read 14·90 and 15·10 per cent., showing the effect of alcohol in lowering the reading. He attributes larger differences to bad sampling, imperfect subdivision of the pulp, and consequent imperfect extraction, or inaccurate tests, and suggests as the direct proof of the presence of active bodies the Clerget inversion (this Journal, 1897, 67).

—L. J. de W.

Reducing Sugars, Determination of, in Terms of Cupric Oxide. G. Defren. J. Amer. Chem. Soc. 1896, **18**, 749—756.

The author employs O'Sullivan's method (J. Chem. Soc. 1876, ii., 130) for the determination of the reducing powers of sugars. The Fehling's solution is prepared according to Soxhlet's formula, with a slight modification:—69·278 grms. of crystallised copper sulphate are dissolved in water and the solution made up to one litre. 356 grms. of Rochelle salt, and 100 grms. of sodium hydroxide are dissolved in water and made up to a litre. The two solutions are kept in separate bottles. The cuprous oxide may be collected on paper in a Soxhlet tube, or Gooch crucible; the last two being packed with prepared asbestos. It is then well washed, ignited, and weighed as cupric oxide.

It has been stated by O'Sullivan (*loc. cit.*) that 1 grm. of glucose produces 2·205 grms. of cupric oxide. The author, however, finds that this value is not a constant for all weights of glucose, the amount varying from 2·27 grms. of cupric oxide for small quantities, and 2·22 grms. for large; this is the case also with maltose and milk sugar. The weights of dextrose, maltose, and milk sugar respectively, corresponding to a given weight of cupric oxide, are obtained by the following formulæ, in which W = the weight of cupric oxide:—

$$D = (0{\cdot}4400 + 0{\cdot}000037\ W)\ W$$
$$M = (0{\cdot}7215 + 0{\cdot}000061\ W)\ W$$
$$L = (0{\cdot}6270 + 0{\cdot}000053\ W)\ W.$$

Tables are given in which the weights of dextrose, maltose, or milk sugar, corresponding with given weights of cupric oxide, are obtained. A supplementary table is added for application to mixed products such as commercial glucose, in which the weights of cupric oxide and equivalent weights of dextrose, as obtained by the 3·86 divisor, have the relation 386 : 381 to the absolute equivalents.

—J. L. B.

Starch, Conditions affecting the Volumetric Estimation of, by Means of a Solution of Iodine. F. T. Littleton. Amer. Chem. J. 1897, **19**, 44—49.

The author concludes, as the result of a series of experiments, that on account of the dissociating effect of water on starch iodide, the iodometric method is available only with

substances containing a large amount of starch, and is best used when the substances compared, contain starch in proportions not widely differing. The solutions to be colorimetrically compared must be in all respects under conditions very nearly identical.

The so-called iodide of starch, if a chemical compound, is one very easily dissociated, *e.g.*, by rise of temperature, by increase of the amount of solvent water, and by the introduction of another liquid, such as chloroform, carbon bisulphide, &c., having a superior solvent power for iodine. —A. S.

Aldehyde in Alcohol, Estimation of. J. Paul. Zeits. anal. Chem. 1896, **35**, 647.

In Mohler's method of using Schiff's reaction for the colorimetric estimation of aldehyde in alcohol (Ann. Chim. Phys. 1891, 121), a solution of magenta decolorised by fixed quantities of sodium bisulphite and sulphuric acid is used. The coloration is, however, different when different sulphite solutions are used, probably because solutions of the same specific gravity have not always the same composition. The author overcomes this difficulty by the use of sulphurous acid solutions, the strengths of which can be iodometrically determined. The reagent contains 0·05 grm. of magenta and 0·5 grm. of H_2SO_3 in 100 c.c., and is made by mixing a solution of the purest "Diamant Fuchsine" of known strength (filtered, whether clear or not) with the corresponding quantity of sulphurous acid, and making up to the volume given. After an hour or two the solution is colourless. It may be kept in well stoppered bottles for a few days, but long keeping is to be avoided.

When alcohol free from aldehyde is required, the author finds that Mohler's method—boiling 1 litre of absolute alcohol with 10 c.c. of aniline and 10 c.c. of phosphoric acid of 45° B. for an hour with return-condenser—gives an alcohol containing both aldehyde and aniline. All attempts to purify alcohol by chemical methods were unsuccessful: finally the following process was adopted:—A 10-litre flask was connected with a fractionating column inclined at an angle of 45° with the horizon, and this with a reflux condenser through which water at 50°—60° C. slowly flowed. The flask contains 8 litres of about 95 per cent. alcohol, not too rich in aldehydes, which is kept boiling on the water-bath. Aldehyde escapes at the open end of the condenser; its exit is facilitated by inserting in the condenser to the level of the water, a tube connected with an aspirator. After several days of such treatment the alcohol was slowly distilled off, a second condenser being connected to the first. The first fraction of 3 litres was rejected; the second, also of 3 litres, was used for the preparation of the typical solutions. In most cases the colorations are sufficiently intense when the alcohol is diluted to 30 per cent.; only when the liquid would contain after such dilution less than 15—20 mgrms. of aldehyde per litre, is dilution to 50 per cent. necessary. The typical solutions contain 25, 50, 100 mgrms. of acetaldehyde in 1 litre of alcohol of 30 volume per cent. For alcohols very poor in aldehyde a solution containing 10 mgrms. in 50 per cent. alcohol, was used. The observations were carried out with the Krüss's polarisation colorimeter and Duboscq's colorimeter. The following is a detailed description of the process with a solution of known strength:—10 c.c. of the alcohol to be tested and 10 c.c. of the typical solution of 25 mgrms. content were placed in two dry test tubes swimming in a large vessel of water at 16° C., where the temperatures were equalised. 1 c.c. of the reagent was added to each tube as rapidly as possible, the contents mixed, and the tubes again placed in the water. After 25 minutes the two liquids were poured at the same time into the cylinders of the colorimeter, 10 observations were made rapidly, the height of the solution under investigation being 25 mm. The mean of the 10 observations of the height of the typical solution was 24·1 mm.; thus the aldehyde content of the alcohol is $25 \times \frac{24 \cdot 1}{25} = 24 \cdot 1$ mgrm. per litre. In practice, the aldehyde content of the unknown alcohol will not be so nearly equal to that of the typical solution: a rapid preliminary experiment is made, using a typical solution of less strength than that of the unknown alcohol, and from the result of the test the alcohol is diluted to such an extent as to give about the required tint. When the polarisation-colorimeter of Krüss is employed, the typical solution of 100 mgrms. is unusable, and that of 50 mgrms. to the litre gives intense colorations with which it is difficult to work.

If the greatest accuracy is not required, the colour comparisons may be made in test-tubes of equal diameter. The author finds, in agreement with Mohler and in opposition to Bornträger, that acetal may be estimated by this method, being apparently entirely split up by the sulphurous acid. 70 parts of furfurol give only the same coloration as 1 part of acetaldehyde.

Of other triphenylmethane colours, Crystal Violet does not give the reaction, Methyl Violet BB from Meister, Lucius, and Brüning, and Methyl Violet 1 B conc. from Farbenfabriken vorm. F. Bayer and Co. are both decolorised by sulphurous acid and give bluish violet colorations proportional to the quantity of aldehyde, but the intensity is much inferior to the magenta coloration. Methyl Violet 5 R from the last-mentioned firm gives very favourable results, almost surpassing those with magenta. In the experiments quoted, using magenta, the maximum error is 8 per cent. —A. C. W.

Alkaloids and their Salts, Note on the Estimation of Water in. D. B. Dott. Pharm. J. 1897, **58**, 21.

Drying on the water-bath is recommended as the most convenient course in the case of the alkaloids and their salts, when these are being estimated by pharmacopœial methods, and should be adopted when not contra-indicated. But as the temperature of a water-bath varies according to its size, ventilation, and the amount of water contained in it, a substance which does not readily part with its water a few degrees below 100° C., should not be dried in the water-bath but in an air-bath, or otherwise at a temperature over 100° C. Morphine hydrate is such a substance. In an ordinary copper water-bath, 10 ins. internal measurement, pure morphine hydrate in fine powder lost, after two hours, 3·31 per cent.; after three hours, 3·56 per cent.; the bath being well-filled with water briskly boiling. The morphine was then heated in an air-bath for an hour at 120° C., when it indicated a loss of 6·35 per cent., and lost nothing farther. Another sample dried in the water-bath containing very little water, had lost after two hours only 0·17 per cent., then on drying at 120° C., a loss of 6·49 per cent. was indicated. The formula, $8C_{17}H_{19}NO_3, 9H_2O$, requires 6·63 per cent. loss on complete desiccation. The morphine precipitates of the Pharmacopœia should therefore be dried either at a low temperature (as U.S.P.), or at 110° to 120° C. When an alkaloid has been extracted by alcohol or chloroform, it is much more convenient to dry at 110° to 120° C., assuming that there is no decomposition at this temperature, as the alkaloid may only slowly attain a constant weight in the water-bath. In the case of salts which lose only a proportion of their water under 100° C., and the remainder at a higher temperature, it is preferable to dry at the higher temperature so as to estimate the total water of hydration.—A. S.

Morphine in Opium and its Preparations, Assay of. A. Grandval and H. Lajoux. J. Pharm. Chim. 1897, [6], **5**, 153.

Opium.—10 grms. of opium are triturated in a glass mortar with 40 c.c. of water until thoroughly disintegrated, then thrown on a small pleated filter, washing the mortar with another 40 c.c. of water, which is poured over the filter. This is thoroughly drained, the filter and its contents again rubbed down with a third portion of water and filtered, and washed until the filtrate is free from bitterness. The washings and the filtrates are evaporated together until reduced to 13 grms., the same weight of alcohol (95 per cent. by volume) added, and the mixture allowed to stand for half an hour to permit the sulphate or meconate of lime to separate; the liquid is then filtered through a small filter previously moistened with 60 per cent. alcohol. The precipitate is washed with (60 per cent.) alcohol, which is dropped gradually on to the edge of the filter, kept carefully covered with a glass disc to prevent drying of the

edges. To the filtrate, ammonia is added cautiously until a slight ammoniacal odour persists, stirring is continued for some minutes, when the morphine is gradually precipitated in the tracks of the rod. The mixture is allowed to stand for 12 hours in the cool, and the precipitated alkaloids collected on a tared filter, thoroughly drained, and cautiously washed with 40 per cent. alcohol until the filtrate is colourless; this should require about 25 c.c. The filter and its contents are then well drained, dried at 100°, and weighed. The weight of morphine and narcotine is thus obtained. The filter is again placed in the funnel and washed first with 5 grms. of ether and then with 10 grms. of chloroform, which latter solvent removes the narcotine. The filter is again dried and the morphine weighed as hydrated morphine. It is stated that chloroform does not dissolve hydrated morphine, although it acts as a solvent upon the anhydrous alkaloid.

Extract of Opium.—5 grms. of extract are dissolved in 5 c.c. of water, 5 grms. of alcohol of 95 per cent. added, allowed to deposit, and filtered through a small filter, moistened with alcohol of 60 per cent. The precipitate is washed with 40 c.c. of alcohol of 40 per cent., and the assay continued as described above.

Tincture of Opium.—26 grms. of the Codex tincture (corresponding to 2 grms of the extract) are evaporated to 7 grms., 5 grms. of alcohol of 95 per cent. are then added drop by drop and a slight excess of ammonia. The assay is completed as above.

Special minutiæ for manipulation in the case of assaying Rousseau's laudanum, Black drop, and Sydenham's laudanum, are given.—J. O. B.

Antipyrine, Chemical Estimation of. C. Kippenberger. Zeits. Anal. Chem. 1896, **35**, 659.

On the addition of a solution of iodine in an iodide, to solutions of antipyrine in water, acidified or not, there is produced antipyrinehydriodide periodide, $C_{11}H_{12}N_2O.HI.I_2$ (compare this Journal, 1896, 223, 747), this is a brown tarry mass which does not crystallise on standing, is insoluble in water and in dilute hydrochloric and sulphuric acids, soluble in alcohol and chloroform, and especially in acetone. Hydriodic acid and its salts increase the solubility of the precipitate in water; it separates quantitatively in the presence of sufficient iodine solution, and may be used for the separation of antipyrine from phenacetin, sulphonal, acetanilide, and aniline salts, if acid be present, hydrochloric acid being most suitable. In the presence of aniline salts the filtration of the separated periodide should be accomplished as rapidly as possible. The details of the process are as follows:—To the solution of antipyrine, which should be as concentrated as possible, there is added in a stoppered flask an iodine solution, made by mixing 100 c.c. of $\frac{1}{20}$ N iodine solution, containing 10 or 20 grms. of potassium iodide to the litre, with about 4 c.c. of hydriodic acid of sp. gr. 1·7 (52 per cent. HI). Only a small excess of iodine solution is added and the flask is then shaken until the liquid becomes clear, the precipitate adhering to the sides of the flask. The liquid is filtered through a small asbestos filter, the filtrate being best collected in a dry burette. In an aliquot part of the filtrate the iodine is determined by $\frac{1}{20}$ N thiosulphate. 21·3 c.c. of $\frac{1}{20}$ N iodine solution are equivalent to 0·1 grm. of antipyrine. The error due to solubility of the periodide in solutions of iodides is negligible unless absolute accuracy be required, in which case the iodine solution should be standardised against an antipyrine solution of known strength. Iodine solution in large excess would hinder this solvent action of hydriodic acid or its salts, but irregularities in the action of the iodine occur. Instead of the iodine solution containing hydriodic acid, as described above, a solution containing more potassium iodide may be used, but in that case hydrochloric acid must be added, and the reaction takes longer time. Salipyrine (antipyrine salicylate) and other antipyrine salts may be estimated in the same way.—A. C. W.

Oil of Lemon, Analysis of. A. Soldini and E. Bertè. Chem. and Druggist, 1897, **50**, [872], 25.

The authors state that in using the method of fractional distillation and subsequent polarisation of the distillate, for detecting adulteration of oil of lemon with oil of turpentine and oil of orange (Schimmel and Co.'s Bericht, Oct. 1896, this Journal, 1896, **15**, 925), they have found that the distillate from pure lemon oil invariably has a higher rotation than the original oil, and not a lower, as is stated to be the case.

For the estimation of citral, the following method is used:—A solution of bisulphite of potash is shaken up with the oil of lemon, whereby the whole of the citral in the oil is fixed and separated from the limonene and other compounds that have no aldehydic function. The decrease in volume is then estimated under identical conditions of temperature, and the volume of citral calculated. When relatively large quantities of turpentine or orange oils and even limonene are present, a correction must be made. The greater the proportion of citral to be estimated, the larger is the difference between the ascertained and the calculated percentage, but oils do not generally contain more than 7·5 per cent. of citral. The presence of turpentine, oil of orange, and even limonene somewhat falsifies the results. An oil containing less than 5·5 per cent. by weight of citral should be viewed with suspicion, but should not be described as adulterated if neither oil of turpentine nor any other foreign matter can be found present.—A. S.

Guaiacol and Creosotes, Rapid Method of Analysis. L. Adrian. Bull. de Therapeut. [Sec. Pharmacol.], 1897, **2**, 1.

The method is a colorimetric one, based on the solubility of guaiacol in water and the colour reaction afforded by that body with nitrous acid. From 5 to 6 grms. of the guaiacol or creosote to be tested, are placed in a flask of about 200 c.c. capacity along with some water, shaken for two minutes and filtered; this filtrate will contain more or less guaiacol, according to the quality of the substance tested; it is diluted with twice its volume of water, and 1 c.c. of this liquid is placed in a test-tube, which is then filled to one-third with more water, and the mixture is well shaken; in the meanwhile a 10 per cent. aqueous solution of sodium nitrite is prepared, 2 drops of which are added by means of a dropping tube to the mixture, followed by a drop of nitric acid, when the colour reaction is slowly developed. Commercial guaiacol containing less than 50 per cent. of the pure body will give by this test a yellowish coloration, quite distinct from that obtained with a rich sample. The guaiacol content of a sample may be approximately determined according to this method, by preparing artificial mixtures of guaiacol free from creosote, containing from 10 to 90 per cent. of that body subsequently added. These types are treated with water and sodium nitrite as above described; the intensity of the colour of the sample under examination is then matched by means of these preparations of know guaiacol strength.—J. O. B.

Musk-Pouches, Identification of Adulterated, by Means of the Röntgen Rays. Wolff. Pharm. Centr. H. N. F. 1896, **17**, 827.

The transparency of musk-pouches under the Röntgen rays allows of the ready detection of the presence of fragments of lead used for adulterating and adding weight. A photograph is unnecessary, as the adulterant stands out quite plainly on the dark background.—J. L. B.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Onocerin. H. Thoms. Ber. **29**, 2985—2991.

This body is obtained from the roots of *Ononis spinosa L.* (*Papilionaceæ*) by boiling out with 90 per cent. alcohol. The residue, on cooling, is treated with 60 per cent. alcohol, when the onocerin is left behind. By dissolving in absolute alcohol and passing the solution through animal charcoal, very small colourless prisms are obtained on evaporating off the alcohol. The solubility of onocerin in alcohol is very slight (0·25 per cent. in hot absolute alcohol). It is also only slightly soluble in acetic ether, ether, and chloroform, but is readily soluble in amyl alcohol and turpentine. The author shows, from a number of analyses made of the compounds formed by onocerin with acetic anhydride,

bromine, &c., that the formula of the body is $C_{26}H_{44}O_2$. On oxidation with potassium bichromate a ketone is formed, and from this a hydrazone by treating with phenylhydrazine. Analyses of these bodies lead the author to the conclusion that there is present in onocerin a dihydroxysecondary alcohol, which on oxidation is converted into a diketone. Experiments were made to see whether this ketone is further oxidised by chromic acid mixture. A substance was obtained, the smell of which resembled butyric acid, but the yield was so small that it could not be chemically examined. The above-mentioned secondary alcohol ($C_{26}H_{44}O_2$) only differs from cholesterol by one atom of oxygen.—W. P. S.

Stannous Iodide, Apparent Action of Light in Inducing Crystallisation of. C. J. H. Warden, Pharm. J. 1897, **58**, [1387], 61.

A sample of stannous iodide, prepared by acting on stannous chloride with potassium iodide, was placed in a stoppered flint-glass bottle and exposed to diffused daylight, a piece of thin white paper cut in the shape of a cross being gummed to the bottle by the top of the cross. After some weeks, that portion of the contents not protected by the cross, was coated with ruby-coloured needles, while the salt behind the paper cross was apparently amorphous.
—A. S.

Linseed Meal or Mustard Flour, Adulteration of with Colza Marc. Jaworowski. Pharm. Zeits. für Russ. 1896, **35**.

The presence of colza-seed marc in linseed or mustard flour may be detected by boiling 2 or 3 grms. of the sample with a solution of 10 grms. of salt, 20 grms. of water, and 30 centigrms. of dilute hydrochloric acid. Care should be taken not to add the powder to the solution until the latter has reached the temperature of 70°; on cooling, the mixture is filtered and neutralised with sodium carbonate, and two or three drops of a 1 per cent. solution of ferricyanide of potassium are then added. If the powder is pure no coloration takes place, but if colza marc is present, a brown, red, or violet reaction is obtained.
—J. O. B.

Luciferase, a Light Producing Zymase. L. Dubois. J. Pharm. Chim. 1897, [6], **5**, 81.

The light produced by glow-worms, by putrifying fish, and other organised light giving structures, is stated to be derived from a peculiar ferment, which the author has named luciferase. From his experiments it appears that this physiological light results neither from combustion nor directly from slow oxidation, although oxygen is necessary for the production of the light, luciferase acting as a carrier of oxygen. The luminous organs of glow-worms as well as the eggs in the ovary of the female insect, give a blue coloration with tincture of guaiacum. The same reaction is given by the mucus obtained from the body of dead fish, when treated with chloroform water and filtered.—J. O. B.

Prizes.

Chemical Prizes by the Belgian Government. Chem. and Druggist, March 13, 1897, 416.

The Belgian Government offer several prizes, ranging in value up to 20,000 fr., for the best solutions of questions of industrial and scientific interest. The questions relating to chemistry are the following :—(1.) The determination of the constitution of camphor by means of analytical and synthetical reactions, and the differentiation of the optic isomers with the aid of chemical reactions. (2.) A practical method for the cheap conversion of atmospheric nitrogen into NH_3. (3.) A practical method for the preparation of chlorine from calcium chloride at a less cost than that now in use. (4.) A new process, better than the methods now known, for dyeing various textile yarns. (5.) A simpler method for the preparation of HI. (6.) Improvements in fractional distillation. Full details concerning these questions may be had upon application to the General Commissary of the Belgian Government, 17, Rue de la Presse, Brussels.

New Books.

Traité Élementaire de Chimie, à l'usage des Candidats au Certificat d'Aptitude des Sciences Physiques, Chimiques et Naturelles et des Candidats aux Baccalauréats Scientifiques. Chimie Minérale. 1re Partie. Par A. Haller et P. Th. Muller. Georges Carré et C. Naud, 3, rue Racine, Paris. 1896. Price 6 fr.

8vo volume containing 336 pages of subject-matter, illustrated with 50 wood engravings, and an alphabetical index of subject-matter.

The term "Chimie Minérale" is clearly intended to represent what we usually term "Inorganic Chemistry."

Chimie Organique. 2me Partie. Par A. Haller et P. Th. Muller. Georges Carré et C. Naud, 3, rue Racine, Paris. 1896. Price 4 fr.

8vo volume containing 205 pages of subject-matter, illustrated with two woodcuts. There is no alphabetical index of subjects treated of.

Recueil de Procédés de Dosage, pour l'Analyse des Combustibles, des Minerais de Fer, des Fontes, des Aciers et des Fers. Par G. Arth, Professeur de Chimie Industrielle à la Faculté des Sciences de Nancy. Georges Carré et C. Naud, 3, rue Racine, Paris. Price 8 fr.

8vo volume, containing 313 pages of subject-matter, illustrated with 61 wood engravings. There is no alphabetical index, but the work closes with a classified table of contents.

The text is divided into three parts:—Part I., devoted to the Assay and Analysis of Fuels and Combustibles; Part II., to the Analysis of Minerals; and Part III., to that of Metallic Products, Iron, Cast Metal, and Steels.

Précis d'Analyse Chimique. Première Partie. I. Analyse Qualitative. Par E. Fink, Chef des travaux pratiques d'Analyse à l'École Municipale de Physique et de Chimie Industrielle de la Ville de Paris. Georges Carré, 3, rue Racine, Paris. 1896. Price 3·50 fr.

This little work on Qualitative Analysis contains 190 pages of subject-matter, and is illustrated with 13 wood engravings. At the end of the book is given a blank form of the record of curriculum to be followed and filled up by each laboratory pupil of the École Municipale. The classification of the work is developed in the table of contents, which is placed at the end, but there is no alphabetical index.

Précis d'Analyse Chimique. Deuxième Partie. II. Analyse Quantitative. Par E. Fink. Georges Carré et C. Naud, 3, rue Racine, Paris. 1896. Price 5 fr.

This small 8vo volume contains 312 pages of subject-matter, and is illustrated with 60 wood engravings. A table of contents is given at the end of the work, so classified as to render an alphabetical index unnecessary.

Les Nouveautés Chimiques pour 1897. Nouveaux Appareils de Laboratoires, Méthodes Nouvelles des Récherches Appliquées à la Science et à l'Industrie. J. B. Baillière et Fils, 19, rue Hautefeuille, Paris. Poulenc Frères, 92, rue Vieille-du-Temple, et boul. St. Germain 122. 1897. Price 2·50 fr.

This is an illustrated volume of small 8vo size, containing a descriptive account of new laboratory apparatus and methods of research applied to science and industry, for the year 1897. It is illustrated with 160 wood engravings, contains 235 pages of subject-matter, preface, classified table of contents, and alphabetical index.

Short Studies in Physical Science, Mineralogy, Chemistry, and Physics. By Vaughan Cornish M.Sc. 1897. Price 5*s*.

8vo volume, containing frontispiece, preface, table of contents, and text nearly filling 225 pages, with an alphabetical

index. There are three illustrative engravings. One chapter of the work is devoted to "Chemical Manufacture." It contains three sections:—Sect. I. The Fuel of Blast Furnaces; Sect. II. Explosives; and Sect. III. By-Products and Waste Products.

A Handy Book for Brewers, being A Practical Guide to the Art of Brewing and Malting. Embracing the conclusions of Modern Research which bear upon the Practice of Brewing. By Herbert Edwards Wright, M.A. Second Edition; thoroughly revised. Crosby, Lockwood, and Son, 7, Stationers' Hall Court, Ludgate Hill. 1897. Price 12*s.* 6*d.*

This work contains prefaces to first and second editions, table of contents, and subject-matter extending to page 503; then follow two Appendices: A. A list of the Hop-growing Parishes of Kent; and B. A Synoptic Table of the Various Processes in Malting and Brewing, with some operative details and abstract of the Chief Physical and Chemical Changes. The work ends with an alphabetical index of subjects. The text is subdivided into thirteen sections or chapters, repectively headed as follows:—I. Introductory. II. Barley, Malting, and Malt. III. Water for Brewing. IV. Hops and Sugars. V. The Brewing Room. VI. Chemistry as applied to Brewing. VII. The Laboratory. VIII. Mashing, Sparging, and Boiling. IX. Ferments in General. X. Fermentation with Commercial Yeast: its Science and Practice. XI. Culture from a Single Cell. Wild Yeasts. XII. Treatment of Beer. XIII. The Brewery and Plant.

Tabellen für Gasanalysen, gasvolumetrische Analysen, Stickstoffbestimmungen, &c. Von Prof. Dr. G. Lunge. Friedrich Vieweg und Sohn, Brunswick, Germany. 1897. Price M. 2. H. Grevel and Co., 33, King Street, Covent Garden, London.

These tables are five in number, and are arranged in sheets, which could be detached from the book-cover and hung upon the walls of a laboratory. The chief tables, I. and II., were calculated out by the author in 1879, and published in Dingler's Polyt. J. **231**, 522. A supplement to Table II., embracing the pressures from 680 to 708 mm., was given by Lwoff in the Zeits. f. angew. Chem. 1893, 433 (see also this Journal, 1893, 1030).

Table I. is for the Reduction of the Volume of Gas found, to zero Centigrade; whilst Table II. is for the "Reduction of the Volumes of Gas found to the Normal Barometric Pressure of 760 mm."

Table III. is for the Tension of Aqueous Vapour at Temperatures from 10° to 26° C.

Table IV. serves specially for "Nitrometric" Analyses in which nitric oxide is to be measured. It equally serves in the interpretation of the results of the Schlösing-Grandeau process.

Table V. is for the "Conversion of the c.c. read off into Milligrms. of Substance," and may be used for the gasometric determinations involved in Dumas' Method of Nitrogen Determination, in that of the Nitrometer and similar Apparatus when used for Determinations of Carbonic Acid, Active Oxygen (from Permanganates), Manganese Peroxide, Chlorine (from Hypochlorites), Nitrogen or Ammonia (according to the Sodium Bromide Method, the numbers being so calculated as to render dispensable Dietrich's Table).

Chemisch-Technisches Repertorium. Uebersichtlicher Bericht über die neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der technischen und industriellen Chemie, mit Hinweis auf Maschinen, Apparate und Literatur. Herausgegeben von Dr. Emil Jacobsen. 35. Jahrgang. 1896. Erstes Halbjahr. Zweite Hälfte. R. Gaertner's Verlagsbuchhandlung, Hermann Heyfelder, Schönebergerstrasse 26, Berlin, S.W. 1897. H. Grevel and Co., 33, King Street, Covent Garden, London.

The second issue for the first half-year of 1896 of Jacobsen's Repertory of Chemical Technology, containing reports of the progress of the following branches of chemical industry:—I. Foods. II. Paper. III. Photography. IV. By-Products, Residues, Manures, Sanitation, Disinfectants, &c. V. Soaps. VI. Explosives, Matches, &c. VII. Preparation and Purification of Chemicals. VIII. Chemical Analysis. IX. Apparatus, Machinery, Electro-technology, Thermo-technology, Appendix, and New Books.

Theorie und Praxis der Analytischen Elektrolyse der Metalle. Von Dr. Bernhard Neumann. W. Knapp, Halle a/S. 1897. Price M. 7 H. Grevel and Co., 33, King Street, Covent Garden, London.

This work contains preface, table of contents, subject-matter filling 216 pages, short appendix, and alphabetical index. The text is illustrated with 65 engravings, and is subdivided into three Sections:—I. Theory of Electrolysis. II. The Production, Measurement, and Regulation of Current; and III. Carrying out the Electrolysis. A. General. B. Separation of Metals from Solutions of Pure Salts. C. Separation of Metals; and D. Technological Examples.

Elektrochemische Uebungsaufgaben für das Praktikum, sowie zum Selbstunterricht. Zusammengestellt von Dr. Felix Oettel. W. Knapp, Halle a/S. 1897. Price M. 3. H. Grevel and Co., 33, King Street, Covent Garden, London.

8vo volume containing preface, table of contents, and subject-matter covering 53 pages, illustrated with 20 engravings. This series of Exercises in Electrolysis is classified according to the following scheme:—General Instructions as to the necessary arrangements, apparatus, &c. I. Testing the Apparatus for Measurements. II. Dependence of the Resistance of the Bath on the Factors: Current Density; Concentration and Temperature of the Solution; Distance of Electrodes. III. Influence of Current Density and Concentration on the Course of Electrochemical Reactions. IV. Employment of Gas Analysis in carrying out Electrochemical Reactions. V. Experiments with Diaphragms. VI. Precipitations of Metals with Soluble and Insoluble Anodes. VII. Introduction of Contributory Reactions. VIII. Experiments with Glowing and Fused Electrolytes. IX. Experiments with Double-pole Electrodes. X. Organic Electrolyses.

The Chemistry of Dairying. An Outline of the Chemical and Allied Changes which take place in Milk, and in the Manufacture of Butter and Cheese; and the Rational Feeding of Dairy Stock. By H. Snyder, B.S., Professor of Agricultural Chemistry, University of Minnesota, &c. Chemical Publishing Co., Easton, Philadelphia. 1897. Price $1·50.

Small 8vo volume, with preface, table of contents, and subject-matter filling 137 pages. Supplementary tables with Average Composition of American Feeding-Stuffs, &c., and appendix with Questions for use in the Class-room, and an alphabetical index. The pages are illustrated with 21 woodcuts. The book, as the author says, has "been prepared with the object of furnishing useful information to a class of young men who intend to become farmers and dairymen, rather than scientific experts." The character of this information will appear in the following excerpt of the table of contents:—I. General Composition of Milk. II. Milk-testing. III. Milk Fats. IV. Milk Sugar and Lactic Acid. V. The Lactometer and the Detection of Adulterated Milk. VI. The Chemistry of Butter making. VII. The Chemistry of Cheese-making. VIII. The Ash, and Miscellaneous Compounds of Milk. IX. Dairy Salt and Commercial Problems relating to Milk. X. The Sanitary Condition of Cows' Milk, and that of other Domestic Animals. XI. Preserving Milk. XII. Composition of Skim-milk, Buttermilk, and Whey. XIII. Other Simple Methods used in Milk-testing, and the Adulteration of Dairy Products. XIV. The Effects of Food upon the Quality of Dairy Products. XV. Composition of Fodders and the Calculation of Rations.

Trade Report.

TARIFF CHANGES AND CUSTOMS REGULATIONS.

(*From the Board of Trade Journal.*)

France.

Tariff Decision.

Thorium salts are to pay a specific duty of 20 francs per kilo., instead of the previous *ad valorem* rate.

Tunis.

Reduction of Export Duty on Halfa Grass.

Mr. Haggard encloses copy of a decree published in the *Journal Officiel Tunisian* of the 16th February 1897, reducing the export tax on halfa grass from 1 fr. 27 cts. to 50 cts. a metric quintal.

Italy.

Tariff Decision.

According to the *Gazette Ufficiale* of 9th January last, alcoholic solutions of colours are assimilated to spirit varnishes, and should be classified under No. 81A of the Customs tariff.

United States.

Tariff Decisions.

Carbonate of strontia is assessed for duty at 25 per cent. under paragraph 60, Act of 1894.

Lysidine, a medicinal coal-tar preparation, is dutiable at 25 per cent. under paragraph 443.

Perfumed smelling salts are dutiable at 25 per cent. as chemical salts.

Queensland.

Exemptions from Duty.

Under an Act recently passed by the Legislature of Queensland, the following articles will be exempted from duty from the specified dates :—

30th September 1896.	31st March 1897.
Alkaline earth.	Artists' colours.
Alumina.	Asbestos (unmanufactured).
Antifermentine.	Asphalt.
Cotton-seed oil.	Bark for tanning.
The following drugs and chemicals :—	Castor oil in bulk.
Cyanide of potassium.	Chalk.
Sulphate of soda.	Cylinders and tubes containing gas and ammonia.
Nitrate of soda.	Drugs and chemicals (except those already exempted).
Caustic soda.	Explosives.
Sulphate of baryta.	Electro-plating materials.
Sulphate of iron.	Gold and silver leaf.
Sulphate of magnesia.	India-rubber solution.
Sulphate of potash.	Leather.
Sulphide of sodium.	Scientific instruments and materials.
Fish oil.	Photographic instruments and materials.
Infusorial earth.	Paper.
Linotype metal.	Resin.
Liquid stain for leather.	Sheep dip.
Myrobolans (unground).	Stockholm tar.
Phosphorised pollard.	Tinfoil.
Pottery materials.	
Preservitas.	
Sizeoline and hytra-size.	
Soap colourings.	
Valonia (unground).	

OFFICIAL NOTICES.

Carbide of Calcium.

The following is the memorandum which has been issued by Colonel Sir Vivian Majendie, Her Majesty's Inspector of Explosives, under date of March 1 :—

With a view to assisting local authorities in dealing with applications for licences to keep carbide of calcium, the following observations as to the character and prevention of the risks attaching to such keeping have been drawn up :—

1. Carbide of calcium is a solid substance which, while not itself inflammable, evolves, when brought into contact with moisture, a gas (acetylene) which is of a highly inflammable character.

2. This gas, when mixed with air, or when under even slight compression, becomes powerfully explosive.

3. Carbide of calcium, unless carefully manufactured from suitable materials, is liable to contain impurities which, when the carbide is acted on by water, evolve phosphoretted or siliciuretted hydrogen- gases which, when evolved in appreciable quantities, would render the acetylene produced liable to spontaneous ignition.

4. In addition to the risks indicated above, acetylene gas is capable of forming an explosive compound when brought into contact with copper.

5. The character of the apparatus to be used in connection with the production of acetylene gas from carbide of calcium is intimately connected with the question of safety; for example, grave risk may arise from the heat developed by a too rapid conversion of the carbide into acetylene gas; or from the apparatus permitting escape or leakage of gas which is being formed, or not allowing a free passage of the gas from the generator into the holder, and so causing excess of pressure in the generator or other part of the apparatus.

6. Risk might also arise if proper provision were not made for dealing with the residue of the carbide of calcium which has been used for making acetylene gas (*viz.*, lime), as such residue might contain carbide of calcium which has escaped decomposition.

It will therefore be obvious that local authorities, in granting licences for carbide of calcium, should have regard to the foregoing risks, and with that view should make provision :—

(*a.*) For the exclusive use of hermetically closed packages for the keeping and conveying of carbide of calcium.*

(*b.*) The adequate ventilation of the place where the carbide of calcium is present.

(*c.*) The prohibition of any powerful compression of the gas produced in the apparatus or receptacles employed.

(*d.*) The keeping and use of pure carbide of calcium only, and the establishment of efficient arrangements for the sampling and testing of the carbide.

(*e.*) The exclusion of copper from all vessels or apparatus used with or for carbide of calcium and the gas produced therefrom.

(*f.*) The use only of an apparatus which the local authority have satisfied themselves, under competent advice, is of a safe and suitable character, and the prohibition of the employment for the manipulation of such apparatus of any person other than a properly instructed and capable operator.

(*g.*) The safe disposal of the residue and the prohibition of its introduction into sewers, cesspools, &c., unless mixed with at least ten times its bulk of water.

It will, in all cases, be for the local authority very carefully to determine whether the premises proposed to be licensed are themselves suitable for the purpose, regard being had to the formidable consequences (from fire or explosion) which might result from the careless or imperfect observance of any of the imposed conditions, especially in dwellings or in places or premises where large numbers of persons are liable to assemble.

Acetylene.

Chem. and Druggist, March 6, 374.

The Associated Fire Offices have drawn up the following regulations in regard to insurance of premises in which the generation or storage of acetylene is carried on :—

(1.) That the storage of calcium carbide, liquid acetylene, and (or) acetylene gas, and every apparatus, whether portable or otherwise, for the generation of such gas, must be only in a building detached at least 10 ft. at the nearest point from every other building.

* The 9th section of the Petroleum Act specifies, among the conditions which may be included in a licence under that Act, the mode of carrying within the district of the licensing authority.

(2.) That suitable valves or devices for controlling the pressure of the gas must be placed inside the detached building, and a cut-off provided between such building and the building supplied with the gas.

(3.) That in addition to the above, the piping in the building in which the gas is used must be provided with a pipe-outlet into the open air, controlled by a safety device so arranged as to let the gas escape outside the building whenever the pressure of the gas exceeds 4 oz. to the square inch.

It should be understood that fire insurance offices will not admit liability for explosion of acetylene gas occurring elsewhere than in any building which is, or the contents of which are, the subject of the insurance effected under the above rules, which, however, are under revision, as they have been found to be practically prohibitive of the use of acetylene.

STATISTICS.

German Trade Returns for 1896.

Ch. of Comm. J., Feb., 39.

The trade returns issued by the Imperial Statistical Office provide the following statement by classes of the values (in 1,000's of marks) of imports and exports during 1895 and 1896:—

Classes of Goods.	Imports.		Exports.	
	1896.	1895.	1896.	1895.
Lead and leadware	7.440	6,487	10,805	11,641
Drugs, dyestuffs, &c. ...	243,810	232,913	318,414	298,809
Iron and ironware	46,987	30,765	322,785	301.792
Earths, ores, precious metals, &c.	373,494	233,341	282,584	152,883
Glass and glassware	10,705	9,659	43,381	46,956
Hides and skins	170,290	186,378	75,659	81,825
Hops	9,884	6,612	24,866	27.291
Caoutchouc and gutta-percha and wares of.	42,903	35,736	30,460	27,432
Copper and copper wares	69,517	55,537	84,546	72,960
Hardware, cutlery, &c. .	39,897	36,117	116.997	73,500
Leather and leather wares.	57,518	54,808	151,316	165,807
Candles..................	102	115	658	581
Oils and fats............	144,205	136,518	27,140	26,394
Paper and paste goods ..	12,626	10,754	101.172	104,354
Petroleum	80,959	76,321	1,325	1,151
Soap and perfumery	1,850	2,628	12,450	10,734
Stone and stoneware ...	31,915	31,756	25,577	21,860
Coal and other fuel	117,150	110,742	157,395	145,769
Tar, pitch, resin, and asphalt.	37,720	33,353	9,759	8,312
Potteryware	6,141	5,527	43.136	38,646
Oilcloth, &c.	973	1,235	1,310	1,140
Zinc and zincware	5,487	5,294	11,528	25,038
Tin and tinware	18,038	13,927	5,273	4.923
Total value (1000 m.) .	4.573,448	4,246,111	3,631,629	3,424,076

The Mineral Production of Canada.

Eng. and Mining J., March 6, 1897, 232.

The total value of the mineral output of Canada for 11 years past has been reported as below:—

	Dols.
1886	12,000,000
1887	12,500,000
1888	13,500,000
1889	14,500,000
1890	18,000,000
1891	20,500,000
1892	19,500,000
1893	19,250,000
1894	20,900,000
1895	22,000,000
1896	23,627,305

In the table below, which gives the production in 1896, the figures show the quantity or value of the product marketed. Where tons are used, they are short tons of 2,000 lb.

Summary of the Mineral Production of Canada in 1896.

Product.		Quantity.	Value.
METALLIC.			Dols.
Copper (fine, in ore, &c.)..........	Lb.	9,385,556	1,021,148
Gold		..	2,810,206
Iron ore	Tons	88,206	184,313
Lead (fine, in ore, &c.)............	Lb.	24,199,977	721,384
Nickel (fine, in ore, &c.)...........	Lb.	3,500,000	1,155,000
Silver (fine, in ore, &c.)..........	Oz.	3.205,343	2,147,589
Total metallic		..	8,039,640
NON-METALLIC.			
Asbestos...........................	Tons	12,250	429,856
Chromite	,,	2,362	25,982
Coal	,,	3,743,234	8,006,305
Coke	,,	49,817	111,560
Felspar............................	,,	480	1,368
Fireclay	,,	667	1,427
Graphite	,,	..	9,455
Grindstones	,,	3,663	32,810
Gypsum	,,	205,203	174,403
Manganese ore	,,	12	900
Mica		..	60,000
Mineral pigments:—			
Baryta...........................	Tons	145	715
Ochres..........................	,,	2,250	10,925
Mineral water	Galls.	706,372	111,736
Natural gas		..	276.301
Petroleum..........................	Bbls.	726,882	1,155,646
Phosphate (apatite)	Tons	570	3,990
Pyrites	Tons	33,715	101,155
Salt	,,	43,956	169,677
Soapstone	,,	410	1,230
Tripolite	,,	664	9,960
STRUCTURAL MATERIALS AND CLAY PRODUCTS.			
Bricks*		..	1,600,000
Building stone*		..	1,000.000
Cement, natural....................	Bbls.	69,705	60.500
,, Portland...................	,,	78,085	111,005
Flagstones..........................		..	6,710
Granite............................	Tons	18,717	106,709
Lime*		..	650,000
Marble	Tons	74	905
Pottery.............................		..	163,905
Sands and gravels*	Tons	..	120,000
Sewer pipe		..	153,875
Slate		..	53,370
Terra-cotta		..	110,855
Tiles*		..	225,000
Total non-metallic..................		..	15,087,665
Total metallic		..	8,039,640
Estimated value of mineral products not returned.		..	500,000
Total value........................		..	23,627,305

* Partly estimated.

In this table the copper includes the contents of ore and matte, and is valued at 10·88 c. per lb. Lead, which includes that in ores and bullion, is valued at 2·98 c. per lb. Nickel, all of it in ores and matte, is rated at 33 c. per lb. Silver is taken at an average of 67 c. per oz. The value given for natural gas is the gross return from the sales of the gas. In arriving at the quantity of petroleum, the barrel of 42 galls. is taken, but the value is computed at an average of 1·59 dols. per barrel of 35 galls.; 42 galls. refined oil are assumed as equal to 100 galls. crude oils.

The increases shown, in comparison with 1895, are:—Coal, 229,738 tons, or 278,859 dols.; copper, 596,394 lb., or 71,919 dols.; lead, 1,124,085 lb., increase in quantity, but 28,582 dols. decrease in values; silver, 1,129,660 oz., or 988,956 dols.; gold, 899,306 dols. The decreases were:—Iron ore, 14,595 tons, or 53,757 dols.; nickel, 388,525 lb., 205,904 dols.; natural gas, 146,767 dols.

The gain in coal is altogether due to Nova Scotia, which produced 296,153 net tons more than in 1895, but this increased output is offset in a large measure by a decrease of 104,629 tons in the production of British Columbia. Copper shows a large decrease in Ontario and a small increase is Quebec, while British Columbia's contribution is 3,818,556 lb., which amount is 1,848,193 lb. more than in 1895. In gold large increases are to be credited to Nova

Scotia and Ontario, but British Columbia heads the list with an increase over the previous year of 197,675 dols.

The production of lead is altogether that of British Columbia, and has increased in the year by over 1,100,000 lb., but a decrease in the market price of some 8 per cent. lessens the total value by 28,592 dols. Natural gas shows a decrease in value of 116,761 dols., or about 31 per cent., which would seem to be due to a heavy falling off in the production of the Welland gas field. Ontario's nickel production is reduced during the year by nearly 400,000 lb., and shows a decrease in the value of over 200,000 dols. British Columbia shows an output of silver amounting to 3,135,343 oz., valued at 2,100,689 dols., an increase over 1895 of 1,429,660 oz. in quantity, and in value of nearly a million dollars.

GENERAL TRADE NOTES.

Sale of Edible Oils in Belgium.

Ch. of Commerce J., Feb., 36.

A Royal decree, made December 28, will come into force on April 1, regulating the sale of edible oils in Belgium. Thenceforward the receptacles in which edible oils are to be sold, exposed for sale, stored or transported for sale or delivery, must bear an inscription comprising, immediately after the word "oil," in identical character of an equal size to those used for that word, an exact indication of the raw material or raw materials from which the oil has been obtained (olive oil, poppy oil, earthnut oil, sesame oil, cotton oil, olive and sesame oil, &c.). The receptacles must also bear the name or firm as well as the address, or at least the mark of the manufacturer or seller; while the descriptive particulars of the oil must also appear in the invoices, carriers' notes, or bills of lading. The sale or exposure for sale for food purposes of damaged (gâtées) or adulterated (corrumpues) oils is prohibited, as well as sale, exposure for sale, storage for sale or for delivery in the same premises or in premises communicating other than by the public thoroughfare; the carriage simultaneously in the same vehicle, for sale or delivery, of edible condiments and vegetable oils not intended for food, unless the receptacles of the latter bear a label such as "non-edible oil," indicating in distinct characters that it is not intended for use as food. The Minister of Agriculture and Public Works is to put the law into operation, infractions being punishable by penalties provided under the law of August 4, 1890, without prejudice to the penalties established by the Penal Code.

Window Glass in Turkey.

Ch. of Commerce J., Feb., 40.

The French Chamber of Commerce in that city reports that Constantinople requires annually 34,000 to 40,000 cases of window glass, which is almost exclusively imported from Belgium. The quality of this glass is as bad as possible, greenish and defective, and is only preferred on account of its cheapness. The price varies from 15 to 25 francs per case, according to size. There are only a few small glass factories at Constantinople.

A New Turpentine District.

Chem. and Druggist, 1897, **50**, 337.

The pine forests of Georgia and Carolina, which have hitherto supplied most of the turpentine of commerce, becoming worn out, the industry is being transferred further south towards Florida.

Seven large turpentine "farms" have been established within the past two months in Alachua County, Florida, U.S.A.—A. S.

Lead Shipments from the United States.

Eng. and Mining J., Feb. 27, 1897, 205.

Shipments of a considerable quantity of lead have recently been made from the Colorado smelters to Europe by way of Galveston, Tex., going to that port by rail from Pueblo. At present prices of the metal abroad, there is probably more profit in shipping lead in this way than in marketing it in the East, where demand is at present light and sales are made below the European quotations. It is a question, however, how far this trade can be carried on without causing lower prices on the other side. A good deal of Mexican lead, refined in bond, has also been going abroad, and there is a limit to the demand there. The Spanish mines were very active last year, under the stimulus of higher prices, and supplies have been increased until there is some risk that they may exceed the demand; although business is very active all over Europe.

Sulphur Production in Sicily.

Eng. and Mining J., Feb. 27, 1897, 214.

According to the *Rassegna Mineraria*, the exportation of sulphur from Sicily in long tons (2,240 lb.) for the year 1896 was 396,745 tons, as compared with 347,636 tons in 1895 and 328,930 tons in 1894. Nothwithstanding the gain last year of 49,109 tons in the exports, the stocks on hand at the shipping ports show an increase of 19,243 tons. These stocks amounted to 222,999 tons on December 31st, 1896, as compared with 203,756 tons at the close of 1895 and 198,513 tons at the close of 1894. As the consumption at home is comparatively nothing, a comparison of the exports and the stocks shows that the total production in 1896 was 415,988 tons. Of the exports the United States took last year 124,923 tons, a quantity which compares with 99,227 tons in 1895 and 105,773 tons in 1894. The average price at shipping port showed a considerable improvement: in 1894 it was 56·50 lire (10·85 dols.), and fell to 52·10 lire (10 dols.) in 1895, but in 1896 the effect of the combination of producers was shown, and the average rose to 68 lire (13·05 dols.) per ton.

Exports to all countries from Sicily showed an increase in 1896, with the exception of Great Britain, where there was a falling off of 2,000 tons. This was more than made up by an increase of 9,000 tons in the shipments to Sweden, Norway, and Denmark.

Russian Petroleum Exports.

Eng. and Mining J., Feb. 27, 1897, 214.

The exports of oil from Russia during the year 1896 are reported as follows:—Residuum and crude oil, 199,300 bbls.; lubricating oil, 726,200 bbls.; distillate, 846,700 bbls.; illuminating oils, 5,481,400 bbls.; making a total of 7,253,600 bbls. Of this total, Batum shipped 5,243,500 bbls., and Novorossisk the remainder. A Belgian company has been organised to develop the Grosni oil field on a more extensive scale. The work on the pipe line between Baku and Mikalovo, or half the distance between Baku and Batum, will commence as soon as the weather opens. Drilling operations are being pushed actively at Petrovsk.

The Export of Petroleum from the United States.

Imp. Inst. J., March, 93.

It appears from recent returns that the petroleum exported from the United States during the year 1896 was the largest in the history of that important commodity. The shipments of the crude product reached a total of 117,921,276 galls., which shows an increase of 1,967,145 galls. over the exports during 1895. Of naphthas, there were exported last year 13,420,769 galls., being an increase of 662,829 galls. The shipments of illuminating oil amounted to the enormous total of 749,305,844 galls., showing the increase to have been 71,805,197 galls. The exports of lubricating oils have increased by 3,859,578 galls., the total shipments having amounted to 50,629,143 galls. There was a sudden and rapid increase by 364,140 galls. in the exports of residuum, which amounted for the year to 507,990 galls. The total shipments of all petroleum products reached a total of 931,785,022 galls., the increase over 1895 being 78,658,892 galls.

Petroleum in Sumatra.

Eng. and Mining J., Feb. 20, 186.

The Royal Netherlands Petroleum Company has completed a well at Langkat, Sumatra, which exceeds any drilled so far on that island. Its daily production is 4,800 bbls. None of the older wells produced at any time more than 500 barrels. If the production of the new well keep up, it will be able to furnish 3,000,000 cases of refined oil a year.

The Petroleum Committee.

Chem. and Druggist, Feb. 27, 1897, 354.

The Government have reappointed the Select Committee to inquire into and report upon the sufficiency of the law relating to the keeping, selling, using, and conveying of petroleum and other inflammable liquids, and the precautions to be adopted for the prevention of accidents with petroleum lamps. The members of the Committee are Sir Thomas Sutherland, Mr. Jesse Collings, Mr. Alexander Cross, Mr. Flannery, Sir Edward Hill, Mr. Wootton-Isaacson, Mr. Kenyon, Mr. McKillop, Mr. Mundella, Mr. Pollock, Mr. Harold Reckitt, Mr. Compton Rickett, Sir Benjamin Stone, Mr. Tully, and Mr. Ure.

German Patents in 1896.

Chem. and Druggist, Feb. 27, 1897, 336.

The returns of the Imperial German Patent Office for 1896 show that the applications in the year amounted to 16,486, an increase of 1,423 on the previous year. The number of patents granted, however, was smaller—*viz.*, 5,410 against 5,720 in the year before. The great severity with which applications for patents are scrutinised by the German Patent Office is shown by the fact that only one-third of the applications are successful. Eighty applications were made last year for patents relating to alkali and the wholesale preparation of chemicals generally, and 51 of these were granted. The applications for patents for hygienic inventions numbered 3,317, of which 135 were granted; and those for chemical processes and apparatus generally 478, of which 198 were successful.

German Chemical Trade.

Chem. and Druggist, March 13, 1897, 416.

The German trade returns for 1896 show that although the gross weight of chemicals and other articles for use in chemistry and pharmacy imported from abroad exceeds considerably the weight of similar articles exported from Germany, the opposite is the case with regard to the value of the goods. This is apparent from the following figures relating to the imports and exports of drugs, pharmaceutical goods, and dyestuffs:—

	1896.		1895.	
	Tons.	Value.	Tons.	Value.
		M.		M.
Imports	1,021,537	243,810,000	1,001,905	232,913,000
Exports.........	642,425	318,414,000	609,983	289,809,000

The imports show an increase of 1·9 per cent. in weight and 4·6 per cent. in value; the exports an increase of 5·3 per cent. in weight and 6·5 per cent. in value.

The Proposed New United States Tariff.

The *Times* has printed a synopsis of the new Tariff Bill as published in the *Philadelphia Press*, from which the following details relating to chemicals and pharmaceutical goods may be taken as substantially correct, although they are not official. The comparisons are with the existing Wilson tariff:—

Acids.—Acetic, 3 c. per lb. (20 per cent. *ad val.*); boracic, 3 c. (3 c.); chromic, lactic, 4 c. (4 c.); citric, 8 c. (25 per cent. *ad val.*); salicylic, 10 c.; sulphuric, or oil of vitriol, not specifically provided for, $\frac{1}{4}$ c.; tannic, 50 c. per lb. (60 c.); gallic, 10 c.; tartaric, 7 c. (20 per cent. *ad val.*); all other acids not specifically provided for, 25 per cent. *ad val.*

All alcoholic perfumery, 45 c. per lb. and 45 per cent. *ad val.* (against 2 dols. per gallon and 50 per cent. *ad val.*).

Alkalis and all chemical compounds and salts not specially provided for, 25 per cent. *ad val.*

Alumina, alum, alum cake, patent alum, sulphate of alumina, and aluminous cake, and alum in crystals or ground, $\frac{1}{2}$ c. per lb. (against $\frac{4}{10}$ths of 1 c. per lb.).

Ammonia, carbonate of, $1\frac{1}{4}$ c. per lb. (20 per cent. *ad val.*); muriate of, $\frac{3}{4}$ c. per lb. (10 per cent.); sulphate of, $\frac{1}{2}$ c. per lb. (20 per cent.); argols or crude tartar, $1\frac{1}{2}$ c. per lb.; tartars and lees crystals or partly refined argols, 4 c. per lb.; cream of tartar, 6 c. per lb.

Borax, crude, or borate of soda, or borate of lime, 2 c. per lb. (against 2 c. for borate of soda and $1\frac{1}{2}$ c. for borate of lime); refined borax, 3 c. (2 c.).

Camphor, refined, 4 c. per lb. (10 per cent.). Chloroform, 20 c. per lb. (25 c. per lb.).

Collodions, rolled or in sheets, 50 c. per lb.; if unfinished or partly finished articles, including celluloid articles, 75 c. per lb. and 45 per cent. (45 per cent. *ad val.*).

Ethers, sulphuric and spirits, nitrous ether, 25 c. per lb.; fruit ethers, oils or essences, 2 dols. per lb.; other ethers, 1 dol., provided no article pays less rate than 25 per cent. *ad val.*

Iodine, resublimed, 20 c. per lb.

Malt extract, fluid, in casks, 25 c. per gallon (15 c.); in bottles or jugs, 40 c. per gallon (30 c.); solid or condensed, 40 per cent. *ad val.* (30 per cent. *ad val.*).

Potash, bichromate and chromate, 3 c. per lb. (25 per cent.); caustic, 1 c.; chlorate, 3 c.; prussiate of, red, 8 c.; cyanide of, 6 c.; prussiate of, yellow, 4 c. (25 per cent.).

Quicksilver, 10 c. per lb. (7 c. per lb.).

Soda bicarbonate, $\frac{3}{4}$ c. per lb. ($\frac{1}{2}$ c.); bichromate and chromate, 2 c. (25 per cent.); crystal carbonate, $\frac{1}{2}$ c.; caustic, $\frac{1}{2}$ c.; nitrate, 2 c. and $2\frac{1}{2}$ c.; crystals, not concentrated, and soda ash, $\frac{1}{4}$ c. (against $\frac{1}{8}$ c. for crystals); sulphate of soda, 1 dol. 25 c. per ton.

Sulphur, refined, 8 dols. per ton (20 per cent.).

Bay rum or bay water, whether distilled or compounded or first proof, and in proportion for any greater strength than first proof, 1 dol. 50 c. per gallon (against 1 dol. per gallon).

Waters (aërated and mineral).—Ginger-ale, ginger-beer, lemonade, soda-water, &c., in bottles containing each not more than three-fourths of a pint, 18 c. a dozen; in $\frac{3}{4}$ to $1\frac{1}{2}$ pint bottles, 28 c. per dozen; but no separate or additional duty shall be assessed on the bottles (against ginger-beer or ginger-ale, 20 per cent. *ad val.*).

All mineral waters, all imitations of natural mineral waters, and all artificial mineral waters not specially provided for, in bottles up to 1 pint, 30 c. per dozen bottles; if containing more than 1 pint and not more than 1 quart, 40 c. a dozen bottles, but no separate duty shall be assessed upon bottles (against 20 per cent. *ad val.*).

Methylated Spirit in India.

Chem. and Druggist, March 20, 459.

The quantity of spirit methylated in Bengal last year shows a considerable increase. In 1894–95 it was 54,282 gallons; in 1895–96, 59,854 gallons. Methylated spirits are, of course, professedly imported or prepared for use in arts and manufactures and in chemistry, and in this idea the duty thereon is fixed at a low rate of 5 per cent. *ad valorem*. It has lately been shown, however, that spirit methylated with wood-naphtha can be manipulated into a drinkable spirit, and the Bengal administration have therefore taken under consideration a scheme for denaturing the spirit in a more effective way. The medium with which the spirit is to be rendered "completely and permanently unfit for human consumption" is "caoutchoucine," with which it is to be mixed.

BOARD OF TRADE RETURNS.

Summary of Imports.

Articles.	Month ending 28th February. 1896.	1897.
	£	£
Metals	1,669,857	1,830,336
Chemicals and dyestuffs	917,247	673,474
Oils	777,085	711,481
Raw materials for non-textile industries.	2,746,428	2,795,651
Total value of all imports	35,476,736	37,245,164

Summary of Exports.

Articles.	Month ending 28th February. 1896.	1897.
	£	£
Metals (other than machinery)	2,589,972	2,542,282
Chemicals and medicines	717,887	745,880
Miscellaneous articles	2,660,224	2,414,666
Total value of all exports	19,704,095	17,864,682

Imports of Metals for Month ending 28th February.

Articles.		Quantities. 1896.	1897.	Values. 1896.	1897.
				£	£
Copper:—					
Ore	Tons	7,165	2,271	53,086	27,032
Regulus	„	6,192	9,148	159,842	273,661
Unwrought	„	4,377	4,019	193,159	202,854
Iron:—					
Ore	„	450,081	586,168	304,876	449,146
Bolt, bar, &c.	„	2,618	3,307	22,017	25,243
Steel, unwrought	„	587	7,358	6,150	39,117
Lead, pig and sheet	„	15,874	13,004	174,132	150,374
Pyrites	„	48,219	54,671	86,590	81,785
Quicksilver	Lb.	1,652,223	1,257,023	157,594	115,359
Silver ore	Value £	..	..	107,842	95,000
Tin	Cwt.	59,181	27,938	180,982	84,284
Zinc	Tons	6,552	5,908	85,554	86,031
Other articles	Value £	..	..	138,033	200,150
Total value of metals		..	..	1,669,857	1,830,336

Imports of Chemicals and Dyestuffs for Month ending 28th February.

Articles.		Quantities. 1896.	1897.	Values. 1896.	1897.
				£	£
Alkali	Cwt.	5,869	13,775	4,945	6,728
Bark (tanners', &c.)	„	12,803	16,290	4,216	5,660
Brimstone	„	82,740	40,392	13,486	10,088
Chemicals	Value £	..	..	118,834	94,318
Cochineal	Cwt.	77	119	463	680
Cutch and gambier	Tons	3,235	1,863	65,082	26,654
Dyes:—					
Alizarin	Value £	..	..	25,096	21,587
Anilin and other	„	..	..	38,421	41,764
Indigo	Cwt.	21,958	17,249	422,505	307,649
Nitrate of potash	„	24,310	29,706	25,494	22,648
Valonia	Tons	5,149	2,803	36,729	28,993
Other articles	Value £	..	..	161,976	106,725
Total value of chemicals		..	..	917,247	673,474

Imports of Oils for Month ending 28th February.

Articles.		Quantities. 1896.	1897.	Values. 1896.	1897.
				£	£
Cocoa-nut	Cwt.	27,439	21,275	31,756	24,115
Olive	Tuns	3,161	1,852	107,704	59,760
Palm	Cwt.	94,689	62,906	99,069	65,313
Petroleum	Gall.	18,094,640	17,663,525	373,961	322,603
Seed	Tons	2,618	3,196	52,962	68,911
Train, &c.	Tuns	927	254	15,496	4,869
Turpentine	Cwt.	10,636	75,539	10,810	77,081
Other articles	Value £	..	..	85,327	88,829
Total value of oils		..	..	777,085	711,481

Imports of Raw Material for Non-Textile Industries for Month ending 28th February.

Articles.		Quantities. 1896.	1897.	Values. 1896.	1897.
				£	£
Bark, Peruvian	Cwt.	2,796	1,147	4,563	2,316
Bristles	Lb.	218,220	387,902	33,716	42,779
Caoutchouc	Cwt.	41,396	40,311	441,862	508,496
Gum:—					
Arabic	„	7,951	4,407	14,115	10,270
Lac, &c.	„	12,588	13,292	59,470	63,938
Gutta-percha	„	2,024	3,816	15,081	37,099
Hides, raw:—					
Dry	„	27,070	27,940	67,419	70,679
Wet	„	37,778	40,700	80,337	87,999
Ivory	„	872	577	39,504	24,902
Manure:—					
Guano	Tons	2,204	726	14,990	3,413
Bones	„	7,936	3,679	32,697	13,998
Nitrate of soda	„	19,447	9,561	153,049	79,263
Phosphate of lime	„	23,431	26,047	38,652	41,141
Paraffin	Cwt.	35,957	68,874	36,959	61,536
Linen rags	Tons	1,708	1,789	16,604	18,925
Esparto	„	26,249	19,001	110,937	83,850
Pulp of wood	„	25,326	24,629	130,049	121,831
Rosin	Cwt.	122,604	291,729	28,663	72,840
Tallow and stearin	„	174,150	78,129	204,386	79,196
Tar	Barrels	2,159	1,253	1,678	270
Wood:—					
Hewn	Loads	113,641	123,972	204,022	237,300
Sawn	„	61,073	76,451	150,711	213,562
Staves	„	6,123	5,907	40,634	30,339
Mahogany	Tons	3,835	6,441	33,144	59,531
Other articles	Value £	..	..	793,156	830,678
Total value		..	..	2,746,428	2,795,651

Besides the above, drugs to the value of 72,130*l*. were imported, as against 68,797*l*. in February 1896.

Exports of Metals (other than Machinery) for Month ending 28th February.

Articles.		Quantities. 1896.	1897.	Values. 1896.	1897.
				£	£
Brass	Cwt.	11,030	8,508	41,660	33,660
Copper:—					
Unwrought	„	50,968	28,813	117,863	76,055
Wrought	„	38,116	17,943	100,645	57,768
Mixed metal	„	31,033	15,044	66,542	37,006
Hardware	Value £	..	..	156,149	148,276
Implements	„	..	..	104,672	103,988
Iron and steel	Tons	237,346	261,930	1,728,089	1,881,791
Lead	„	2,808	2,876	37,370	39,074
Plated wares	Value £	..	..	18,367	25,577
Telegraph wires	„	..	..	109,840	43,710
Tin	Cwt.	11,325	7,179	36,152	23,624
Zinc	„	18,840	8,941	11,738	7,402
Other articles	Value £	..	..	60,885	64,351
Total value		..	..	2,589,972	2,542,282

Exports of Drugs and Chemicals for Month ending 28th February.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Alkali............ Cwt.	340,031	329,668	90,066	83,516
Bleaching materials „	92,755	73,441	33,123	23,721
Chemical manures Tons	36,516	34,207	194,467	178,938
Medicines....... Value £	..	..	85,144	87,312
Other articles... „	..	..	313,987	372,393
Total value	..	..	717,387	745,880

Exports of Miscellaneous Articles for Month ending 28th February.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Gunpowder...... Lb.	553,300	696,600	12,210	16,951
Military stores.. Value £	..	..	188,660	78,974
Candles.......... Lb.	2,207,500	2,013,000	35,336	28,516
Caoutchouc ..... Value £	..	..	92,181	91,078
Cement.......... Tons	27,612	29,150	45,591	46,533
Products of coal Value £	..	..	174,211	116,062
Earthenware ... „	..	..	139,534	123,947
Stoneware ...... „	..	..	10,669	13,207
Glass:—				
Plate........... Sq. Ft.	150,120	118,703	8,205	6,881
Flint........... Cwt.	7,479	7,034	16,514	16,812
Bottles......... „	56,260	53,015	26,534	24,381
Other kinds.... „	17,844	13,899	13,404	10,600
Leather:—				
Unwrought .... „	9,807	13,899	99,662	111,622
Wrought ..... Value £	..	..	29,524	27,815
Seed oil.......... Tons	4,442	4,549	85,074	78,137
Floorcloth ..... Sq. Yds.	2,359,800	2,318,400	92,616	98,238
Painters' materials Val. £	..	..	135,337	119,576
Paper .... Cwt.	74,556	80,748	116,771	122,489
Rags............ Tons	4,479	2,681	23,836	15,135
Soap............ Cwt.	55,967	54,297	56,185	58,935
Total value	..	..	2,660,224	2,414,666

Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—PLANT, APPARATUS, and MACHINERY.

Applications.

3999. F. Eveleigh and P. H. Mellor. Improved fuel economiser or apparatus for heating the feed water of boilers by means of the waste products of combustion. Feb. 15.

4095. H. Howell. Improvements in valves more especially adapted for use in self-discharging saturators used in the manufacture of sulphate of ammonia from gas liquor or ammoniacal liquor. Feb. 15.

4221. A. W. T. Hülssner and P. Röhrig. Improvements in clearing or clarifying and filtering apparatus. Complete Specification. Feb. 16.

4249. J. Bryson. *See* Class III.

4348. C. Bourdon. Improvements in condensers. Filed Feb. 17. Date applied for July 18, 1896, being date of application in France.

4612. D. A. Blair. Improvements in and relating to "Coffey's" distilling apparatus and the like. Feb. 20.

4690. J. R. Wright. An improvement in means or apparatus for carbonating liquids. Feb. 22.

4744. R. Moodie. Improvements in apparatus for washing or leaching granular or powdered substances and separating the same from the liquids used. Feb. 22.

4762. R. Matchett. Improved apparatus for reducing the pressure of liquids and gases. Complete Specification. Feb. 22.

5045. F. Berdal and V. Ducoulombier. Apparatus for preventing the scaling or incrustation of boilers. Feb. 25.

5066. O. Herrmann. New or improved process and apparatus for heating, concentrating, evaporating, and drying gaseous liquid and solid substances. Feb. 25.

5588. W. L. Wise.—From F. J. Brischar. Improved closing-device for bottles and the like designed to contain gas or other fluid under high pressure. March 2.

5589. W. L. Wise.—From F. J. Brischar. Improved closing-device for bottles and the like designed to contain gas or other fluid under high pressure. March 2.

5823. Maignen's "Filtre-Rapide" and "Anti-Calcaire" Co., Ltd., and A. A. Ellis. Improvements in apparatus for the treatment of water or other liquids with powdered materials. March 4.

6083. M. Crawford. Improvements in apparatus for charging liquids with gas. March 8.

6086. J. Kent and W. Lingley. Improvements in minimum thermometers. March 8.

6087. J. Kent and W. Lingley. An improved electric circuit-closing thermometer. March 8.

6157. L. Maiche. Improvements in apparatus for heating and cooling fluids by fluids. March 9.

6415. T. Bennett. An improved milk-testing apparatus. March 11.

6423. H. McV. Miller. Improvements in stills or condensers. March 11.

6550. L. Baseque. Centrifugal galactometer and analysing process connected therewith. March 12.

Complete Specifications Accepted.*

1896.

4725. W. L. Wise.—From Solvay et Cie. Apparatus for filtering, washing, and drying, suitable for the treatment of bicarbonate of soda. March 3.

5849. F. H. Tyacke and C. E. Gittins. Water softening and purifying apparatus. March 10.

6934. F. Jurschina, Z. Schnitzer, and F. Hersch. An improved filtering material, and process of making same. March 10.

15,374. J. Pogson. Surface condensers. March 3.

1897.

746. C. Gautier and X. Wehrle. Carburetting apparatus. March 3.

853. T. B. Lightfoot.—From The Gesellschaft für Linde's Eismaschinen. Safety and regulating appliance for apparatus for compressing vapours of ammonia and other volatile liquids. March 3.

1138. H. H. Lake.—From D. H. Burrell and E. J. Burrell. Centrifugal liquid separators. Feb. 24.

II.—FUEL, GAS, and LIGHT.

Applications.

4125. J. R. Wigham. Improvements in apparatus for the production of artificial light applicable for lighthouse and ordinary illumination. Feb. 16.

4209. M. Horwitz. Improvements in the manufacture of incandescent bodies for incandescent lighting. Feb. 16.

4282. E. L. Carr. A new or improved portable acetylene gas generator. Feb. 17.

4302. C. E. Melville, J. Horne, and H. Roantree. An improved method of obtaining oxygen gas from atmospheric air. Feb. 17.

4424. E. Gossart and H. Chevallier. Improvements in or relating to means for producing and burning acetylene. Filed Feb. 18. Date applied for Sept. 10, 1896, being date of application in France.

4426. J. H. W. Stringfellow. Improvements relating to the manufacture of luminous and non-luminous gas. Feb. 18.

4428. K. Stattler and A. Strejz. An improved process for producing gas for burning and lighting purposes. Feb. 18.

4488. T. R. Fowler. Improvements in and connected with apparatus for the manufacture of acetylene gas. Feb. 19.

4583. W. P. Thompson.—From The Continental Gas Gluhlicht Actien Gesellschaft Meteor vormals Kroll Berger and Co. Improvements in or relating to incandescence burners. Feb. 20.

4613. A. T. Fletcher and J. S. Marsland. Improvements connected with inclined gas retorts. Feb. 20.

4734. E. Barillot and C. C. Leclaire. Improvements in or relating to the storage of acetylene gas. Feb. 22.

4761. P. A. M. Lacroix. An improved automatic apparatus for producing acetylene gas. Feb. 22.

4775. H. H. Lake.—From P. Octure and A. Desmazures. An improved burner applicable more especially for incandescent gas lighting. Feb. 22.

4781. A. J. Boult.—From E. Melia. Improvements in or relating to the production of acetylene gas. Feb. 22.

4790. F. H. Smith. An acetylene gas lamp. Feb. 23.

4791. C. W. Kemp. Improvements in or relating to incandescent gas lighting. Feb. 23.

4826. R. Cremer. Bottle for compressed gases. Feb. 23.

4870. E. J. Frost and B. Middleditch. Improvements in devices for automatically regulating the quality of gas. Complete Specification. Feb. 23.

4987. G. A. E. Rousset and A. Cobler. Improvements in apparatus for the production of "acetylene." Feb. 24.

5044. F. Missire and M. L. Missire. Improvements in burners for incandescent lighting. Feb. 25.

5051. A. W. Holden and F. W. Brown. Improvements in acetylene gas generators and ometers. Feb. 25.

5149. A. Roedel. Process and apparatus for removing graphite crusts from the interior of gas retorts. Complete Specification. Feb. 25.

5180. S. J. Farnsworth. Improvement in apparatus for condensing smoke and producing liquor carboline. Feb. 26.

5182. F. A. E. Samuelson. Improvements in the construction of coke ovens. Feb. 26.

5236. C. B. v. Franco. Improvements in means for generating, holding, and burning acetylene gas. Complete Specification. Filed Feb. 26. Date applied for July 27, 1896, being date of application in Spain.

5250. T. H. Gittins and A. Forshaw. Improvements in the manufacture of fuel. Feb. 26.

5297. H. Emmett. Improvements in incandescent gas and other burners. Feb. 27.

5415. G. Baldwin and C. Crastin. Improvements in the apparatus for the manufacture of acetylene gas. March 1.

5669. J. Kohlndorfer. A process for producing a solid or consistent fuel containing petroleum. Complete Specification. March 3.

5756. H. R. Bean and H. Ringwood. An improved combined automatic generator, condenser, and holder for acetylene and other gases. Complete Specification. March 4.

5786. H. Hansen. Process for the solidification of refined petroleum and mineral oils for heating and lighting purposes. March 4.

5817. Count de Hamel de Manin. Improvements in the manufacture of incandescence bodies for illuminating purposes. March 4.

5913. H. E. Alexandre. An improved apparatus for the manufacture of acetylene gas. Complete Specification. March 5.

6016. A. Mengers, H. Hurwitz, and A. Franke. Improvement in the treatment of incandescent bodies for gas lighting. March 6.

6033. J. W. Roberts and E. C. Theedam. The use of gunpowder, gun-cotton, or any explosive used in firearms, whether of a chemical or mechanical mixture, in stationary and portable engines, as a motive force. March 8.

6078. A. J. Boult.—From F. Janssens. Improvements in or relating to apparatus for the production of acetylene gas. March 8.

6132. W. Climie and A. Watson, jun. Improvements in gas producers. March 9.

6159. J. Moeller. Improvements in oil lamps for incandescence lighting. March 9.

6271. A. Guasco. An improved apparatus for producing acetylene gas. March 10.

6292. A. Stowell. Incandescent lamp. March 10.

6404. P. Muschamp and S. Burridge. Improvements in manufacture of coke, and in apparatus and appliances connected therewith. March 11.

6538. W. R. Biss. An improved apparatus for generating and storing acetylene gas. March 12.

6548. R. F. Faure. Improved apparatus for producing acetylene gas. March 12.

6658. W. W. Strode and G. H. White. Improvements in or relating to the manufacture of acetylene. March 13.

6659. W. W. Strode and G. H. White. Improvements in or relating to illuminating apparatus. March 13.

6673. J. Hamilton. An improved mantle for incandescent gas lamps. March 13.

Complete Specifications Accepted.

1896.

4980. W. Wheatley and G. Kros. Apparatus for the production of carburetted water-gas. March 10.

5291. A. Kitson. Apparatus for the manufacture of gas, partly applicable to other purposes. March 10.

5813. T. Holliday. Apparatus for use in the production and application of acetylene gas. March 3.

6254. W. Parlby. Apparatus for quenching or cooling coke produced in the manufacture of coal-gas. Feb. 24.

6380. Sir E. Green, Bart. Fuel economisers. March 17.

6408. A. Sweetser. Apparatus for the manufacture of oxygen gas. March 10.

6719. R. Reitmeyer.—From A. Cerckel. Apparatus for lighting and heating by means of acetylene gas. Feb. 24.

6739. J. C. Bayley. Apparatus for generating and storing acetylene gas, especially suitable for use in portable installations. March 3.

7286. C. Billington, jun. An improved method of and means for removing "scurf" or "retort carbon" from gas retorts. March 3.

7559. W. Hampson. Apparatus for separating mixed gases by refrigeration, especially applicable to the separation of oxygen from air. March 10.

9681. H. L. P. Boot. Street lighting by glow lamps. Feb. 17.

12,662. A. Tobler. Apparatus for the automatic generation and distribution of acetylene gas. March 3.

17,644. R. Goodwin. A gas plant and system to make and produce acetylene gas, or acetylene and carbonic acid gas combined. March 24.

23,591. G. Trouve. Apparatus for producing and storing acetylene gas. March 17.

26,516. G. Tresenreuter. Method and apparatus for producing gas from liquid combustible materials. Feb. 24.

29,613. A. Rommoser and A. von Hake. Incandescent lights. March 3.

29,966. J. Y. Johnson.—From The Electric Rectifying and Refining Co. Apparatus for producing ozone. March 3.

1897.

139. F. Trendel and J. Mucke. Process and apparatus for producing acetylene gas. March 10.

328. J. Zimmermann. Processes and apparatus for forming and burning acetylene and similar gases. Feb. 24.

1653. G. de R. de Sales. Process for the agglomeration of carbides. March 17.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Applications.

4249. J. Bryson. Improvements in or relating to retorts for the distillation of shale and other bituminous substances, or for the calcining of ironstone, lime, and other substances. Complete Specification. Feb. 17.

5698. A. Basch and H. Graepel. An improved process for carbonising, charring, or coking combustible substances. Complete Specification. March 3.

IV.—COLOURING MATTERS and DYES.

Applications.

4086. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of paranitraniline red having very bluish tints fast to washing and light. Feb. 15.

4369. W. H. Claus, A. Ree, and L. Marchlewski. Production of new polyazo dyes. Feb. 18.

4446. B. E. R. Newlands and J. A. R. Newlands. Improvements in the preparation of a colouring matter for and its application in the manufacture of sugar. Feb. 18.

4657. J. Turner and Co., Ltd., and P. Schidrowitz and O. Rosenheim. Manufacture of new products from piperidine. Feb. 20.

4703. W. G. Heyes. — From The Chemical Works, Sandoz. The manufacture or production of substantive disazo colouring matters. Feb. 22.

5119. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. A method for dyeing half-wool with basic azo dyestuffs in an acid bath. Feb. 25.

5259. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. A process for dyeing fast tannin antimony lakes of polyazo dyestuffs and basic safranine azo dyestuffs on the vegetable fibre. Feb. 26.

5351. A. G. Green, A. R. Wahl, and The Clayton Aniline Co., Ltd. The manufacture of derivatives of diphenylethane and of diphenylethylene. Feb. 27.

5535. H. R. Vidal. New carboxylated products derived from amido or phenolic compounds and amides and phenolics of triphenylmethane. March 2.

5689. H. R. Vidal. Improvements in the manufacture of colouring matters. March 3.

5690. H. R. Vidal. Improvements in the manufacture of colouring matters. March 3.

5691. H. R. Vidal. Improvements in the preparation of paradioxythiazine and paramidoxythiazine. March 3.

5697. H. R. Vidal. Improvements in the preparation of paramidophenol and the like. March 3.

5924. I. Levinstein and Levinstein, Ltd. Production of new sulphonic acids which may be used in the manufacture of colouring matters. March 6.

6581. W. Majert. Method of producing and purifying orthotoluol-sulpho-chloride. Complete Specification. March 12.

6694. H. R. Vidal. Improvements in manufacture of colouring matters. March 13. Date applied for March 10, 1897, being date of application in France.

Complete Specifications Accepted.

1896.

7538. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of anthracene compounds and of dyestuffs derived therefrom. March 3.

7709. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of new derivatives of amido-1, alphyl-2, alkyl-5 pyrazolones. Feb. 24.

7766. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. The manufacture and production of new colouring matter from dinitronaphthalene. March 17.

8582. H. H. Lake.—From Lepetit, Gansser, and Co. Manufacture of dyestuffs and tanning extracts. March 17.

8857. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation. Manufacture of colouring matters of the rosinduline series. March 17.

15,294. W. E. Heys. — From The Chemische Fabrik vormals Sandoz. The manufacture or production of polyazo colouring matters. March 3.

16,039. J. J. M. Ville. New red colouring matters—rodazines—of the triphenylmethane group, and method of producing the same. March 17.

V.—TEXTILES, COTTON, WOOL, SILK, Etc.

Applications.

4165. R. J. Eke. Improved machinery for the treatment of textile vegetable fibres. Feb. 16.

5350. M. Beck. Improvements in connection with apparatus for finishing, stretching, mordanting, and drying textile fabrics. Feb. 27.

5414. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Improved method of mercerising vegetable textures and yarns. March 1.

6038. J. Morton. Improvements in the manufacture of figured fabrics. March 8.

Complete Specifications Accepted.

1896.

877. J. J. Mann. Manufacture of impermeable fabrics. Feb. 24.

6519. J. Ingleby and E. Ostlere. Manufacture of linoleum, cork carpets, and the like fabrics, and apparatus therefor. March 17.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

Applications.

4540. A. Sokoloff. Improved process for dyeing by means of electricity and the X-rays. Complete Specification. Feb. 19.

4953. A. Schmidt. Dyeing apparatus for carded wool. Feb. 23.

5122. S. Pitt.—From L. Cassella and Co. Producing fast dyeings upon the fibre by means of direct-dyeing colouring matters. Feb. 25.

5573. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. New process for dyeing cotton. March 2.

6112. W. E. Kay and The Thornliebank Co., Ltd. Improvements relating to the fixation of pigments or metallic powders in printing on textile fabrics or similar surfaces. March 9.

6498. J. Waugh.—From The Neue Augsburger Kattunfabrik. Improvements in the method of washing and boiling off, and in the preparation of fabrics to be dyed or printed. March 12.

Complete Specifications Accepted.

1896.

8491. B. Thies. Process of and apparatus for bleaching, dyeing, washing, &c., spun fibres and stuffs of every kind. March 10.

22,823. J. Kent. Bleaching cotton and other vegetable textile materials. March 3.

VII.—ACIDS, ALKALIS, and SALTS.

Applications.

4179. A. J. Boult.—From S. Ganelin and J. Block. Improvements in the method of making chloride of lead. Complete Specification. Feb. 16.

4227. T. Ryan, jun., A. Dyson, and The United Alkali Co., Ltd. Improvements in the manufacture of caustic soda or caustic potash from carbonate of soda or carbonate of potash. Feb. 16.

4675. F. Bosca. Improvements in the manufacture of calcium carbide. Feb. 20.

4721. H. Walker. Improvements in the method of and means for manufacturing calcium carbide. Feb. 22.

4747. A. Knop. Manufacture of nitrites. Feb. 22.

5483. R. N. Lennox. Process for the separation and purification of nitrate of ammonia. March 2.

5564 T. Jennings. An improved process for the production of magnesium hydrate. March 2.

6082. M. Crawford. Improvements in the production of carbonic acid and in apparatus to be employed therein. March 8.

6648. T. Keene. Improvements in furnaces for the manufacture of calcium carbide or the like. March 13.

Complete Specifications Accepted.

1896.

4189. G. Craig. Improvements in obtaining alkaline salts, and apparatus therefor. March 3.

4725. W. L. Wise.—From Solvay et Cie. *See* Class I.

7464. A. MacNab. Treatment of bay salt. March 10.

7796. W. Shedlock. Apparatus for obtaining salt from brine and for similar purposes. March 17.

15,489. F. H. Haviland, A. Holoway, J. B. Collier, and W. H. Murch. Method and apparatus for the production of calcium carbide. Feb. 24.

28,199. H. H. Lake.—From C. Pieper. Process of treating substances containing nitrogen for the manufacture of ammonia. March 10.

VIII.—GLASS, POTTERY, and ENAMELS.

Applications.

4085. H. G. Harris. An improvement in the manufacture of plate glass and apparatus for that purpose. Feb. 15.

4141. T. W. Horn. Improvements in window glass. Complete Specification. Feb. 16.

4304. E. Masquelier Improvements in the manufacture of glass face plates. Feb. 17.

4527. C. Paulus and H. Heim. Improvements in the manufacture of ceramic ware and in machinery or apparatus for the same. Complete Specification. Feb. 19.

4539. C. C. Schirm and O. Lessing. Improved process for producing ceramic masses. Complete Specification. Feb. 19.

4885. T. Detal-Thiran. Improvements in and relating to the manufacture of mosaic tiles. Feb. 23.

6104. H. Heckert. Improvements in glass hollow-ware. Complete Specification. March 8.

6184. E. Walsh, jun. Improvements in plate-glass annealing kilns. Complete Specification. March 9.

6347. A. H. Moore and J. W. Moore. Equable firing kilns. March 11.

Complete Specification Accepted.

1896.

4093. G. H. Grundy and G. A. Liogard. Method or means of decorating tiles, plates, and other articles of ceramic ware. Feb. 24.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, and CEMENTS.

Applications.

4048 W. S. Robinson and S. M. Brookfield. Improvements in cement and concrete. Complete Specification. Feb. 15.

5290. T. Parker.—From G. W. Parker. An improved means of crystallising gypsum for commercial use. Feb. 27.

5439. W. S. Wilkinson. Improvements in the manufacture of paving blocks, tiles, and the like. Complete Specification. March 1.

6413. D. Burness and J. Burness. Improvements in concrete paving and the like. March 11.

6459. W. C. Reid and F. Crosland. Improvements in lime or cement kilns. March 11.

6525. G. Christie. Improvements in the method of and means for making artificial stone. March 12.

Complete Specifications Accepted.

1896.

3597. A. Hannemann and G. Boisly. Process of manufacture of artificial asphalt. Feb. 24.

7567. W. C. Lawton. Process of manufacturing ornamental strips of soft clay. March 17.

18,990. A. J. Boult.—From Tortorici and Grasso. Process and apparatus for the treatment of articles of cement, plaster, chalk, and the like. March 17.

19,047. A. L. C. Nodon and L. A. Bretonneau. Process and means for drying or senilising wood. March 10.

X.—METALLURGY, MINING, Etc.

Applications.

4136. W. B. Johnson.—From The Pennsylvania Salt Manufacturing Co. Improvements in apparatus for casting metals. Complete Specification. Feb. 16.

4202. A. F. M. V. Baron. Manufacture of an alloy of copper and iron. Complete Specification. Feb. 16.

4218. E. Dodé. Process of separating metals from ores. Feb. 16.

4309. S. Neffgen. Process of treating granular or pulverulent iron ores and slag, or other residues containing iron, to facilitate the extraction of the same. Complete Specification. Feb. 17.

4358. A. Phillips. A plunger for the purifying or mixing of molten metals or other bodies liquid at high temperatures. Feb. 18.

4377. T. Twynam. Improvements in the manufacture of iron and steel. Feb. 18.

4655. W. H. Hyatt. Improvements in the extraction of gold. Feb. 20.

4857. W. A. Koneman and W. H. Hartley. Improvements in or relating to the treatment of ores. Complete Specification. Feb. 23.

5011. P. Dronier. Improvements in apparatus for the manufacture of magnesium. Feb. 24.

5340. The Lu-mi-num Manufacturing Co., Ltd., and J. G. Accles. Improvements in and connected with the casting of metals such as aluminium and alloys of the same. Feb. 27.

5670. B. H. Thwaite and H. V. Holden. An improvement in the manufacture of armour plates. March 3.

5732. T. Millward. An improved plant for planishing, flattening, blueing, and tempering iron and steel, sheets, plates, and the like. March 4.

5796. T. Korovine, N. Klatschko, and J. Berthenson. Improvements in treating iron and steel. March 4.

5880. W. Sawyer. Improvements in the manufacture of alloys of metal capable of receiving and retaining a high polish. March 5.

5926. J. de Moya. Improvements in the manufacture of steel. Filed March 6. Date applied for Aug. 10, 1896, being date of application in France. Complete Specification.

6051. J. E. Preston. Improved method and apparatus for treating refractory ores containing gold, silver, nickel, &c. March 8.

6151. F. A. Gooch. Improvements in processes for reducing aluminium. Complete Specification. March 9.

6489. J. Davies and A. Sanders. Improvements in fluid furnaces for heating or smelting or refining metals. March 12.

Complete Specifications Accepted.

1896.

4165. E. L. Oppermann. A new or improved process for amalgamating and extracting gold or other suitable metals from dry crushed ore. March 3.

4899. J. C. Bull. Improvements in alloys. March 3.

6033. W. Pearson and J. A. Bott. Manufacture of aluminium alloys. March 3.

7783. W. P. Thompson.—From E. Wohlwill. Process for obtaining chemically pure gold from fine gold and alloys rich in gold. March 3.

8595. T. Smith. Improvements in or connected with readily fusible metal or alloy and the manufacture of articles therefrom, or from other metals or alloys. March 3.

12,106. E. Govett. An improved process for treating refractory ores. March 3.

14,330A. A. G. Brown. Manufacture of rods and tubes mainly from aluminium and aluminium alloys. March 17.

27,899. H. Imray.—From La Com. Anonyme des Forges de Chatillon et Commentry. Method of pouring steel ingots. Feb. 24.

1897.

94. J. D. Grey and T. Gwynne. Treatment of sheet iron and steel in the manufacture of polished sheets. March 17.

XI.—ELECTRO-CHEMISTRY and ELECTRO-METALLURGY.

Applications.

4046. E. Zappert.—From T. Bergmann. Improvements in electric batteries. Feb. 15.

4353. J. B. Scammell. Improvements in electrical storage batteries or accumulators. Feb. 18.

4375. W. Rowbotham. Improvements in and relating to accumulators or storage batteries. Feb. 18.

4420. J. L. Dobell. Improvements in or connected with electric batteries. Feb. 18.

4434. A. B. Pescatore. Improvements in or relating to secondary batteries. Feb. 18.

4442. J. L. Dobell. Improvements in or connected with electric batteries. Feb. 18.

4916. E. Heyl. Improvements in electric accumulators. Feb. 23.

5102. W. S. Squire. Improvements in the construction of secondary batteries. Feb. 25.

5166. D. Neale. Improvements in primary and secondary electric batteries. Feb. 26.

5383. S. F. Walker. Improvements in primary galvanic batteries. March 1.

5687. M. M. Bair. Improvements in electric batteries. March 3.

5805. D. G. FitzGerald. Improvements in and in the manufacture of elements for secondary batteries. March 4.

5826. A. Schanschieff. Improvements in the active material of secondary batteries. March 5.

6188. J. Hargreaves. Improved means applicable for use in the electrolysis of salts. March 9.

Complete Specifications Accepted.

1896.

1905. H. Maxim. Improvements in furnace blasts, and in process and in means or apparatus for electro-thermally treating materials, for the reduction of metals, production of carbides, acetylene, cyanogen, cyanides, nitrides, and the fixation of nitrogen. March 10.

5263. H. A. V. Worth. Improved process for the electroplating or metallisation of wood. March 10.

5584. A. Cohen. Treatment of carbon in electro-chemical or electrolytic processes, and apparatus therefor. March 17.

7255. H. H. Lake.—From J. Julien. Secondary batteries. March 10.

8410. J. C. Graham. Electro-deposition of metals, particularly applicable to the deposition on wires. March 10.

10,095. P. Schmidt. Electric batteries. March 3.

13,690. A. Le Royer, A. E. Bouna, and P. van Berchem. Electrolytic apparatus. March 3.

20,035. D. Mathieu. Electrical batteries. March 17.

23,868. L. Honig. Insulating compound. March 3.

1897.

251. G. W. Harris and R. J. Holland. Secondary batteries, and process of forming same. March 10.

1535. A. James. Electric precipitation of gold and silver from their cyanide solutions. March 10.

1657. G. A. Webb and W. A. Thoms. Electro-deposition of metals and alloys. March 17.

XII.—FATS, OILS, and SOAP.

Applications.

4830. F. L. Bartelt. Improved washing and cleansing compound. Feb. 23.

5256. H. H. Lake.—From F. W. Klever. A method of dissolving iodine in mineral oils. Feb. 26.

5750. R. E. Green. Improvements in the production of glycerin. March 4.

5820. A. F. Dunn and A. Waithman. Manufacture of an antiseptic soap powder. March 4.

5889. E. G. Scott. Improvements connected with the concentration or distillation of glycerin. March 5.

6564. P. Cullen. The manufacture of an improved cleansing composition. March 12.

Complete Specifications Accepted.

1896.

5791. P. R. de F. d'Humy. Production of lubricants. March 17.

5792. P. R. de F. d'Humy. Manufacture of soap. March 17.

7269. The Fish Utilisation Syndicate, Ltd., and J. C. W. Stanley. Liberation or recovery of oils and the like. March 17.

1897.

123. G. Rezek. An improved washing material, and process for making the same. March 10.

1208. W. P. Thompson.—From J. Van de Bucken and S. Gattfeld. Soap for carpets. March 17.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

4947. W. Peel. A new paint or composition for ships' bottoms and submerged works. Feb. 24.

5121. J. G. Ingram. Improvements in the manufacture of parti-coloured india-rubber. Feb. 25.

5289. C. R. Lindsey. The manufacture of a pigment from aniline waste products. Feb. 26.

5577. W. W. Horsburgh and T. Macintosh. A new or improved composition of india-rubber or the like with other material. March 2.

5909. W. Negus.—From J. B. Gualco. Improvements in the manufacture of zinc white or oxide of zinc. March 5.

6222. E. B. Day. Improvements in india-rubber for pneumatic tyres and other purposes. March 9.

6324. T. C. Sanderson. Improvements in and relating to the manufacture of white lead. March 10.

Complete Specifications Accepted.

1896.

6830. W. J. Clapp and F. S. Dauncey. Manufacture of india-rubber, and machinery and apparatus therefor. March 3.

7785. J. Sanders. A new or improved composition applicable for covering or protecting ships' bottoms and other structures. March 3.

1897.

28,950. F. Beindorff. A process for making colours for water-colour painting. March 17.

XIV.—TANNING, LEATHER, GLUE, and SIZE.

Applications.

4439. C. H. O. Collins and J. Hoar. Improvements relating to the manufacture of artificial leather. Feb. 18.

5762. A. J. Boult.—From G. L. Lippold. An improved substitute for leather or like material and the method of manufacturing the same. Complete Specification. March 4.

6217. P. G. Sanford. Improvements in the treatment of tanning liquids for clarifying, bleaching, or decolorising them. March 9.

Complete Specifications Accepted.

1896.

8582. H. H. Lake.—From Lepetit, Dollfus, and Gansser and Co. See Class IV.

14,293. G. W. Adler. An improved process of and compound for tanning hides and skins. March 3.

1897.

2746. A. J. Boult.—From C. M. Higgins. Improvements in or relating to adhesive compounds. March 17.

XV.—AGRICULTURE and MANURES, Etc.

Applications

4542. C. Tejero. Process and apparatus for the fabrication of dissolved leather, its enrichment and transformation into an azotised product for manure. Feb. 19.

5974. H. E. Newton.—From A. Caron. New process for increasing the growth of cereals and their yield by means of bacteria, and a method for developing and conserving the latter on a large scale. March 2.

Complete Specification Accepted.

1896.

10,109. O. G. Blunden. New or improved artificial manure or fertiliser, and process for manufacturing the same. March 17.

XVI.—SUGARS, STARCHES, GUMS, Etc

Applications.

4446. B. E. R. Newlands and J. A. R. Newlands. See Class IV.

4467. E. R. Savigny. A new or improved sweetening substance and a process for its manufacture. Feb. 19.

5242. E. D. Smythe. Improvements in apparatus for reclaiming crystallised syrups. Feb. 26.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

4089. L. Maiche. An improved process for the purification of spirit. Feb. 15.

4441. A. J. Boult.—From K. E. N. Fryklind. Improvements in the treatment of spirituous liquors and the products obtained therefrom. Feb. 18.

4612. D. A. Blair. See Class I.

4902. E. Guillaume. Improvements in distilling and rectifying processes and in apparatus therefor. Complete Specification. Feb. 23.

4926. C. Ameye. A machine for aërating wine, beer, and other fermented and unfermented liquids. Complete Specification. Feb. 23.

5582. L. Prochazka and E. Frischauer. Improvements in the process of and apparatus for mashing and brewing. Complete Specification. March 2.

5953. J. R. Swift. The beer preserver. March 6.

6082. M. Crawford. See Class VII.

6083. M. Crawford. See Class I.

6278. F. Billing and W. E. Partridge. Improvements in apparatus and process for extracting the properties from hops and other such like purposes. March 10.

6343. E. G. Major-Lucas. Improved apparatus for separating beer from yeast. March 10.

6463. F. W. H. Hempel. Process for producing luminous spirit. Complete Specification. March 11.

6605. D. B. Bentley. Improvements in the method of and means employed for maturing wines and spirits. March 13.

Complete Specifications Accepted.

1896.

6147. F. B. Pike. Method of treating brewers' and distillers' grains. March 17.

1897.

1162. F. C. Haeck. A new or improved process and apparatus for rapidly and economically ageing or maturing alcoholic beverages. March 10.

XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

Applications.

A.—Foods.

4147. J. Imray.—From La Soc. Anonyme des Matières Colorantes et Produits Chimiques de St. Denis and D. A. Rosenstiehl. A process for preserving alimentary substances. Feb. 18.

4695. J. Hogarth. Improvements in and relating to the treatment of alimentary substances. Feb. 22.

4730. B. L. Ryder. Improvements in method and apparatus for the preservative desiccation of vegetable and animal substances. Feb. 22.

5072. S. Griffin. Improvements in apparatus for conserving animal and vegetable food and other substances. Feb. 25.

6064. H. Humbser. Improved process for preserving substances. Complete Specification. March 8.

6065. H. Humbser. Improved process for drying substances. Complete Specification. March 8.

6346. W. Dodds. Improvement in preserving meat. March 11.

B.—Sanitation.

4040. F. P. Candy. Improvements in the purification of sewage and polluted water and in apparatus therefor. Feb. 15.

4406. T. Cosham. Improvements in sewage precipitating tanks. Feb. 18.

5005 W. J. Dibdin. Improvements in the treatment of sewage and appliances for that purpose. Feb. 24.

5007. L. Maiche. Improvements in apparatus for sterilising water. Feb. 24.

5793. P. Altmann. An improved process for rendering drinking water free from germs. Complete Specification. March 4.

5803. W. M. Ducat. Improvements in or connected with the purification of water, sewage, and other foul liquids. March 4.

6316. A. Apps. Improved method of sterilising, purifying, and deodorising receptacles for beverages and other materials. March 10.

C.—*Disinfectants.*

4145. R. Stewart. Improvements in the manufacture of detergent fluids. Feb. 16.

5820. A. F. Dunn and A. Waithman. *See* Class XII.

5853. J. H. Peace. An improved antiseptic fluid for destroying microbes and other bacteria. March 5.

Complete Specifications Accepted.

A.—*Foods.*

1896.

7480. A. J. Boult.—From F. Fritsche and Co. Preservation of organic substances. March 17.

26,506. H. Pape and W. Henneberg. Method and apparatus for producing fresh water from water containing salts. Feb. 24.

B.—*Sanitation.*

1896.

6132. H. B. Sharp. Manufacture of materials or compounds for use in the treatment of sewage and impure waters. March 17.

7268. The Fish Utilisation Syndicate, Ltd., and J. C. W. Stanley. Treatment of fish and other offal or similar refuse. March 10.

XIX.—PAPER, PASTEBOARD, Etc.

Applications.

4084. E. J. Pape. An improvement in blotting paper. Feb. 15.

4099. A. A. Hunting and E. A. Leigh. An improved method of and apparatus for drying paper and similar materials. Complete Specification. Feb. 16.

6054. J. S. Rigby, H. Sweetapple, and J. Clarke. Improvements in or connected with apparatus or machinery for the manufacture of vegetable parchment paper. March 8.

Complete Specifications Accepted.

1896.

4497. O. Tietze. Process for increasing the suppleness or pliability of paper. March 10.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

Applications.

4073. A. Mosticker. Improvements in the manufacture of vanillin. Feb. 15.

6220. J. C. Fell.—From W. J. Matheson and Co., Ltd. Process of producing acetanilid or acetoluids. Complete Specification. March 9.

6475. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Improvements in the manufacture and production of acetylanthranilic acid. March 11.

6653. H. Oppermann and R. Goehde. New formic aldehyde compounds and processes of preparing the same. March 13.

6686. E. H. C. Durkopf. Improvements in the manufacture of methylenediltannic acids from formaldehyde and tannin. March 13.

Complete Specifications Accepted.

1896.

6858. C. D. Abel.—From E. Bonnert and T. Schlumberger. Manufacture of solution of tetranitro-cellulose (collodion pyroxyline). March 3.

1897.

28,968. L. Lederer. New or improved process for manufacturing oxyphenoxacetic acids. Feb. 24.

XXI.—PHOTOGRAPHY.

Applications.

5026. A. Rateau. Improvements in chromo-photographic apparatus. Complete Specification. Feb. 24.

6593. W. B. Ferguson. A new process of toning photographic prints and transparencies. March 13.

Complete Specifications Accepted.

1896.

15,852. E. P. Schoenfelder and E. Kehlf. Chemical compositions for preparing the surface of suitable material for photographic prints. Feb. 24.

25,980. J. Liddle.—From The International Photographic and Supply Co. Process of printing photographs on textile fabrics, and solutions connected therewith. March 3.

XXII.—EXPLOSIVES, MATCHES, Etc.

Applications.

4062. S. A. Rosenthal.—From J. S. von Romocki. Improvements in the manufacture of safety explosives. Feb. 15.

4279. P. S. D. Dunn. Improvements in or connected with matches. Feb. 17.

4566. R. Crooke, jun. Improvements in the manufacture of explosives especially applicable for use in coal mines. Feb. 19.

4672. L. Roux. Improvements in the preparation of explosives. Feb. 20.

5003. H. L. Robinson and E. J. Ryves. An improved smoke-producing mixture for use with explosives. Feb. 24.

5399. G. de Wolf. From B. C. Pettingell. An improved blasting and gun powder. March 1.

6140. R. N. Lennox. Process for the separation and purification of nitrate of ammonia. March 9.

6409. H. Maxim. Improvements in explosive propelling compounds and methods of manufacture. March 11.

6438. W. F. Brown. Improvements relating to matches. March 11.

6461. M. van Look. Improvements in or relating to explosives. Complete Specification. March 11.

6610. C. O. Lundholm and J. Sayers. Improvements in the manufacture of explosives. March 13.

Complete Specification Accepted.

1896.

6431. A. V. Newton.—From A. Nobel. Improvements in explosive compounds. March 3.

Printed and Published by Eyre and Spottiswoode, East Harding Street, London, E.C., for the Society of Chemical Industry.

THE JOURNAL

OF THE

Society of Chemical Industry.

A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

No. 4.—Vol. XVI.] **APRIL 30, 1897.** [Non-Members 30/- per annum; Members 21/- per Set of extra or back numbers; Single Copies (Members only) 2/6.

NOTICES.

Annual General Meeting.

The Annual General Meeting will be held in Manchester on the 14th, 15th, and 16th July next. Tickets of Membership will be issued in time for the Meeting, and will form, as heretofore, vouchers for visits to works and excursions. A draft programme will be issued with the May number.

In accordance with the provisions of Rule 18 of the Bye-laws, notice is hereby given that those Members whose names are placed in italics in the list of Council will retire from their respective offices at the forthcoming Annual General Meeting.

Prof. F. Clowes, D.Sc., has been nominated to the office of President; Dr. Edward Schunck, F.R.S., has been nominated Vice-President under Rule 11; Mr. A. Gordon Salamon and Dr. Wm. Jay Schieffelin have been nominated Vice-Presidents under Rule 24; and Mr. D. Howard, Mr.

Ivan Levinstein, and Mr. Wm. Thorp have been nominated Vice-Presidents under Rule 8.

The Hon. Treasurer and Hon. Foreign Secretary have been nominated for re-election to their respective offices.

Members are hereby invited to nominate fit and proper persons to fill four vacancies among the Ordinary Members of Council under Rule 18. Nomination forms for this purpose can be obtained from the General Secretary upon application.

Extract from Rule 18:—"No such nomination shall be valid unless it be signed by at least ten Members of the Society who are not in arrear with their subscriptions, nor unless it be received by the General Secretary, at the Society's Office, at least one month before the date of the Annual General Meeting, to the election to take place at which it refers. Nor shall any such nomination be valid if the person nominated be ineligible for election under Rules 12 or 15. No member shall sign more than one nomination form."

Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, part of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows:—

To Members (see Rules 25 and 27) and Past Members (see Rule 30); Libraries, Corporations, and Exchanges on the Society's List — Each copy 10*s.*

To Subscribers .... " 12*s.* 6*d.*

To others........................ " 15*s.*

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. Eyre and Spottiswoode, the Society's printers and publishers, to whom all communications respecting them should be addressed. The circulation of the Journal is now more than 3,000 per month.

LIST OF MEMBERS ELECTED 23rd APRIL 1897.

Aspinwall, H. C., Pempton Lakes, Passaic Co., N.J., U.S.A., Explosives Manufacturer.

Berry, Albert E., c/o Boake, Roberts, and Co., Chemical Works, Stratford, E., Works Manager.

Brown, W. D., Know Mills Printworks, Entwisle, Lancashire, Colourist.

Canfield, F. D., jr., c/o National Sugar Refining Co., Yonkers, N.Y., U.S.A., Sugar Refiner.

Carnell, Wm. C., 3232, Wabash Avenue, Chicago, Ill., U.S.A., Chemist.

Clarke, Wm. B., Edison-Swan Electric Works, Ponders End, N., Electro-Chemist.

Cooper, Wm. R., Carisbrooke, Upper Tulse Hill, S.W., Electrical Engineer.

Farmer, John E., Salford Sewage Works, Weaste, Salford, Assistant Manager.

Gamble, Chas. W., Photo-Process School, 6, Bolt Court, E.C., Director.

Harris, W. D., Lanebeg, Lisburn, Co. Antrim, Ireland, Bleach and Dyeworks Manager.

Hill, George, Barton-on-Humber, Chemical Works Manager.

Israel, Louis A., c/o T. Wavell, California Street, San Francisco, Cal., U.S.A., Manufacturing Chemist.

Jansen, Dr. Robt., 43, Plymouth Avenue, Longsight, Manchester, Technical Chemist.

Jones, Chas. H., Rua Figueroa 7, Minas de Rio Tinto, South Spain, Chemist and Assayer.

Jorissen, Dr. Wm. P., 168, Oostzee dijk, Rotterdam, Holland, Editor.

Jowett, Wm. Hall, Ardencraig, Grassendale, near Liverpool, Chemist.

Lloyd, G. Hamilton, 7, St. Mildred's Court, London, E.C., Civil and Mining Engineer.

Low, Albert H., P.O. Drawer 1537, Denver, Colo., U.S.A., Metallurgical Chemist.

Miller, Jas., 2, Ailsa Terrace, Hillhead, Glasgow, Metallurgical Chemist.

Pickert, Leo W., American Sugar Refining Co., Granite Street, South Boston, Mass., U.S.A., Chemist.

Power, Dr. Fred. B., Wellcome Research Laboratories, 42, Snow Hill, E.C., Director.

Prentice, Dr. Bertram, Royal Technical Institute, Salford, Lecturer on Chemistry.

Raper, Thos. H., 18, Victoria Embankment, Darlington, Analytical Chemist.

Smith, Allan, 100, Bury Road, Edenfield, near Bury, Lancashire, Paper Mills Chemist.

Verity, Victor, 83, Lexington Street, East Boston, Mass., U.S.A., Chemical Works Foreman.

Wahl, André R., 331, Ashton New Road, Clayton, Manchester, Technical Chemist.

Walker, Henry V., 21, Cliff Street, New York City, U.S.A., Chemist.

Yates, Wm. H., 327, Bolton Road, Stoneclough, near Manchester, Lecturer on Chemistry.

CHANGES OF ADDRESS.

Barden, Sam, l/o Far Bank Farm; Woodhouse Mills, Shelley, near Huddersfield.

Beanes, Edw.; Journals to Moatlands, Paddock Wood, Kent.

Brown, Jas., l/o Bloomfield; 30, Castlereagh Avenue, Mountpottinger, Belfast.

Bumby, H., l/o Askam; Coltness Ironworks, Newmains, N.B.

Burn-Murdoch, J. V., l/o Pembroke; 23, Cadogan Gardens, S.W.

Clennell, J. E., l/o New York; 501, Dooly Building, Salt Lake City, U.S.A.

Dickenson, F. M.; Journals to c/o Broken Hill Proprietary Co., 31, Queen Street, Melbourne, Victoria.

Ewen, E. D.; Journals to c/o West Indian Tobacco Co., Ltd., Port of Spain, Trinidad, B.W.I.

Fawcett, J. H., l/o Perth; c/o Mrs. Jamieson, 31, Queen Street, Melbourne, Victoria.

Green, J. Wilberforce, l/o Hayle; 36, Crystal Palace Park Road, Sydenham, S.E.

Haigh, De Lagnel; Journals to Avenue A and 13th Street, Bayonne, N.J., U.S.A.

Hamilton, Oswald, l/o Northfleet; Lancaster Cottage, Old Stratford, near Stony Stratford.

Handy, J. O.; Journals (temp.) to c/o Mrs. Maars, 10, Martensen's Allée, Copenhagen, Denmark.

Hawkins, J. D., l/o Gillett; c/o Colo. Phila. Reduction Co., Colorado City, Colo., U.S.A.

Hennin, A., l/o Monongahela Furnaces; National Tube Works, McKeesport, Pa., U.S.A.

Hotson, A. E., l/o Union Square; 147, East 125th Street, New York, U.S.A.

Hunt, B., l/o Stevenson Street; 216, Sansome Street, San Francisco, Cal., U.S.A.

Littell, R. Ballantine; Journals to Setauket, Long Island, N.Y., U.S.A.

Lymn, A. H., l/o Nottingham; 11, Henry Street, Warrington.

Mabery, Prof. C. F.; Journals to Case School of Applied Science, Cleveland, O., U.S.A.

McEwen, A. F., l/o Rossland; Lillooet, Laurie, C.P.R.R., British Columbia.

Manoukian, Wahan, l/o Manchester; 9, Torrington Square, W.C.

Pethybridge, Walter, l/o Clarendon Road; 3, Rhodesia Road, Clapham Rise, S.W.

Sinclair, Dr. Wm., l/o Uphall; 18, Cluny Drive, Edinburgh.

Smith, Wm., l/o 10; 38, Corn Street, Bristol.

Swan, David, l/o Glasgow; Joppa Lodge, Innellan, N.B.

Waterhouse, Col. J., l/o Calcutta; retain Journals until further notice.

Watson, Jno., l/o Johannesburg; c/o Lancaster G.M. Co., P.O. Box 144, Krugersdorp, S.A.R.

Welch, J. Cuthbert, l/o Reading; Kaslo, British Columbia.

White, W. Gilchrist; Journals to The Hodge, Broadbottom, near Manchester.

Wright, Lewis T., l/o London; The Mountain Copper Co., Keswick, Cal., U.S.A.

CHANGE OF ADDRESS REQUIRED.

Currie, Stanley C. C., l/o 45, Broadway, New York, U.S.A.

Deaths.

Ascough, Jesse, The Grange, Handsworth. April 11.

Mead, Frank, Gasworks, Sutton, Surrey.

London Section.

THE CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE, W.

Chairman: R. Messel.

Vice-Chairman: Boverton Redwood.

Committee:

B. Blount.	H. de Mosenthal.
C. F. Cross.	B. E. R. Newlands.
W. J. Dibdin.	*S. Rideal.*
R. J. Friswell.	*F. G. Adair Roberts.*
S. Hall.	F. Napier Sutton.
D. Howard.	Wm. Thorp.
E. Grant Hooper.	T. Tyrer.

Hon. Local Secretary:

John Heron, 110, Fenchurch Street, E.C.

The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Committee:* E. J. Bevan, Otto Hehner, J. B. Knight, A. R. Ling, and Lewis T. Wright.

SESSION 1896–97.

May 3rd, 1897:—

Dr. W. S. Squire. "On a New Slow-Combustion Arc Lamp suitable for Factories and Street Lighting."

Dr. J. Lewkowitsch. "On the Conversion of Oleic Acid into Candle Material."

Meeting held on Monday, April 5th, 1897.

DR. R. MESSEL IN THE CHAIR.

THE CHEMICAL STABILITY OF NITRO-COMPOUND EXPLOSIVES.

BY OSCAR GUTTMANN, ASSOC. M. INST. C.E., F.I.C.

I do not think it is necessary for me to explain the meaning of the term "nitro-compound explosives," nor their chemical composition, as this is now well understood by technical men, and has, moreover, been fully set forth by me in the treatise on the Manufacture of Explosives which I published in 1895. I can therefore at once proceed to the subject of my paper, namely, the "chemical stability" of such explosives.

Chemically speaking, they may be described as somewhat loosely bound. If exposed to a heat of about 180° C., they will almost without exception explode, but far below this temperature there is a splitting up of the molecules going on, which results in the development of nitric peroxide, and this in its turn will, if once formed, rapidly promote decomposition. In speaking therefore of the chemical stability of nitro-compound explosives, we have chiefly to deal with the question of the circumstances under which these explosives are liable to be decomposed with the development of nitric peroxide.

At present the chief nitro-compound explosives are nitrocellulose—be it made of cotton, wood, or other cellulose material—and nitroglycerin, or mixtures of these two; and also nitrobenzene, nitrotoluene, picric acid, picrates, &c. All these are manufactured nowadays in such a perfect way that the only possible cause of decomposition is the action of heat. But in some cases additions made to these explosives, either for the purpose of neutralising any free acid, or for the purpose of moderating or increasing the explosive effect, and sometimes also the mechanical treatment given may influence the stability.

In order to ascertain whether an explosive is liable to decomposition at temperatures that may occur under ordinary circumstances of storage and use, various means have been proposed, all of which should show the presence of nitric peroxide. In Austria it is still compulsory by law to keep a piece of litmus paper in each box of dynamite, so that on opening it the storekeeper can see whether the dynamite has become acid or not. Colonel Hess, of the Austrian Military Committee, heated the explosive, and carried the vapours into a solution of potassium iodide. In some other countries the explosive is subjected to a temperature varying between 100° and 135° C. for periods ranging from a day to a week, and the absence of red fumes is taken as an indication of the stability of the explosive. In this country the Home Office has, I believe upon the proposition of Dr. Dupré, prescribed a test which is familiar to everybody who has had to deal with explosives, since it has been adopted in almost every country, and has undoubtedly been the direct cause of great progress in the manufacture. It consists of heating the explosive in a test-tube at a given temperature (at present 170° F. for guncotton, and for most other explosives 180° F.) and noting the number of minutes which elapse before a brown line of a certain depth of tint appears on a piece of potassium iodide starch-paper that has been moistened with a solution of equal parts of glycerin and water, and which is suspended from a hooked glass rod passing through an india-rubber cork. In Germany zinc iodide starch-paper is used instead of potassium iodide paper. I shall deal with this test later on, and will now enumerate the various possibilities of decomposition, both during manufacture of the constituent nitro-compounds and the finished explosive.

Starting with guncotton, it was first recognised by Sir Frederick Abel that the various accidents with the original product of Schönbein and von Lenk were due to the imperfect removal of the acids that served to nitrate the cotton. Sir Frederick devised the pulping of guncotton in beating engines, whereby the fibres of the guncotton were thoroughly opened, and the washing water could easily penetrate. This process gives a guncotton of such high stability that there is now no difficulty of making it to comply with the Government heat test. Now and then, through imperfectly wringing out the acids in the centrifugal machine, a larger quantity of acids than usual remains in the guncotton, and as a definite number of washings are generally given, it may happen that, after the guncotton has gone through the whole process of boiling and washing, it is still sufficiently acid to give a bad heat test. Of course, this can be easily remedied with a little attention.

The practice of adding alkaline matters, such as carbonates of soda, lime, or magnesia, &c., is gradually dispensed with, and in this country the Government do not prescribe it. I believe I was the first to point out that the addition of alkaline matters to either guncotton or nitroglycerin is quite unnecessary, and in fact a deception. If there is a real decomposition going on in an explosive, the

small quantity of alkali will soon be consumed, whilst some of these alkalis have a direct tendency to decompose the nitro-compounds, thereby doing more harm than good. A properly purified explosive will, under ordinary circumstances of storage, keep practically for ever without showing the least sign of decomposition, and there is no better example than the bottle of nitroglycerin kept at the Nobel factory at Avigliana, which was the first nitroglycerin made by Prof. Sobrero in 1847, and which has been tested every year since, but has not yet shown any deterioration in stability. That the various alkaline substances added for neutralisation act differently has been shown by Dr. Dupré in the report of H.M. Inspectors of Explosives of 1887. He found that adding calcium or magnesium carbonate to gelatine dynamite does not appreciably affect the heat test, but the sodium carbonate has an uncertain effect, tending to increase the duration of the heat test in bad samples and to decrease it in good samples. This is quite consistent with what I said before, because in a bad sample the sodium carbonate would neutralise the acid already developed, whilst it would tend to decompose a good sample. In my own experience the addition of ammonium carbonate to blasting gelatine for the purpose of neutralising any acidity, has caused decomposition to such a degree that the guncotton partly disappeared, and the nitroglycerin was sweating out from the cases of blasting gelatine, so that the walls and the floor of the magazine were literally wet with it. The ammonia, which is so easily liberated from the carbonate of ammonia, acts very readily on nitro-compounds, and more readily on guncotton than on nitroglycerin.

In the case of picric acid and picrates, nitrobenzene, nitrotoluene, &c., similar remarks apply as to guncotton. When properly washed and freed from acid, they are stable, and the degree of stability will simply depend upon the complete removal of acids. In some of these nitro-compounds greater purity can be obtained by recrystallising, in others by repeated washing, if necessary with the addition of alkaline matters, so as to attack more readily the free acids.

Nitroglycerin, being an oily liquid, is not so easily purified, because it is more difficult to get it into small particles that can be attacked by the means employed for purification. Formerly very crude methods of washing, such as a petroleum barrel sunk into the ground to hold the nitroglycerin, and a churn-like perforated board to agitate the liquid, were used. Nowadays compressed air has been taken into requisition for thoroughly agitating the nitroglycerin with the washing liquids, and it serves at the same time to oxidise the lower nitro-compounds that generally accompany the impure product. With nitroglycerin it may also happen that a larger amount of acid is drawn off to the nitroglycerin in separating the latter, and then more frequent washing will have to be resorted to. In order to neutralise the bulk of the acid, sodium carbonate or soda in powder is added to the washing water, and the remaining acid is taken out by repeatedly washing with either hot or cold water or very weak soda solution. Some years ago a case came under my notice, where for days it was found impossible to make any nitroglycerin charge pass the heat test, although most careful and frequent washing was resorted to. Suspicion rested upon the glycerin and the sulphuric acid. The purity of the nitric acid, which had been made by my process, was above suspicion. However, careful examination with absolutely pure glycerin and sulphuric acid on the same large scale showed that the materials used were not at fault. The simple suggestion that I made from the beginning proved to be correct, namely, that the apparatus had not been sufficiently cleaned. As you know, nitroglycerin is made in leaden apparatus, and, although one would think that from the mixture of nitric and sulphuric acid only lead sulphate would be formed, yet the lead sulphate is of spongy, porous character, and will retain nitrous impurities mechanically. This is especially the case on the line of contact between the air and the surface. In testing small quantities of lead sulphate from different parts of an apparatus, and adding minute quantities to samples of nitroglycerin of undoubted excellence, the heat test at once went down in an alarming degree. The explanation now came as a matter of course: small particles of lead sulphate are always detached from the lead apparatus and suspended in the nitroglycerin; through the washing with compressed air they are thrown up and get little time to settle. The water alone does not remove the nitrous impurities, except in the course of long treatment, and therefore minute particles will find their way into the samples tested.

A similar possibility, although of an opposite character, was suggested by me and proved experimentally by washing nitroglycerin with soda. In adding soda in powder instead of in solution, soda mud is thrown down. If a small quantity of such mud is taken up, it will be distinctly alkaline to litmus paper, yet if a drop of nitroglycerin is put on the top of it, and allowed to stand for some time, there will be an acid reaction on the line of contact between the nitroglycerin and the soda mud, showing that the nitroglycerin is attacked.

From these comparatively simple nitro-compounds I pass to mixtures such as blasting gelatine, for instance. It must not be assumed that because two nitro-compounds are very stable under the influence of heat, a mixture of the same will have equal stability. There are many circumstances that may occur to the contrary. Blasting gelatine, for instance, is made by dissolving soluble guncotton in nitroglycerin with the aid of gentle heating. I will show later on that nitric peroxide can be developed from some nitro-compounds at much lower temperatures than that of the official heat test, and it will therefore depend much upon the temperature at which the solution of blasting gelatine is made, and upon the mechanical treatment it has undergone, whether the finished product will retain the degree of stability of its components or have it reduced. The same may occur with mixtures of picrates, solutions of nitrobenzene and guncotton, molten masses of nitro-compounds of low melting point, &c. In the manufacture of smokeless powder in flakes, the paste is frequently rolled into sheets by means of steam-heated rolls. With other smokeless powders the paste is made by prolonged treatment in kneading machines, whereby the temperature of the paste is increased. These operations, in my opinion, reduce the duration of the heat test, since, as I said before, treatment at elevated temperatures will result in the local development of nitric peroxide, and, if once started, it will develop more rapidly than before.

What I have said so far is perhaps of more interest to the manufacturer only, and not so much to the general consumer, who has a right to demand that the finished explosives, whether they be simple or composite products, should be of reasonable safety of handling. A good many of the explosives in use are mixtures of a number of constituents sometimes of a most complex nature, and the influence of these constituents has to my knowledge so far not been sufficiently studied. I have, in my long experience with explosives, often had an opportunity of investigating some of the questions at issue. Recently I had occasion to go fully into the matter, and will now give you the results of my experiments as far as I have yet carried them.

The finished explosives of most interest are guncotton, dynamite, blasting gelatine, gelignite, and the many smokeless powders, whether they contain nitroglycerin, nitrobenzene, &c., or not. Of guncotton I have said what is important. Dynamite is, as you know, nitroglycerin absorbed in kieselguhr, a pure silicious earth. Blasting gelatine is, as mentioned already, a solution of soluble guncotton in nitroglycerin, and gelignite is a similar solution of thinner consistency mixed with an absorbing powder consisting of potassium nitrate and wood pulp. From both dynamite and blasting gelatine the nitroglycerin may exude when it is not sufficiently held by the absorbent. Properly speaking, this is not a chemical, but only a mechanical instability. In the case of dynamite the exudation depends both upon the absorbing capacity of the kieselguhr and upon the influence of temperature to which the dynamite may be subjected during storage. If the temperature is too low, the nitroglycerin freezes in the explosive and contracts nearly one-tenth of its volume. It will thus leave the outer layers of the absorbent, and if the explosive is thawed up again, it may not distribute itself uniformly through the porous absorbent as before. If the temperature is too great,

then the nitroglycerin expands, and, should the guhr in the explosive have been originally slightly supersaturated, the nitroglycerin will ooze out. There is also the influence of moisture on dynamite, water by osmotic action displacing the nitroglycerin and going in its place. In the case of blasting gelatine, the quality of the soluble guncotton employed is of great importance, because it is well known now that neither the percentage of nitrogen nor the complete solubility of the guncotton in the nitroglycerin is a reliable criterion for the suitability of guncotton to safely retain nitroglycerin.

The possibility of dynamite or blasting gelatine becoming influenced by heat increases with the number of constituents that enter into its composition. Although it may be thought that in dynamite the nitroglycerin only has to be taken into consideration, yet it was found some 12 years ago that with perfectly good nitroglycerin and what was apparently excellent kieselguhr a good dynamite could not be made. On examination it was shown that the kieselguhr contained, besides traces of iron and charred organic matter from calcining, comparatively large amounts of aluminium sulphate. Even a small quantity of this proved to have a decomposing action on the nitroglycerin, with the consequent development of nitric peroxide.

Several years ago great difficulty was found in various factories to ship gelignite to Australia that on arrival would pass the heat test, and some cargoes were condemned and destroyed on that account. Of course, if such explosives travel by a sailing boat in tightly closed hatches without ventilation, exposed to the burning sun on crossing the equator, and arrive in the summer in India or Australia, the manufacturers will have to be careful to allow a sufficient margin for the heat test, and to prevent improper stowing on board ship. Nevertheless, most careful treatment and manufacture, and examination of gelignite before shipment and after a year of storage, revealed the fact that in some cases the gelignite stood all the heat tests in England, whilst in Australia it was condemned. On investigating the matter thoroughly I came to the conclusion that the wood pulp was at the bottom of the trouble. It is of importance that as little moisture as possible should be in the explosive, and therefore the wood pulp is subjected to a process of drying, which in some factories is carried so far that the wood pulp assumes a light chocolate colour; in other factories it is simply submitted to a temperature of about 120° C., so as to drive off all moisture. Generally the wood pulp is so dried in iron drums, with or without a stirring arrangement, that are exposed to a slow fire. If the wood pulp is slightly charred, it is of course evident that partial distillation of the wood and consequently development of acetic acid is going on; but even with comparatively low temperatures there may be accumulation of heat locally, and small nuclei of charring formed, which may spread rapidly in extent. Little notice was usually taken of these particles of charcoal. Acetic acid has a marked reaction with potassium iodide, and the following table shows from actual experiments that acetic acid, formed during the drying of the wood pulp, will influence the heat test very considerably :—

Name of Material.	With the Addition of	Heat Test in Minutes. Before the Addition.	After the Addition.	Loss.
Gelatine for gelignite	Wood-pulp and saltpetre.	31	20	11
" "	Wood-pulp.........	33	20	13
" "	"	50	29	21
" "	Saltpetre	31	32	2
Nitroglycerin	3 drops acetic acid .	30	3	27

Side by side with the development of acetic acid some methylic alcohol is also formed, and if the gelignite contains an alkali, like sodium carbonate, the presence of alcohol will make the alkali decompose the nitro-compounds more rapidly.

On the other hand, I do not think that the presence of acetic acid in gelignite that does not contain an alkali has any detrimental effect on the stability against heat, or that it would decompose the nitro-compounds, and I have, therefore, little doubt that gelignite may be condemned on account of low heat test when the nitro-compounds, and, in fact, the whole explosive is perfectly stable. The gelignite may be capable of standing heat for a very long time without any nitric peroxide being developed, and yet a reaction on the iodide paper caused through the presence of the harmless acetic acid.

We now come to a class of explosives that has developed so enormously within the last nine years as to revolutionise one branch of the industry, namely, the smokeless powders. It cannot be said that the last word as to their manufacture has been spoken, nor that the ideal powder has as yet been found; every year sees the invention of some new powder, and the old ones are being constantly improved. It is, therefore, not surprising that more attention has so far been directed to their good and safe manufacture than it was possible to bestow on their keeping qualities, since, whatever laboratory tests would be applied, the real test for the stability of a powder under the varying circumstances of its use can only be made by years of experience in practice.

You are now all familiar with the fact that modern smokeless powders are made either from guncotton alone or from mixtures of nitrocellulose and nitroglycerin, nitrobenzene, &c., with or without nitrates and similar substances. In trying to apply the iodide heat test to smokeless powders, I can well imagine the Home Office authorities must have had considerable difficulties, because, as will be apparent later on, these powders sometimes behave in a very erratic way as far as the regulation heat test is concerned. However, the difficulty was seemingly overcome by prescribing that in the case of Schultze and E.C. powder, &c., the powder should be dried in an oven at 120° F., then exposed for two hours to the air before the usual heat test was applied; whilst cordite and similar smokeless powders have to be ground in a mill, sieved through a set of three sieves, and only the powder retained by the second sieve used for the heat test.

It was in September 1895 that my friend Mr. Hermann Güttler, of Reichenstein, the well-known gunpowder manufacturer, pointed out to me the strange fact that, whilst he could get a heat-test reaction with iodide paper from his own powder, the so-called Piastomenite, he could get no reaction from Walsrode powder, or only after hours of heating; and that, moreover, if he put a paper that had the brown mark of the heat-test reaction into a test tube with Walsrode powder, the brown line disappeared almost immediately. The fact was so strange that it demanded investigation, and on my return, in October, I had an opportunity of going fully into the matter in connection with some other questions. It was apparent to me at once that there must be something contained in the Walsrode powder that would act upon the iodine liberated on the starch paper. However, I also found, as will be intelligible to everybody, that on putting a coloured iodide paper into an *empty* test tube and heating the tube to 180° F., the paper became white again in about five minutes through the evaporation of the iodine. The German zinc iodide paper proved to be more sensitive than the English paper, because I could get reactions with it in a third less time and make the iodine disappear in about half the time. Although this phenomenon threw me off the scent for a few days, yet I soon found that the time for the discoloration of the iodide paper was much too long to have such marked influence as shown in the case of Walsrode powder. I then found that old Walsrode powder gave a much quicker heat-test reaction than recently prepared one, and without going into the details of the many hundreds of experiments that I have made in order to ascertain positively all the circumstances connected with this disappearance of iodine, I had better give you the final rather startling conclusions that I arrived at.

You know that there are a large number of bodies that will either absorb iodine, such as fats and oils, or such that will combine with iodine under certain circumstances, or such that will dissolve it. I have been able to prove *that a large variety of ingredients can be and are contained in*

different smokeless powders that will stultify the iodide heat test. Perhaps foremost amongst them are acetic ether, acetone, and oils; but also vaseline, aniline, and various others. The greater the density the more difficult it is to drive off the solvent used in the manufacture of the powder, such as acetone, acetic ether, &c. Some powders will have retained as much as 1 per cent. of the solvent, some only a small fraction of a per cent. Although the latter may not show the presence of the solvent by the smell, yet, when such a powder is ground up and heated in the test tube, it will be found that a certain amount of the solvent has always been retained in the interior of the grains of the powder. This solvent or other suitable ingredient, if it acts upon the iodine, will prevent the formation of the brown line on the test paper, because, as the iodine is developed by the nitric peroxide, so the particular solvent or ingredient will make it disappear. This is what I call "hiding" the heat test, and the result will be that a powder may be fully in a state of decomposition, yet the solvent, like acetone or acetic ether, that is in the test tube will for some time prevent the formation of the brown line until either the development of nitric peroxide has become too great, or part of the solvent is driven out through leakages round the stopper and glass rod.

You will not be astonished now to hear that, with a powder made according to Mr. Hiram S. Maxim's patent, consisting of 88 parts of guncotton, 10 parts of nitroglycerin, and 2 parts of castor oil, using acetone as a solvent, I have sometimes not been able to get a heat-test reaction after even two hours of exposure to 90° C. The same thing, although in a lesser degree, happened with Walsrode powder and with cordite. In the case of cordite, iodide test papers made by two different chemists behaved differently, paper A showing the heat-test reaction after double time than paper B; yet when the same papers were tested on another kind of powder the order was reversed, and it was paper B that showed the reaction twice as late as paper A; but this was not due to a difference of sensitiveness of the test papers.

Considering the high temperatures to which oils can be submitted without apparently boiling or giving off visible vapours, it looked improbable that in the case of a powder containing it, it should be the oil that was hiding the heat test, and I was inclined to attribute it to the acetone only, but I soon found that, for instance, castor oil gave off volatile parts comparatively readily at somewhat low temperatures. I exposed castor oil in a beaker in a layer 1 in. deep to a temperature of 180° F. for 30 minutes, which is good average time and temperature for the Government heat test, and it was found that it lost 0·245 per cent., which, of course, is a far greater percentage than would be required to absorb the small amount of iodine on the test paper. I had also 20 grms. of castor oil exposed in a flat glass basin to a temperature of 38° C. for 72 consecutive hours, and the loss was 0·656 per cent.

It did not seem at all likely that vaseline would affect the heat-test reaction, and for a time it did not occur to me to try its influence, but some erratic behaviour of cordite caused me to approach the question from a different point of view by dissolving both castor oil and vaseline in hot ether and preparing three samples of the same batch of guncotton, one sprinkled with 5 c.c. of ether only, the other with three drops of castor oil dissolved in 5 c.c. of ether, and the third with three drops of molten vaseline in the same quantity of ether. I then submitted the three samples to a temperature of 40° C. in a drying oven to drive off the ether, and applied the iodide heat test. The times were as follows:—

	Minutes.
Guncotton alone	9
Guncotton and 3 drops of castor oil	19
Guncotton and 3 drops of Chesebrough vaseline	44

There seemed to me a possibility that, during the drying process of the finished explosives containing castor oil or vaseline, some volatile matter may go off that would influence the heat test, and therefore mineral jelly was also submitted for 72 consecutive hours to a temperature of 38° C., when the loss was found to be 0·061 per cent. Three drops of the residue of this exposed vaseline and castor oil were then dissolved in ether, as before, using a fresh batch of guncotton of good quality. I may say here already that all my comparative heat tests were made on 1·500 grms. of explosive at a temperature of 80° C., this being the nearest approach in the metric system to 25 grains at 180° F. The results were as follows:—

	Minutes.
Guncotton alone, sprinkled with ether	16
Guncotton with dried castor oil in ether	32
Guncotton with dried mineral jelly in ether	22
Guncotton with undried mineral jelly in ether	22

I had now the undoubted proof that there existed a number of ingredients of smokeless powders that would hide the heat-test reaction, and why some powders behaved in the iodide heat test in such erratic way. A powder that was ground in the evening and tested at once showed much longer heat test than when it was only tested next morning. Coarsely ground powders gave worse heat test than finely ground ones. Sometimes a powder that was exposed for many months gave a better heat test than a freshly prepared one. All this is due to the state of division of the powder when the heat test is made, and to the greater or smaller quantity of acetone or other ingredients retained that could be evaporated in the given time of the heat test. Moreover, the mill as used for grinding smokeless powder is one that does not grind but chips the powder, and although only those grains that are retained on a certain sieve are used for the test, yet there are great differences in size, whereby different quantities of solvent may be given off. All these experiments proved to me conclusively *that the iodide heat test, as at present prescribed, is absolutely inapplicable for most of the modern smokeless powders* and also some blasting explosives, since the iodine that is supposed to be liberated is acted upon by some ingredients contained in them. If you consider that the heat test is one of the chief criterions for the suitability of a powder for prolonged storage, and that every country in the world has a similar regulation test, you will understand the great importance of my discovery. A manufacturer, or even Government factories, cannot afford to have their deliveries rejected and suffering sometimes great pecuniary losses through an unsuitable method of testing.

It seemed to me that, before abandoning an old trusted friend, I had to get another, and I therefore set about to find a test that could not be hidden by some ingredients of the explosive, that is, one that would only be acted upon by the nitric peroxide developed through heat, and that would as much as possible be capable of application in a similar manner as the iodide heat test.

I have looked up all the various reagents that have been proposed during the last 40 years for the detection of small quantities of nitric peroxide. A number of them I could discard at once, because they would either require a complicated preparation or a treatment of the explosive by chemical operations, or they would act upon the explosive itself. I also did not wish to have a reagent in which the explosive would have to be dissolved or with which it would have to be mixed, because accurate time results could not be expected. Reagents for nitric peroxide have been proposed by Griess (a mixture of sulphanilic acid and naphthylamine in acetic acid), by Plugge (mercuric nitrate and carbolic acid), Jorrissen (fuchsine dissolved in glacial acetic acid), Vogel (Rosaniline), Meldola (para-amidobenzeneazodimethylaniline), Curtman (antipyrine), Kopp (diphenylamine), Frankland (sulphanilic acid and phenol), and Griess (*m*-phenylenediaminehydrochloride).

My chief aim was to get such a reagent that could be applied in a similar manner as the iodide heat test, namely, by placing a drop of the reagent on a piece of filter paper, which can be suspended on a hooked glass rod in a test tube, this way of testing having now become so universally adopted and giving such easy manipulation that it was desirable that any new test should be capable of being done in the same manner. Besides, such a test would not come into contact with the explosive itself, and no chemical combination between the explosive and the reagent could therefore be anticipated. Of the various methods, the antipyrine, metaphenylenediamine, fuchsine, and diphenylamine reactions recommended themselves most. Antipyrine

I had to disregard very soon, because it only gives a very slight emerald-green colour, which is not sufficiently distinguishable. *m*-Phenylenediamine gives a yellow reaction, but proved to be uncertain in effect, and is not well visible in artificial light. For fuchsine, Jorrissen's and Vogel's description was that 0·01 grm. should be dissolved in 100 c.c. of glacial acetic acid. This was found too strong for my purpose, and I therefore diluted the glacial acetic acid with water. A drop of this fuchsine solution put on a test paper gives a most beautiful reaction with nitric peroxide: its colour changes from a light purple-red into violet, then gradually into sky-blue, then green, then yellow, and finally disappears altogether. A drawback to it is that free mineral acid turns it yellow at once. I had some hopes of being able to use this test, but after numerous experiments with different explosives I found that the change from red to blue, which I considered to be the safe sign of development of nitric peroxide, was not by any means regular. Sometimes the violet colour appeared immediately before the blue one, but at other times the red changed at once into violet, and it took a very long time before a blue reaction could be got at all. I therefore concentrated all my efforts to the diphenylamine reaction, which, as is well known, is a very sensitive and good one. However, the usual way—as, for instance, applied in the examination of water, namely, dissolving the diphenylamine in strong sulphuric acid and adding a drop of it to the water—could not be resorted to, because the strong sulphuric acid would not be without effect on the test paper, whilst at the same time the reaction would not be sufficiently sensitive unless this solution could be diluted. Diphenylamine dissolves readily enough in strong sulphuric acid, but if this solution is diluted with water, the diphenylamine is precipitated. I used various devices, such as putting the diphenylamine solution on a ground glass plate, then on a milk glass plate, then dipping vegetable parchment paper into it, but in all these cases I got a drop of the solution hanging down, which required a large quantity of nitric peroxide to show coloration, whilst in my case only very slight traces of it were to be detected. I then found that on putting a few drops of water into a flask with the concentrated sulphuric acid, the heat generated would dissolve the diphenylamine, and if further quantities of water were gradually added, still more heat was generated, and a clear solution of diphenylamine in the ratio of 1 to 500 obtained. In order to prevent the too rapid evaporation of the water on the test paper, I mixed equal quantities of this solution and Price's glycerin, and a few drops of this liquid put on the filter paper proved to be an excellent reagent for nitric peroxide. Nevertheless, after keeping this solution for several months, some pink flocculent matter was thrown down, evidently a product of reaction between the strong sulphuric acid and the diphenylamine; and after some trouble I found that the following method of preparation and the following way of carrying out the test answered all purposes, and that diphenylamine solution so prepared could be kept for a very long time:—

Take 0·100 grm. of diphenylamine crystals, put them in a wide-necked flask with a ground stopper, add 50 c.c. of dilute sulphuric acid (10 c.c. of concentrated sulphuric acid to 40 c.c. of water), and put the flask in a water-bath at between 50° and 55° C. At this temperature the diphenylamine will melt, and at once dissolve in the sulphuric acid, when the flask should be taken out, well shaken, and allowed to cool. After cooling, add 50 c.c. of Price's double-distilled glycerin, shake well, and keep the solution in a dark place. The test has to be applied in the following way:—The explosives that have to be tested are to be finely subdivided, guncotton, nitroglycerin, dynamite, blasting gelatine, &c. in the same way as at present directed by the Home Office regulations. Smokeless powders are all to be ground in a bell-shaped coffee mill as finely as possible, and sifted as hitherto. 1·500 grms. of the explosive (from the second sieve in case of smokeless powder) are to be weighed off and put into a test tube as hitherto used. Strips of well-washed filter paper, or strips of any good chemical filter paper, 25 mm. long, 10 mm. wide, are to be hung on a hooked glass rod as usual. A drop of the diphenylamine solution is taken up by means of a clean glass rod, and the upper corners of the filter paper are touched with it, so that when the two drops run together about a quarter of the filter paper is moist. This is then put into the test tube, and this again into the water-bath, which has been heated to 70° C. The heat-test reaction should not show in a shorter time than 15 minutes. It will begin by the moist part of the paper acquiring a greenish-yellow colour, and from this moment the paper should be carefully watched. After one or two minutes a dark blue mark will suddenly appear on the dividing line between the wet and the dry part of the filter paper, and this is the point that should be taken.

I believe this test will recommend itself as very simple and effective to everybody, and I hope it may be adopted as a standard. I may be therefore permitted to give some explanations for the reasons why certain details have been worked out in the way indicated.

Instead of having to re-crystallise potassium iodide from alcohol, to carefully wash filter paper, to dip it, after drying, into the potassium iodide solution, to dry this again in a dark room, to cut off the edges, divide it into strips, and yet have test papers that will only keep for a short time,—this new test of mine allows to use any good chemical filter paper, such as it is supplied in circular sheets of great purity, and a solution of diphenylamine, which, when once prepared, is practically unaltered, or at least, within my experience, not during six months, and can be made within a few minutes. The diphenylamine solution has a strength of about 1 in 1,000 by volume. The subdivision of the explosive requires some remarks. In all these tests it is essential that the explosive should contain as little moisture as possible, otherwise the test paper will soon be thoroughly dampened, and no test could be made. It is, however, not advisable to dry explosives at higher temperatures, because my experiments have shown that nitric peroxide in minute traces is developed already between 45° and 50° C., and if explosives were dried at this temperature the heat test would not be accurate. The present Home Office regulation of drying guncotton in an open water-oven at 120° F. is satisfactory, although it might be advisable to dry one hour instead of a quarter of an hour, but at 40° C. only. The mixing of blasting gelatine and the like with French chalk is a very good way of subdividing the tough mass. Smokeless powders should, as a rule, not be dried, because many of them contain ingredients that are volatile to some extent even at 40° C., and because they do not, as a rule, contain moisture to an appreciable extent. On the other hand, they should be ground and sifted. The reason for the desirability of subdivision is twofold. Firstly, in a very dense and tough explosive the nitric peroxide developed would have to loosen the structure of the grain before it could escape, whereby the reaction would be retarded. On the other hand, although the test tube has a small diameter and the height of the explosive in the test tube is small, yet the coefficient of transmission of heat cannot at all be neglected. If the powder is in the shape of dust, the emission of nitric peroxide through the layer of powder into the empty space in the test tube takes some time. If the explosive be too coarsely grained, a lesser quantity of nitric peroxide may be set free in a given time. Were a smokeless powder not ground at all, the hard surface resulting from black-leading, as it is sometimes done, would delay the development of nitric peroxide. It is therefore desirable to have the powder in a fairly uniform state of subdivision, so as to get with the same explosive an equal constant, due to the delay in transmission of heat and start of the decomposition.

I have said just now, that the coefficient of transmission of heat cannot at all be neglected, and direct experiments to prove this gave remarkable results. In a water-bath, heated to 70° C., the temperature of the room being 12° C., it takes $8\frac{1}{2}$ minutes to reach a temperature of $69\frac{1}{2}$° C. in a test tube filled with ground cordite or ground ballistite, but 11 minutes if the ballistite is not ground. The temperature rises to 60° C. in about 3 minutes, but the last 10 degrees take so long, and it makes little difference whether the test tube is of thick or thin glass, the time for heating the

interior to 70° C. differing only by the fraction of a minute in favour of the thicker tube.

I was inclined to attribute this delay in heating the interior of the test tube to the fact that half the length is exposed to the cooling action of the ambient air. I had, therefore, a test tube made by Messrs. Müller and Co., of High Holborn, the upper half of which was jacketed, and the jacket completely evacuated; but there was quite a negligible difference in the time. On filling the test tube with mercury, the time for heating to 70° C. is 2 minutes, and, the delay through the thermometer having been found to be 30 seconds, the loss of time by the transmission through the glass is 1½ minutes. The reason for the additional delay of 6½ minutes is to be found in the fact that air is a bad conductor of heat, and it is the air enclosed in the test tube that requires this additional time.

I now tried to find whether the fact of the air expanding through heating, and an increased pressure being set up for the unity of volume, had any influence on the time for heating. For this purpose I tried two empty tubes, first with the india-rubber stopper tightly fixed in, and afterwards with it laid loosely on the top so as to allow for expansion. The following table shows the result, the water-bath being kept at 70° :—

After	Ordinary Test Tube.		Vacuum-jacketed Test Tube.	
Minutes.	Tight Stopper.	Loose Stopper.	Tight Stopper.	Loose Stopper.
	°	°	°	°
0	14	14	14	14
1	31½	36	32	37
2	44¼	52	45¾	53
3	58½	60¾	54¼	62
4	59¾	65	60½	66
5	63¾	67¼	63¾	67¾
6	66¼	68¼	66	68½
7	67½	69	67¼	69
8	68½	69¼	68	69¼
9	69	69½	68¾	69½
10	69¼	..	69¼	..
11	69½	..	69½	..

It appears, therefore, that if the stopper of the test tube or the glass rod in the stopper fit loosely, the duration of the heat test can be reduced by about two minutes.

The temperature for the heat test I have, after very careful consideration, put down at 70° C. A general test like the heat test ought to be the same in every country, so as to be able to compare results; and as we are gradually giving up the use of grains and Fahrenheit temperature scale, it was best to resort to the metric system. The temperature was originally put down by the Home Office at 150° F. for guncotton, Schultze powder, E.C. powder, &c., and was gradually raised to 170° F. for guncotton and to 180° F. for all other explosives. A careful comparison of a large number of explosives at present in the market, both with the iodide and my diphenylamine test, has shown that the temperature of 180° F. is too high to allow of a sufficient margin for observation and errors. I believe this high temperature was resorted to because with the former temperature of 170° F. some smokeless powders that hide the heat test seemed to give such extraordinarily good tests as to necessitate waiting for hours for a reaction, and it was thought to expedite matters by raising the temperature. I have shown by my diphenylamine test that the reaction actually takes place in a very short time at lower temperatures, and even some good powders in the market would, if exposed at 180° F., scarcely stand 10 minutes. It will not be doubted that the temperature of 70° C. is one that is not likely to occur under ordinary circumstances of storage and use.

The sensibility of my reaction is a very marked one; the blue mark on the test paper appears within a few seconds, whilst with iodide paper it takes sometimes two minutes to have a brown line well defined, and I know how even careful operators have often disputes as to whether there was a brown line already or not. At night the blue mark is not so well visible as at daytime, but still sufficiently clear, so that it cannot be missed. It is best to look at the paper with the light shining on to it, and not to look through the paper. In cases of doubt a screen of filter paper behind the test tube will make the blue more visible. The best light for such observations is the incandescent gas light, for reasons that will be readily understood; with electric or ordinary gas light a screen of thin light blue paper will prove to be of advantage.

I now append here a long series of heat tests made at 80° C. with almost every smokeless powder that I could get hold of. I wish to state distinctly that this table is not made for advertising a particular explosive, nor can an inference to the general keeping qualities of an explosive be drawn from such isolated experiments. My tests simply show the behaviour to the heat test of a particular sample of powder that I obtained in the open market, and were chiefly done with a view to compare the relative value of the iodide heat test and my diphenylamine test.

Heat Tests of Various Smokeless Powders.

Temperature, 80° C. (176° F.); Quantity of Powder, 1·500 grms.

I = Iodide paper; G = Guttmann's diphenylamine paper.

Name of Powder.	Remarks.	Composition.	Time in Minutes.	
			I.	G.
Guncotton	From Waltham Abbey	.. ..	9	8½
Schultze	Small white grains	Wood-nitrocellulose, and saltpetre	16	14
E.C., original	From 1882, small yellow grains	Nitrocellulose and saltpetre, without camphor.	10	11
E.C., No. 1	18 months old, greyish, large grains	" "	4	5
E.C., No. 2	18 months old, orange, large grains	Nitrocellulose, saltpetre, and camphor	25	not tested
E.C., No. 2	Recent manufacture	" "	76 no trace.	18
J_1. French Government sporting	1893, greenish-brown, large chips	Nitrocellulose and ammonium dichromate.	10	9
J_2. " " "	1893, same colour, smaller chips	" "	10	8
Cannonite	Small cylinder segments, black, rough surface.	Nitrocellulose, saltpetre, and resin, black-leaded.	13	13
Rifle cannonite	" "	" "	22	23
Amberite	Pink, large grains	Nitrocellulose, barium nitrate, and paraffin.	8	9
Walsrode, K_1	Grey, small grains	Nitrocellulose, dissolved in acetic ether.	57	22
" R.P.	Small white grains	" "	50	20
" W.G.P. 92 A	Flakes, slightly black-leaded	" "	50	21
" revolver	Grey, small grains	" "	45	17
" K P. 2	" "	" "	50	26
" ca[illegible]	Flakes, 5 mm. square, ¼ mm. thick	" "	75 no trace.	2[illegible]

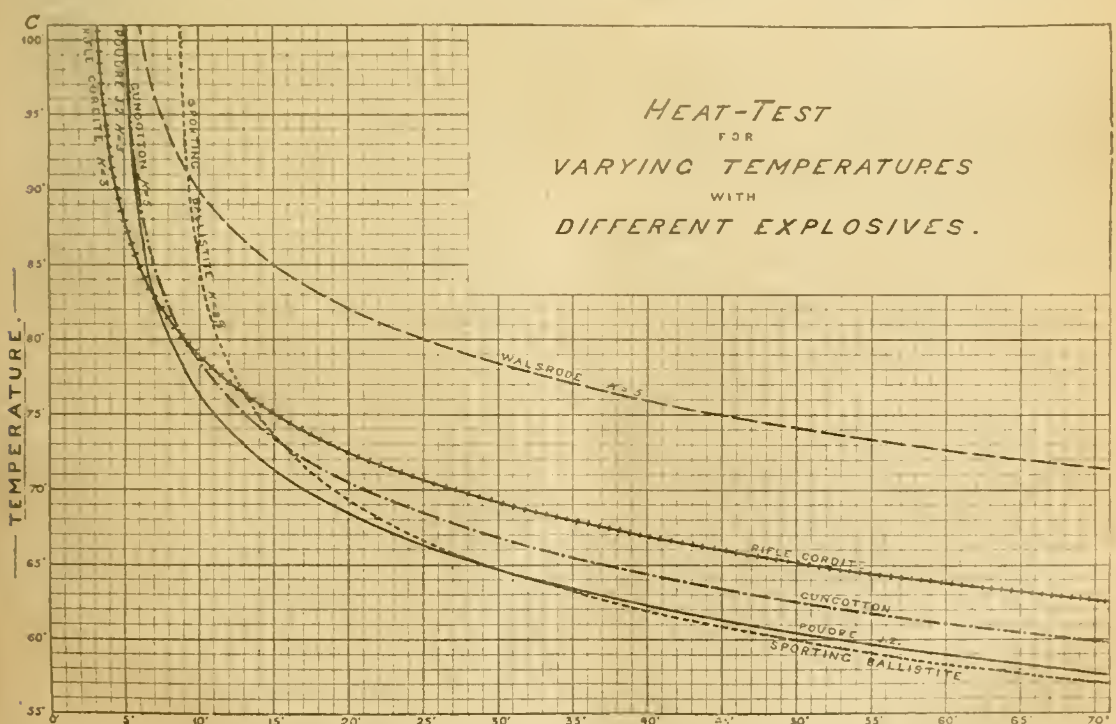
Heat Tests of Various Smokeless Powders—*continued.*

Name of Powder.	Remarks.	Composition.	Time in Minutes. I.	Time in Minutes. G.
Sporting ballistite	Thin flakes, black-leaded	Nitroglycerin and nitrocellulose without solvent.	13	13
v. Forster, sporting............	Thin flakes............................	Nitrocellulose. Solvent, acetone.......	19	8
" rifle, No. 2	" black-leaded, 18 months old	" "	20	18
" " No. 3.........	.. ..	" "	80 no trace.	28
Hudson Maxim's powder	Perforated cords......................	Nitroglycerin and guncotton	..	9
Plastomenite	Brown grains..........................	Nitrocellulose and dinitrotoluene	17	17
0·450 Rifleite	18 months old, dark brown flakes	Nitrocellulose and nitrobenzene	14	18
" "	New, light brown flakes	" "	11	11
0·303 "	Flakes, black-leaded....................	" "	16	17
0·250 "	" "	" "	10	10
Z.Z. "	" "	" "	10	11
Revolver rifleite	" "	" "	9	10
S.S.	White, large grains	" "	10	10
S.R.	Pink, large grains......................	" "	12	10
S.V.	" "	" "	9	7
Coppal	White, large grains	Nitrocellulose, saltpetre, and paraffin. Solvent, acetone	60 no trace.	9
Hiram S. Maxim	Made according to patent	Guncotton and nitroglycerin, with 2 per cent. of castor oil..............	60 120	11
Rifle cordite	Thin strings	.. ..	90 37	10
Normal powder................	Small flakes............................	Nitrocellulose	11	11

It will be seen clearly from the above table that the addition of castor oil, acetic ether, acetone, camphor, vaseline, &c. make the iodide heat test quite impossible, whereas perfectly reliable results are obtained with my diphenylamine test. I have repeatedly made a number of diphenylamine tests with the same batch of powder at very carefully regulated temperatures, and always found the test to coincide very well. This led me to investigate whether a relation could be found between the temperature to which a sample is exposed in the heat test and the time within which the diphenylamine reaction shows itself. I used for my experiments to this effect guncotton from Waltham Abbey, since in this no other constituent could influence the heat test, and checked them by several smokeless powders. The following diagram has been made from actual well coinciding experiments, and you will see from the curves that the relation between the temperature and the time is one that can be expressed for differences of minutes in steps of degrees centigrade in a geometric progression of the value :—

$$k + m, k + 2m, k + 4m, k + 8m, k + 16m, \ldots\ldots$$

k being a constant due to the delay in transmitting the heat through the explosive and in starting the decomposition.



I believe this geometric progression to hold good for all nitro-compound explosives, and at some future time intend to lay before you diagrams of a large number of explosives. Meanwhile I think it will be useful to those wishing to construct similar curves, if I give them simple formulæ by which they can do so. The formulæ are the result of

careful calculations, and I do not think it necessary to state at length how they were arrived at, as their correctness can be easily checked.

Let—

a, b be the times in minutes found by experiment,

$\frac{z}{a}$ the relation between any two real times in the geometrical progression,

$d = b - a$ = difference between the times found by experiment,

$m_1 m_2 m_3 m_4 \ldots\ldots m_n$ the real times for each degree without the constant,

k a constant for the transmission of heat and starting the decomposition,

n the number of degrees centigrade between one determination and the other,

f the factor of the geometrical progression;

then we have—

$$f = \sqrt[n-1]{\frac{z}{a}} = 1{\cdot}1487$$

$$m_1 = \frac{d}{f^n - 1}$$

$$a - m_1 = k$$
$$a - k = m_1,$$
$$m_1 f = m_2,$$
$$m_1 f_2 = m_3,$$
$$m_1 f_3 = m_4,$$
$$m_1 f_4 = m_5,$$

and so forth;

$$m_1 f^{n-1} = m_n.$$

The powers of f can be found for the practical variations of temperature from the following table:—

$f = 1{\cdot}1487$	$f^{15} = 8{\cdot}00$	$f^{29} = 55{\cdot}72$
$f^2 = 1{\cdot}32$	$f^{16} = 9{\cdot}19$	$f^{30} = 64{\cdot}00$
$f^3 = 1{\cdot}52$	$f^{17} = 10{\cdot}56$	$f^{31} = 73{\cdot}52$
$f^4 = 1{\cdot}74$	$f^{18} = 12{\cdot}13$	$f^{32} = 84{\cdot}45$
$f^5 = 2{\cdot}00$	$f^{19} = 13{\cdot}93$	$f^{33} = 97{\cdot}01$
$f^6 = 2{\cdot}30$	$f^{20} = 16{\cdot}00$	$f^{34} = 111{\cdot}43$
$f^7 = 2{\cdot}64$	$f^{21} = 18{\cdot}38$	$f^{35} = 128{\cdot}00$
$f^8 = 3{\cdot}03$	$f^{22} = 21{\cdot}11$	$f^{36} = 147{\cdot}03$
$f^9 = 3{\cdot}48$	$f^{23} = 24{\cdot}25$	$f^{37} = 168{\cdot}90$
$f^{10} = 4{\cdot}00$	$f^{24} = 27{\cdot}86$	$f^{38} = 194{\cdot}01$
$f^{11} = 4{\cdot}59$	$f^{25} = 32{\cdot}00$	$f^{39} = 222{\cdot}86$
$f^{12} = 5{\cdot}28$	$f^{26} = 36{\cdot}76$	$f^{40} = 256{\cdot}00$
$f^{13} = 6{\cdot}06$	$f^{27} = 42{\cdot}22$	
$f^{14} = 6{\cdot}96$	$f^{28} = 48{\cdot}50$	

Take as an example the guncotton curve. Let us say we found by experiment for 80° 9 min., and for 70° 21 min. We have then—

$$m_1 = \frac{21 - 9}{1{\cdot}1487^{10} - 1} = \frac{12}{4 - 1} = 4.$$

$$k = 9 - 4 = 5.$$

Temperatures.		Duration in Minutes. Real.	Apparent.
80°	$= \frac{12}{4 - 1} =$	4·00	9·00
79°	$4 \times 1{\cdot}1487 = 4 \times 1{\cdot}15 =$	4·60	9·60
78°	$4 \times 1{\cdot}1487^2 = 4 \times 1{\cdot}32 =$	5·28	10·28
77°	$4 \times 1{\cdot}1487^3 = 4 \times 1{\cdot}52 =$	6·08	11·08
76°	$4 \times 1{\cdot}1487^4 = 4 \times 1{\cdot}74 =$	6·96	11·96
75°	$4 \times 1{\cdot}1487^5 = 4 \times 2{\cdot}00 =$	8·00	13·00
74°	$4 \times 1{\cdot}1487^6 = 4 \times 2{\cdot}30 =$	9·20	14·20
73°	$4 \times 1{\cdot}1487^7 = 4 \times 2{\cdot}64 =$	10·56	15·56
72°	$4 \times 1{\cdot}1487^8 = 4 \times 3{\cdot}03 =$	12·12	17·12
71°	$4 \times 1{\cdot}1487^9 = 4 \times 3{\cdot}48 =$	13·92	18·92
70°	$4 \times 1{\cdot}1487^{10} = 4 \times 4{\cdot}00 =$	16·00	21·00

If we compare the curves on the diagram, we find that guncotton has a low constant and a high factor of progression, whilst sporting ballistite has a high constant and a very low factor. A high constant means that it takes a long time to start decomposition, a high factor that the explosive decomposes very slowly. The best explosive will, of course, be one that has both a high constant and a high factor.

It seems to me that it will scarcely be sufficient to say that an explosive must stand a certain number of minutes at a particular temperature in order to pronounce it as one that possesses reasonable safety. I can quite conceive a case where an explosive has a constant approaching 0, and yet the decomposition in increased temperatures may take wide intervals of time. On the other hand, you may have to wait rather a long time to start decomposition, but once started it may develop rapidly.

Considering that, as shown before, it takes $8\frac{1}{3}$ minutes to heat the contents of a test tube to 70° C., it may surprise that the constant of some powders on the diagram is 5 minutes, and that of guncotton 3 minutes only, but this is due to the fact that with them decomposition starts at a lower temperature than 80° C. The heat test value of an explosive will, therefore, be the inferior the more its constant is below the time required for obtaining equilibrium between the outer and inner temperature of the test tube, and it will be advisable to fix a minimum limit for the constant, say 5 minutes.

I do not think that with my diphenylamine test all the objections are overcome which have been raised from time to time against the value of the heat test as such. Those well acquainted with such tests know that widely different results are sometimes obtained under the same conditions; that frequently no reaction at all is shown on the test paper, or one that does not give the required depth of tint even after hours of heating. The same will occur with my diphenylamine test; and the reasons for these vagaries of the heat test are difficult to find. I believe they are partly due to the physical state of the explosive, but in a good many cases the treatment of the explosive during manufacture will account for very large differences. The heat test will always be an empirical one to a certain extent, but the beneficial effect obtained by it so far has proved that it is a necessity.

Discussion.

Mr. W. F. Reid considered the paper of even greater importance than it might appear at first sight, since the test which it dealt with was one which, as a condition of the license under which they work, affected all the manufacturers of explosives in this country. There could be no doubt that the present heat test of explosives was most erratic in its behaviour, and had in consequence engaged the attention of experts for many years past. Mr. Guttmann had therefore rendered great service in discovering the reasons of its unreliability. It would, he thought, have been better, in discussing the ingredients which gave rise to the irregularities complained of, to classify them into volatile and non-volatile bodies, seeing they possessed properties of very different character. While he agreed with the author's remarks as to the irregularities of the heat test, he could not quite accept his explanation of the causes. In making experiments on the flashing points of explosives he had devised an apparatus which consisted of a bath of mercury in a porcelain vessel, and capable of being completely covered in. Using this, he found that the flashing point of explosives rose considerably, especially that of guncotton; a sample of soluble guncotton heated in the air-bath in the ordinary way flashed at 125° C., but in the mercury bath at 212° C. Another sample, when mixed with castor oil partially nitrated flashed in the air-bath at 145° C., but in his apparatus at 205° C. Further experiment revealed that the castor oil was itself nitrated by nitrogen peroxide and nitrous acid. He therefore concluded that in all cases where castor oil, or other drying oil of similar composition, was mixed with the explosive, it would absorb nitric peroxide, but in the case of acetone there might be a different action. With regard to the presence of a solvent in a powder, his own experience corroborated Mr. Guttmann's. He had found solvent present in E.C. powder 15 years old. With regard to the volatilisation of the castor oil, he thought that was accounted for by the presence of water, from which the oil was never entirely free. Glycerin would also sometimes contain oxidation products of linseed oil, if the two bodies had been at any time in contact. He hoped that Mr. Guttmann's new test would be of general utility.

Mr. Bertram Blount, referring to the statement that alkalis have a direct tendency to decompose nitric compounds, said he would like to see the equation showing how that came about. He heartily congratulated the author on the prescience which had led to the discovery that particles of lead sulphate in the washing apparatus carried up so much nitrous matter as to impair the quality of the nitroglycerin. He would also be glad to be informed—as the author inferred that neither the chemical composition nor the physical condition of the nitrocellulose was an index to it—how one could discern the best quality of nitrocellulose to be used. Could this knowledge be gained only by direct experiment, or could the author give them any further information as to the cause of the phenomenon referred to? Another interesting point arose out of the author's statement that "acetic acid has a marked reaction of potassium iodide." Save that it might tend to liberate hydriodic acid, he thought that acetic acid would be unlikely to affect potassium iodide; certainly he should not expect it to liberate iodine. If this was so, he did not see how the test could be more sensitive when acetic acid was there. He quite agreed with Mr. Reid's opinion that the loss of weight observed in the case of castor oil was in all probility due to the expulsion of water, and not to any loss of oily matter. Further, supposing that Mr. Guttmann's test were adopted, was it not a fact that the higher oxides of nitrogen were absorbed by oils both of the drying and non-drying classes, and would it not therefore follow that any oxide of nitrogen evolved would not only have its action on the paper smothered, but would be stopped at the very source by the oil present in the explosive? Considering what happened in the elaidin reaction, it was at least possible that the higher oxides of nitrogen might never get away at all. The new test appeared to him to be a very happy idea, and if it led to the substitution of a method depending on something other than the liberation of iodine, the author's action was amply justified.

Mr. P. G. Sanford observed that his own experience with one of the powders shown on the board did not agree with the author's, who gave 13 minutes as the time for the test reaction, both when using the old iodine test and with his own heat test. It was well known that this powder was gelatinised with acetone; he would like to ask Mr. Guttmann how he explained this.

Mr. G. Trench bore witness to the generally satisfactory results obtained by the heat test. The failures of cargoes sent to Australia to pass the test there were due rather to the dirty condition of the holds of the ships employed than to the character of the explosives themselves. He also hoped that some arrangement might be made whereby manufacturers could be supplied with test papers from the Home Office, so that they might work with the same papers as those with which the official test was to be made. At present manufacturers were making their own, and he had found some considerable time-differences between papers, all of which he was assured were recently made. If they were supplied with a new set of test papers at the beginning of each month, it would be a most satisfactory arrangement, and for his part he would be most willing to pay for the convenience.

Mr. W. Macnab, while admitting that the results of the heat tests were sometimes erratic was surprised that the author had made out so bad a case against iodide of starch paper, especially with respect to the testing of Maxim powder. So far as his experience with castor oil and other "suitable oils" had gone the difficulty had not been to get a reaction but that the reaction was got only too readily. As a rule, however, the failures were due to the unsatisfactory character of the nitro-cottons and the nitro-glycerin used. He knew, however, that with castor oil a good powder could be made which would easily stand the heat test for from 15 to 30 minutes. These facts proved that oil could not always have the tremendous masking effect ascribed to it by Mr. Guttmann. He considered the diphenylamine test well worth further study, though the old test was by no means so unsatisfactory as the author considered it.

Mr. R. W. S. Griffith, as a manufacturer, had a deep interest in the question before the meeting. He had used the heat test for 25 years, and quite agreed with the author's statement that it was sometimes apparently erratic. At the same time, those who used it daily, became accustomed to its behaviour, as was the case in his own laboratory, and having that knowledge were able to so use the test as to obtain perfectly reliable results. He quite agreed with the views expressed by Mr. Guttmann, as to the necessity for insisting on great care and purity in the materials used in the manufacture of nitro compounds. In his own manufacture he had been compelled to bar the employment of alkalis and lead. The use of the latter body had been the cause of a serious accident at his own works 18 years ago. Experience also showed that in the preparation of wood pulp, it was essential to avoid any charring during the drying process. He mentioned these facts in confirmation of the statements made by the author. With regard to the shipments to Australia, he agreed with Mr. Trench that the failures were due rather to the condition while in the ships than to the climate, for he had known powder to be sent out and to be brought back again without being landed, and received back the worse for the journey. He regarded the proposed test as a valuable auxiliary to be used in conjunction with the older method. There were some points in the subject extremely difficult to understand; for example, looking through the figures given by the author, and knowing the composition of the powders concerned, he could not see that the law laid down by Mr. Guttmann as to the presence of "hiding" materials producing differences of time was in force throughout the whole of the tests. In considering this question, the physical condition of the material at the time the test was applied should be carefully borne in mind, and that was a point which he hoped the authorities would deal with in a more satisfactory manner than at present.

Dr. A. Dupré, F.R.S., said that, as Chemical Adviser to the Explosives Department of the Home Office, and responsible for the application of the heat test to all explosives manufactured in or imported into this country, it would be understood that he could not criticise the paper fully at that moment, as any remarks from him might hamper future action by the authorities. He could assure the author, however, that he would give the proposed process his best consideration, and if he found it an improvement on the old test would be happy to recommend it to the Home Office, the authorities at which were always ready to receive and fully consider complaints or suggestions in the most courteous manner. Having said this he would make a few general remarks. He could not admit that the iodine test was as bad as Mr. Guttmann made out, but, naturally, an inventor was enamoured of his own process and could hardly be expected to be thoroughly impartial. Many complaints had been made about the heat test and he had often investigated them, but they generally turned out to be due not to the test itself but to the method of applying it. To take the case of maximite and cordite, was it not possible that the difference in those cases was due to the fact that the powders contained nitroglycerin, which was volatilised and decomposed by the sulphuric acid on the test paper? He had never known cordite to stand 120 minutes, rarely for much more than 30 minutes, but there were many points which might account for the difference, for instance, with a leaky stopper the test might be protracted for hours. Again, if the sample was too moist the margin between the dry and the moist part of the paper would shift downwards and there would be no test got at all. The explosive must be of the proper degree of dryness so that the dividing line did not move perceptibly. As regards the time necessary to heat the sample in the test tube, he thought that Mr. Guttmann had over-estimated its influence, the time of the test being governed not so much by the total quantity of explosive used as by the amount of the surface exposed to heating. One might go as low as three grains of gun-cotton, and if it were properly disposed might get the test as fast as with 20 grains. The quantity was given mainly to give sufficient heating surface. If vaseline had the influence attributed to it by the author one would imagine cordite, which had been heated for some time in an open vessel, would give the heat test more readily than cordite heated in a closed vessel, but the reverse was the case. He thought the author was mistaken, too, as regards the action of acetic acid. If that

body were added to good guncotton it would affect it but slightly at first, but if kept for some weeks at 100 the test would be materially lowered. With regard to the shipments to Australia, to which many references had been made, he would point out that the cargoes condemned were as often blasting gelatin as gelatin dynamite, the latter only containing wood meal. He had found some years ago, and mentioned the fact in his annual report, that gelatin dynamite, Nos. 1 and 2 (gelignite was not an officially recognised name) stood the heat test worse than blasting gelatin, and he then thought this due to the wood meal; now, however, it was just the reverse and the latter stood the test worse. This might be due to the fact that the one could be made at an ordinary temperature, while the other required a slightly elevated temperature, therefore it was probably not the wood meal which did the chief damage. Another point not referred to was the very general opinion held among manufacturers and chemists that it was the acid which caused bad heat tests. That he considered a great mistake, as a rule acidity could be readily washed out. It was some compound or compounds which decomposed at a lower temperature than the explosive proper, which were mainly of volatile character, and difficult to get rid of by washing, which caused the difficulty. The difficulty in the case of nitroglycerin was to get the happy medium. If one used too much air the mixture was churned up too much and separated with difficulty, but with sufficient aeration nitroglycerin could be made to stand any reasonable test. All explosives were the better for aeration, and it was of no use to try to get rid of the acid only. No one was more sensible of the shortcomings of the heat test than himself; nevertheless, he was sure that, if carefully applied, it did no injustice. In the process of manufacture the explosive could be followed step by step, and as it was more washed or aerated the test would rise. An explosive which would stand the heat test well was sure to be a stable body in our climate at least, though not, perhaps, in tropical climates; but it did not follow that if it did not stand the heat test that it was necessarily a bad one, but there being no difficulty in getting an explosive which would stand the heat test it was well to insist upon it. Undoubtedly the test was, in a great measure, influenced by the physical condition of the bodies treated, and it was therefore extremely difficult to arrange tests which, while safeguarding the public, would not unduly hamper the manufacturers. This they had endeavoured to do by laying down rules as to the degree of sub-division and using different temperatures. They had also carefully regulated the amount of test tube which should be above the water, and the distance at which the margin between the dry and the wet paper should be above the heated surface. That was an important point; for if the paper were put into too warm a part of the tube the test was necessarily prolonged. It should be near enough to be readily affected, but not too near. In the early days personal equation often influenced the test, or again, it was a question of light, as on a dull day the action would not be defined with sufficient sharpness. To overcome that difficulty they had instituted a standard tint line. The standard paper was placed in a tube side by side with the tube containing the sample to be tested, and the operator had then before him a paper without the line and one with the brown line. That method eliminated the personal equation and enabled the test to be made in any light sufficient for seeing the standard line. Lastly, most of the difficulties of the test arose when dealing with good explosives giving a heat test of, say, 20 minutes and upwards, whereas when dealing with bad explosives difficulties were rarely experienced and different observers generally agreed. The former was of but little importance, for whether a powder stood for half an hour or an hour was, from his point of view, immaterial, but it was of importance that bad explosives should be discovered.

Mr. Otto Hehner observed that possibly the two tests were not on the same footing. The iodide test was used generally to detect nitrous compounds, the diphenylamine test to detect nitrites or nitrates, the latter of which would not be detected at all by the iodide test. It was obvious, therefore, that in many cases the tests could not give the same results, because they did not apply to the same thing.

Dr. S. Rideal thought that the alleged absorption of nitrous compounds by lead sulphate might have some importance in connection with accumulator work, for if nitrous compounds were so absorbed it would influence the behaviour of accumulator plates afterwards. He agreed with Mr. Hehner's remarks as to a *nitrate* test giving different results to one for *nitrites*; and it also seemed to him that the test effected a narrowing of the variations, whereas what was wanted was a test which increased the differences. He feared, therefore, that diphenylamine was too delicate a reagent for use in this connection, as it had been found in the case of milk examination. It would be recollected that its use was suggested for detecting the adulteration of milk by water, but it was afterwards found that milk to which no water had been added would, under some conditions, give the diphenylamine reaction.

Mr. F. W. Jones writes as follows:—Does acetone "hide" the heat test? Mr. Guttmann's experiments say decidedly "No." Many of the explosives of which he gave comparative results, contain acetone, some even as much as 2 or 3 per cent. In some of those I have in mind there was an actual increase of time with the diphenylamine paper, whereas Cooppal's which could not contain more than the merest trace of its hardening solvent (I question if it is acetone), showed a very great difference in the two tests. The diphenylamine paper gave generally tests of shorter duration, and one would naturally expect where the iodide paper gave the longest time the difference would be the greatest, which explains many of the results.

The heat test, we must all admit, is an empirical one; furthermore, it is a washing test, and while it is sufficiently reliable for nitrocellulose and nitroglycerin in the raw condition, it is quite unreliable for them when made up into their various forms. Mr. Guttmann explains this in the case of gunpowders by the action of one of the minor ingredients in these explosives. I maintain, on the other hand, that in the main this difference is accounted for by the change in the physical nature of the base, and what Mr. Guttmann has told us has not changed my views. I believe that until these products are brought to practically the same condition as the base, and that the same test is applied, reliable and comparable results will never be obtained and injustice may be done, and remember we only want to find if the ingredients of these products were properly washed. The Home Office heat tests for nitrocellulose and nitroglycerin are well known, the materials are in certain physical conditions and any alteration which increases or decreases the exposed surfaces affect in an inverse ratio the time they stand any prescribed test. Thus, if the 20 grains of nitrocellulose were gelatinised and formed into 1 grain the time would be the longest, and between this and impalpable dust the time decreases step by step. It is therefore obvious that one particular heat test will never apply to all gunpowders. The grinding, until they pass a fairly large meshed sieve, is not sufficient, because the "chips" from grains of 0·100 in. thickness will be very different from "chips" stopped by the same sieve from grains of 0·010 in. thickness.

I have proposed that gelatinised guncotton preparations be ground in a mill, and only the dust that passes a sieve of 60 meshes to the inch be accepted for the test and that the test be exactly the same as that applied to guncotton. I have satisfied myself by innumerable experiments that such finely divided gelatinised nitrocellulose stands the heat test practically the same as the nitrocellulose from which the powder was made. I believe also for those gelatinised preparations that contain fairly large percentages of nitroglycerin but which are not plastic, the above test would be quite sufficient, but for gunpowders, like cordite, which when rubbed through a 60 mesh sieve are quite plastic this treatment increases the time, because, as a matter of fact, the exposed surfaces are reduced owing to the oily nature of the mass, and here, possibly, the blasting gelatin heat test would be the correct one. Except in the case of cordite it is entirely contrary to my experience with gelatinised compositions to find as Mr. Guttmann states that the coarsely-ground powders give worse tests than the finer. Furthermore, it is contrary to my experience to find that dry heat lessens the heat test; I have always found the opposite to be the case. From time to time we

have had occasion to keep loaded cartridges at temperatures varying from 120° F. to 170° F., and although the powder is practically in a hermetically closed case, yet the heat test improves as the time of heating is prolonged. In one instance cartridges were kept at 160° F. for 72 hours without intermission; and whereas the powder stood 26 and 23 minutes before heating, it stood 63 and 120 minutes after heating; many experiments of the same nature gave similar results. The difference is more rapid and more pronounced if the powder so heated is exposed to the air during heating. Although Mr. Guttmann's test does not seem so different from the official test as to necessitate any change, yet as a corroborative one it will be of great service to explosive manufacturers.

Allow me to correct one statement in the paper as to the composition of the various rifleites, and S.S., S.R., and S.V., which are said to be composed of nitrocellulose and nitrobenzene. As the manufacturer of these products I may state that nitrobenzene does not enter into the composition of any of these explosives.

Mr. O. Guttmann, in reply, said he had fully expected that his paper would draw upon himself and the proposed test some hostile criticism. Such matters were so controversial in their nature, and required so many experiments to appreciate their value, that he could not expect any other treatment. He did not claim that the proposed test was a perfect one, and could quite imagine that a better one would be found; but, so far, diphenylamine had proved itself superior to all others. Although Dr. Dupré suggested that his opinion was due to the natural enthusiasm of a discoverer, Dr. Dupré knew well that nobody was more critical than himself, and he was not likely, therefore, to be over-enthusiastic. Mr. Reid had asked that the bodies concerned should be divided into volatile and non-volatile constituents, and he, in common with another speaker, stated that castor oil and other oils would absorb nitric peroxide and not show it. He could only say that if those gentlemen would take any pure explosive, say guncotton or nitroglycerin, and sprinkle finely-divided castor oil upon it, they would find the heat test to go up. He was convinced that that fact was not due to the absorption through the oil itself. Mr. Blount had called attention to the elaidine reaction, and thought this showed conclusively that nitrogen peroxide was absorbed by the oil itself. He could only say that he had made many experiments, and did not get the elaidine reaction sufficiently quickly to prove that that was the case. To produce such a reaction took hours, and therefore the oil could not be the cause of the disappearance of iodine. Mr. Reid and Mr. Blount also held that the loss in weight of the castor oil was due to the volatilisation of water. In reply, he would point to the tests given in his paper, in which the castor oil was exposed for 72 hours, by which time the guncotton and the oil as well had lost all the water: yet, after all that drying, the castor oil would hide the heat test, and, in fact, would do so at any time. Mr. Blount asked for an explanation why alkalis decomposed nitro compounds. He would be happy to give that information, though probably Mr. Blount was well aware that Colonel Hess, in 1871, showed that with caustic alkalis reaction took place most violently. He also asked why the percentage of nitrogen and the degree of solubility were not sufficient to show the suitability of collodion cotton for making blasting gelatin. The reason was that the physical state of the jelly had to be considered, and the capacity of the collodion cotton for retaining the nitroglycerin in the form of a jelly. Mr. Sanford said he had found that cannonite stood the test for two or three hours, and could not understand how he (the author) had got only 13 minutes. A similar opinion was given by Dr. Dupré. He would point out, however, that it followed from his paper that the age of the powder and the state of division were important factors: with an old powder in a fine state of division and very dry it would be found that the solvent had gone, and so there was nothing to show a difference. Mr. Trench and others had spoken about explosives going to Australia. Seeing that cargoes worth thousands of pounds were destroyed every year because they failed to pass this test in Australia, it was obvious that the matter was a very serious one. All he could say was that a blasting gelatin that did not contain wood pulp would stand the heat test better than gelignite, which did contain it. It must be remembered that blasting gelatin is a tough substance from which nothing can come out of the interior; any decomposition would, therefore, show itself on the outer surface only in the first instance. It was different with dynamite No. 2, some of which was perfectly gelatinous, some very porous. He was not speaking only of explosives manufactured in England; such products were made everywhere; but the same tests were used for explosives wherever and however made; therefore, when he spoke of gelatin dynamite being not in a perfect jelly, he was not necessarily speaking only of explosives made in this country. Nitric peroxide was developed at low temperatures—at 45° C. already,—and if the dynamite were left for some days it would be found that at 50° C. it was gone—it would openly decompose. When, therefore, it was shipped to Australia in badly ventilated vessels, the heat would rise. Moreover, it had been found that explosives stored in hot places, such as Aden, blew up voluntarily, so that it was not only the filthy condition of ships that was to blame. Mr. Macnab stated that he got the reaction too soon. He thought the differences between the tests must be due to the imperfect character of the nitro-cotton. He knew Mr. Macnab's reputation for great accuracy, and therefore accepted his statement, but it did not agree with his own experience. Where erratic behaviour was the order of the day, experiments must differ in the hands of different men. He was glad that such an experienced man as Mr. Griffith could confirm, from his own knowledge, most of the statements he (the author) had put forth. And now he came to the most important contribution to the discussion, *viz.*, Dr. Dupré's remarks. It was difficult for him to reply to Dr. Dupré, because there was no man of greater experience than he on this subject. Ever since he had had to do with explosives—for nearly 25 years—he had been a disciple of Dr. Dupré, and he could quite understand that, as the father of the iodide heat test, he was loth to give way to a newer method. Still he was sure that Dr. Dupré would be the first to put aside a bad test if he was dissatisfied, and his object was to convince him that the iodide heat test was really not reliable. Dr. Dupré said he had never come across a complaint due to the heat test itself. That might be the case, but he must certainly have had many well-founded complaints which were based on it. Both Dr. Dupré and Mr. Blount remarked about the action of acetic acid. No doubt acetic acid does not combine with iodine, but it also cannot be said to affect nitro compounds. The fact is not yet quite cleared up, but it exists; and the acetic acid does not require days to show itself, but shows immediately, as proved in his paper. Let anyone take nitroglycerin of undoubted excellence, and add to it one drop of acetic acid, and down would go the heat test. Then Dr. Dupré said that he had never seen cordite or Maxim powder stand for 120 minutes. He could only reply that he had, and for longer; and the gentleman who had made the test with him was in the room. It was clear, therefore, that the test was absolutely erratic, and the differences in the tests were due to nothing but the fact that one powder was older and more finely ground than another. He was confident that Dr. Dupré would find that he was right, and that the iodide heat test was unreliable. With regard to Mr. Jones's remarks, he was surprised that Mr. Jones did not take the trouble to make a simple experiment, which would have satisfied him, in less time than it took to write his question, that acetone did hide the heat test very much indeed. Both he and Mr. Blount referred to certain powders in the table made with acetone, and which yet gave equal results with the two tests. He could only say that he had had these powders in his possession for 18 months, and they were probably quite that age when he got them, so there might have been no acetone left in them. At the same time it would be interesting to know how Mr. Jones determined the percentage of acetone, as there might be an error in this. The physical state of the explosive had not nearly so great an influence as Mr. Jones attributed to it. That powder in cartridges should give better heat tests after prolonged heating was contrary to experience. He knew that the powders made by Mr. Jones did not contain nitrobenzene, but, in view of an action pending in the courts, he thought it was near enough.

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The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Committee:* A. Smetham and Frank Tate.

SESSION 1896–97.

Wednesday, May 5th, 1897:—

Mr. E. G. Ballard, A.R.S.M. "The Volumetric Estimation of Zinc."

Mr. J. Kent Smith and Mr. J. W. Towers. "Apparatus for Gas Analysis."

Yorkshire Section.

Chairman: Thos. Fairley.
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SESSION 1896–97.

Manchester Section.

Rooms of the Chemical Club, Victoria Hotel.

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SESSION 1896–97.

Meeting held on Friday, March 5th, 1897.

MR. GEORGE E. DAVIS IN THE CHAIR.

ON PARANITRANILINE RED.

BY A. LIEBMANN, PH.D.

Previous publications have always been directed to the following points, viz., purity of the paranitraniline, the manner of its diazotation, and the preservation from decomposition of the β-naphthol on the fibre. I now propose to show what influence the presence of α-naphthol in the β-compound has on the brightness of the red, and to prove experimentally the limits within which α-naphthol may be present in β-naphthol if at all.

The important problem which confronted me at first in regard to the β-naphthols was as to whether and in what way a pure compound could be obtained from the article of commerce. Perhaps it will be best now to shortly state the results of my experiments, and to show by what line of investigations they have been obtained. In the first instance, I have prepared, as far as I know, by a new method, chemically pure β-naphthol from the commercial product. Secondly, I found new analytical tests, qualitative and quantitative, to prove the presence of α-naphthol in β-naphthol, even if present only to the extent of 0·01 per cent. Thirdly, I examined a series of commercial β-naphthols for this impurity. Fourthly, I studied the injurious influence of α-compound if present in the β-naphthol on the final colour; and, finally, I made some experiments to determine by which analytical methods the presence of meta- and orthonitraniline could be detected in paranitraniline. I have not finished my experiments with orthonitraniline, and I will leave this matter to further examination.

The preparation of pure β-naphthol was based on the following observations:—

I had studied the solubility of commercial β- and α-naphthol in toluene. On dissolving a weighed quantity of β-naphthol in a weighed quantity of toluene, and on cooling the solution thus obtained to about 7° to 8° C. for two hours, the whole of the liquid appeared to solidify to one mass of fine crystals, which were separated by filtration from the toluene. The filtered solution was then evaporated on the water-bath, and the solubility at this temperature of β-naphthol in toluene was found to be 1 per cent. A corresponding experiment, carried out with α-naphthol, proved its solubility to be 3·55 per cent.; but no notice is taken in this experiment of a certain loss of naphthol which is always sustained from evaporation of its toluene solution. A further difference between the two compounds as to their solubility in toluene, is that on addition of petroleum ether to the toluene solution, β-naphthol seems to be completely precipitated whilst the α-naphthol remains in solution on the addition of the petroleum ether to the toluene solution.

The purification of the commercial β-naphthol was now an easy matter—50 grms. of commercial β-naphthol were dissolved in 500 c.c. of toluene; the solution was allowed to cool; the precipitate was separated by means of a suction filter, washed with 100 c.c. of a mixture of equal quantities of toluene and petroleum ether; and finally washed with 50 c.c. of petroleum ether—40 grms. of β-naphthol were obtained, melting at 122° to 124° C. With this quantity of β-naphthol, the operation was repeated as before described, and the melting point of the repeatedly purified product was found to be now at from 122° to 123° C. A sample of this naphthol was recrystallised several times from hot water, with the addition of animal charcoal, but the melting point did not alter but remained at 122° to 123° C. This seemed to me not to indicate yet a completely chemically pure product, and another purification was resorted to; 5 grms. of the β-naphthol, which was obtained in the before described manner by purification with toluene and petroleum ether, were suspended in 50 c.c. of water, and were then distilled with steam. From the distillate it crystallised in beautiful white glittering flakes, which were filtered, dried, and repeatedly recrystallised from boiling water. Its melting

point was now 122° to 122·5 C. All the melting points were taken with a normal thermometer tested by the Imperial Physical Institute at Berlin.

To test the purity of the product thus obtained, I was thrown on my own resources, or rather I really arrived at the subject for which this investigation was undertaken. There is in literature no test described to prove the presence of α-naphthol in β-naphthol. I owe, however, to the kindness of Mr. Levinstein a description of the test now in vogue amongst industrial concerns interested in this product. It is as follows:—Dissolve, in a dry 200-c.c. flask, 2 grms. of β naphthol in 10 c.c. alcohol, and add 5 c.c. iron perchloride solution (50 grms. iron perchloride to 1 litre). After two minutes standing, add 40 c.c. water, when a precipitate is formed, shake this well up. If the sample is free from α-naphthol, the precipitate is white (with a greenish shade in the beginning) and must remain white. If the sample contains α-naphthol, the precipitate is at first slightly coloured, and, on standing, turns violet, till blue, according to the quantity of α-naphthol present. By this test the presence of 0·05 α-naphthol can easily be detected if the sample to be tested is allowed to stand a few hours. If the β-naphthol contain more than about 0·2 per cent. of α-, the precipitate formed is at once coloured violet, which on standing becomes deeper in shade. The depth of the violet colour produced when α-naphthol is present, if worked under exactly the same conditions, is proportionate to the quantity of α-naphthol present in the sample, and the method can therefore be used for quantitative tests, viz., by making mixtures of pure β-naphthol with known quantities of α-naphthol, and by comparing the shades produced with the sample to be tested.

Good as this test undoubtedly is, yet I venture to say that the new method which I am going to discuss is superior to it in every way, indicating even the presence of 0·01 per cent. of α-naphthol in β-naphthol, and being easily adapted for quick and reliable analysis. This test is based on the property of α-naphthol to combine readily and in presence of β-naphthol in preference to this compound with diazo-solutions, and to produce combinations with alkalis forming coloured solutions, whilst the corresponding bodies of β-naphthol are insoluble or nearly insoluble in alkalis. Amongst the diazo-compounds applied in this way diazo-benzene was not very successful, as the solution of the oxy-azo compounds in alkalis is not sufficiently pronounced.

A number of experiments have been carried out with this amine, but, as I said before, I soon threw diazo-benzene overboard, and turned to other diazo-compounds to achieve my purpose. Strange to say, the very compound for the benefit of which this research had been undertaken was the one which finally led me to overcome the difficulty, and yielded the most perfect results, viz., the diazo-compound of paranitraniline; and I will now at once give a description of the test, as I at last settled it, to determine the presence of α-naphthol in its isomeric compound.

Weigh 0·144 grm. of the β-naphthol to be tested, dissolve in a graduated test-tube in 5 c.c. pure alcohol, and make up to 15 c.c. with pure toluene. Dissolve 0·14 of paranitraniline in 9 c.c. of dilute hydrochloric acid, cool outside with ice, preferably mixed with common salt and diazotise with 1 c.c. of normal nitrite of soda solution. Now allow 1 c.c. of this diazo-compound, which equals 0·014 of diazotised paranitraniline, to run into the graduated test-tube containing the naphthol solution; shake well, add some water, and separate the two layers which form by means of a small separating funnel. Shake the toluene solution with 5 c.c. of normal caustic soda, and compare the colour of the alkaline solution with the alkaline solution obtained in exactly the same manner from a β-naphthol containing a known quantity of α-naphthol. By comparing two such solutions, the amount of α-naphthol in β-naphthol is readily determined.

My first idea was to prepare a scale of such coloured solutions, ranging from 0·01 per cent. up to 1 per cent.; but I found that the very dilute solutions containing from 0·01—0·1 of α-naphthol will decompose on being kept for some time, and therefore it is always advisable to make new tests side by side.

Of course, a great number of experiments had to be made before I determined on the method which I have before described, and I do not think that anything can be learnt from the more imperfect tests which resulted from these experiments. I should, however, like to mention one experiment which was rather surprising. I was under the impression (and I believe it is a general impression in literature) that β-naphthol does not combine with diazoparanitrobenzene in a hydrochloric acid solution. I have, however, observed that if a hydrochloric acid solution of paranitrodiazobenzene be added to an alcoholic solution of pure β-naphthol, a yellowish-orange precipitate is at once formed. This yellowish-orange precipitate changed as soon as I attempted a purification or even on standing a few seconds or on attempted filtration, into the well-known and beautiful red paranitrobenzene-azo-β-naphthol. This compound could only be either a hydrochloride of the latter body or a paranitrodiazohydroxynaphthalene, and I incline very strongly to take the latter view. Of such bodies there are only very few instances known, if any at all. I am informed by Prof. Meldola that he has described or made one of this class of compounds by acting with paranitrodiazobenzene or orthobromo-β-naphthol.

I have examined about half-a-dozen different products, and I find that, with the exception of one of them, they all contained more or less α-naphthol. The results are as follows:—One of the samples contained 0·3 per cent., another 0·1, the third 0·09, and the fourth 0·7 per cent. of it. One sample I found to be practically free from α-naphthol, containing only $\frac{1}{25}$ per cent. of it.

This investigation would not be complete unless I proved also the influence which these quantities of α-naphthol in β-naphthol have on the final paranitraniline red. The red was produced in the manner described by A. G. Green at one of the meetings of the Society of Dyers and Colourists in Manchester with the exception that I elected to take a stronger solution of β-naphthol, which means that I used a solution containing about 2 per cent. of β-naphthol. The result showed plainly that the presence of 0·25 of α-naphthol in β-naphthol distinctly exercises an injurious influence on the colour, and probably to an experienced eye even the quantity of 0·1 per cent. would be plainly visible.

I produced a series of dyed materials using pure β-naphthol and β-naphthols containing 0·1 per cent., 0·25, 0·5, 1, 2, 3, 4, and 5 per cent. of α-naphthol. In using β-naphthol containing less than 0·1 per cent. of α-naphthol the result was such that I could not detect any difference between the colours resulting from pure β-naphthol and such a product. If, however, the quantities are 1/10 and more of α-naphthol in β-naphthol, as I said before, the experienced eye will be able to detect the difference from the result obtained from the pure product. My attention was then directed to an examination of the commercial paranitranilines and I will here at once say that I found the commercial paranitranilines very pure products. I however succeeded in devising a method for estimating the meta-amidonitraniline in paranitraniline if 0·1 per cent. or more is contained in it. The reaction is based on the well-known fact that metanitraniline after reduction, produces metaphenylene-diamine, which, on treatment with nitrous acid, yields Bismarck brown, the dyestuff perhaps better known as Manchester brown.

As there are certain precautions to be observed in carrying out this experiment I will give a detailed description of it:—

0·25 part of paranitraniline are covered with water in a flask which is provided with a cork fitted with a glass tube and a piece of india-rubber with a slit closed at the end with a glass rod which allows gas to escape but not to enter. Some hydrochloric acid is added and some zinc dust and the flask is shaken, preferably with exclusion of light, until a colourless solution is obtained; it is then quickly filtered, and the solution is made up to 250 c.c. 0·025 parts of metanitraniline are reduced in exactly the same manner and also diluted to 250 c.c. If 10 c.c. paranitraniline solution are diluted to 50 and one or two drops of a weak solution of sodium nitrite are added, only a very slight change to a light yellow can be observed. If, however,

10 c.c. of the reduced paranitraniline solution are mixed with 0·1 c.c of the reduced metanitraniline solution and made up into 50 c.c., a light brown coloration will be produced on the addition of one or two drops of dilute sodium nitrite solution and this coloration will be all the more intense in proportion to the quantity of reduced metanitraniline present.

I intended to devise an analytical method for estimating the presence of orthonitraniline in the commercial paranitraniline by means of the property of the orthodiamido-compounds to form combinations with phenanthrene-quinone, but unfortunately I was, whilst in the middle of these experiments, called away from Manchester and was therefore unable to finish them in a satisfactory way, but I hope on some future occasion to lay before the Society satisfactory results in respect of such compounds also.

Now to recapitulate shortly the results of my investigations: I have discovered that the presence of α-naphthol in β-naphthol is deleterious to the production of a good paranitraniline red: I have further discovered a method which can be easily carried out for the estimation of α-naphthol in β-naphthol, and finally, a method for the estimation of metanitraniline in paranitraniline, for which latter method however, I found that there was unfortunately no necessity as commercial paranitranilines are practically chemically pure products.

I have to mention that I have been very ably assisted, in carrying out these experiments, by Dr. Albert Liebmann, to whom I am very much obliged for the careful and assiduous attention he has devoted thereto.

THE CAUSE OF LUMINOSITY OF HYDROCARBON FLAMES.

BY WILFRID IRWIN.

About 12 months ago I read a paper before this Section entitled "The Effect of Heat on the Illuminating Power of Coal-Gas and its relation to the Theory of Flame," in which I raised what appeared to me to be vital objections to Prof. V. B. Lewes's acetylene theory of luminosity (this Journal, 1896, 80). To this Prof. Lewes replies in a paper entitled "The Development of Light from Gas Flames," read at the late Gas Exhibition in New York, and published in the "Gas World" of Feb. 6th.

During the present century several theories relating to the luminosity of gas and hydrocarbon flames have been put forward.

I. The earliest assumed that when a hydrocarbon was burnt the oxygen of the air united with the hydrogen of the hydrocarbon, leaving solid particles of carbon at a temperature sufficiently high to account for the luminosity. This assumption was shown to be erroneous, from the fact that when hydrocarbons are exploded with oxygen, the latter always combines with the carbon first, and if there be an insufficiency of oxygen, free hydrogen always is left.

II. The theory of Frankland that luminosity is due to dense but transparent hydrocarbon vapours radiating light at a high temperature. The experiments mentioned in my last and to be alluded to presently tell against this theory.

III. That luminosity is due to the decomposition of dense hydrocarbon vapours, the carbon separating out in a solid form for an instant, and owing to the high temperature remitting light. This theory, as stated, fails to account for the whole of the light; as I have shown that hydrogen and smoke raised to a temperature higher than that of the hydrocarbon flame emits very little light.

IV. Prof. Lewes's acetylene theory, according to which the hydrocarbons in the flame are decomposed, or in the case of CH_4 condensed, to form acetylene C_2H_2, which, in its turn decomposes into C and H, the heat of decomposition raising the C liberated to a high temperature, acetylene being an endothermic body. Prof. Lewes has shown that acetylene will, when pure, decompose with evolution of light at 800° C., the temperature being thereby raised to 1,000° C.; also that when mixed with hydrogen a similar decomposition occurs, though at a higher temperature.

He has found that the gas at the top of the non-luminous part of the flame contains far more C_2H_2 than when it leaves the burner, owing to the decomposition of benzene C_6H_6 and other hydrocarbons in the gas.

When Prof. Lewes first laid this theory before the chemical world I must say that it appeared to me the most rational explanation of the cause of luminosity; for all experiments which I have made point to the fact—Frankland's opinion notwithstanding—that solid particles of carbon are necessary to the production of a bright luminous hydrocarbon flame. But I have shown that smoke and hydrogen heated to 1,400° C., a temperature above that at which luminosity commences, emits very little light compared with that from a gas flame.

I have also shown that in a 20-candle gas flame burning for 1 hour, not more than 2·4 grms. of carbon has been actually engaged in emitting light during the whole time. Certainly at any given period not more than 0·0001 grm. of carbon is so employed.

Now this extraordinary brilliancy is altogether out of comparison with anything obtainable from mere heated smoke, and the acetylene theory has the merit of offering another explanation for it. But there are several objections to it alluded to in my last paper which Prof. Lewes does not answer to my satisfaction.

I. I have shown that—

Benzene, C_6H_6, is a better enricher than acetylene, C_2H_2.

Xylene, $C_6H_4(CH_3)_2$, is a better enricher than benzene, C_6H_6.

Naphthalene, $C_{10}H_8$, is a better enricher than Xylene, $C_6H_4(CH_3)_2$.

Given that the acetylene theory is the correct one, one would naturally suppose that the above comparisons would be reversed, but Prof. Lewes says that the reason benzene is a better enricher than acetylene is because less of it is unchanged on its way up the flame, and that decomposition takes place just before the resultant acetylene is required for splitting up into its elements with the emission of light. On the other hand, he says acetylene polymerises to benzene, &c., but he does not show what harm that can do when such bodies are so easily split up again into acetylene just at the point they are required to do so.

Were it not that he assumes that benzene has changed definitely into acetylene to such an extent as to be estimated by analysis, before the decomposition of the latter, I should suppose, assuming his theory to be correct, its greater enriching power to be due to its power of yielding 3 mols. of acetylene, all close to one another, and therefore more concentrated and consequently able to decompose at a lower temperature than would otherwise have been the case.

If acetylene polymerises so readily, as Prof. Lewes assumes, and does not return to its original form, how does he know that it is really acetylene that he is dealing with when he sees it decompose in glass tubes heated to 800° C.?

You may remember where I showed that in passing gas through a heated tube, the illuminating power disappeared when the temperature reached 850° C. to 900° C., but without production of smoke, and that as the temperature increased to 1,400° C., smoke was formed. But that, on looking down the tube, neither at 850° C. nor at the higher temperature of more complete decomposition of the gas, was any glow visible.

This last observation, Prof. Lewes, in his summary of my paper, omits altogether, and charges me with misunderstanding his theory, because I make no reference to what is going on inside the tube. Prof. Lewes says that decomposition with production of luminosity is going on inside the heated tube, but this is what I could never discover; and this leads me to my second objection to his theory.

II. How is it that when passed through a heated tube, coal-gas loses its luminosity without any luminous glow in the tube being produced?

III. My third objection to his theory is that in a feebly-luminous flame, formed by burning acetylene and hydrogen, stratification occurs, as in other feebly-luminous hydrocarbon flames, showing, I think, that at least two different reactions occurring at different temperatures are taking place.

Prof. Lewes answers this by assuming that reactions take place which are very unlikely to occur, and which stultify other conclusions which he has arrived at in his experiments on the action of heat on mixtures of C_2H_2 and H.

The following experiment, which I have lately made, will be of interest in demonstrating my own latest ideas on luminosity.

A horizontal iron tube, 1 in. in width and 4 ft. long, corked at either end, was provided. By a small pipe just passing through one of the corks, a stream of coal-gas was admitted, whilst the temperature of the middle of the tube was gradually raised, by means of a furnace, until the gas issuing from a pipe through the other cork had lost its illuminating power. This latter cork was then withdrawn, and the interior of the tube examined from that end whilst the gas was passing. All that could be seen was that the pipe was red-hot where it had been heated, and neither smoke nor glow of any kind could be detected. The temperature was now raised almost to the melting point of wrought iron, still no glow was discernable, though smoke began to form, owing to complete decomposition of the hydrocarbons.

Next, by means of a small pipe reaching the heated part of the iron tube, a jet of air was introduced, when immediately a very bright glow made its appearance round the end of the jet, which attained its greatest degree of brightness when smoke ceased to be emitted from the open end.

This experiment shows that the presence not only of heat but of air is necessary to the formation of a luminous flame, firstly, in order to decompose the molecules of benzene, &c. before they can be all changed into acetylene; secondly, to combine with the freshly-liberated carbon, to form CO, with production of light.

My theory of the cause of luminosity, then, is as follows:—

The hydrocarbons are first condensed, to form bodies such as benzene, containing in their molecules carbon atoms in combination with very few H atoms.

That in this form they are attacked by oxygen in the lower part of the luminous band of the flame, and split up, yielding CO, free C, and CH_4, which latter passes to above what is the non-luminous band in feebly-luminous flames, where the heat is sufficient to again condense it as aforesaid.

That the free carbon, on being liberated, combines with more oxygen, to form CO, and in this reaction emits the intense light which is characteristic of the flame.

I have shown that the carbon, to possess this light-giving power, must have been recently precipitated in the flame; and if any material time elapses between the precipitation and combination with oxygen, very little luminosity is produced.

Meeting held on Friday, April 2nd, 1897.

MR. GEORGE E. DAVIS IN THE CHAIR.

ON THE ADULTERATION OF SILK BY WEIGHTING.

BY SIR THOMAS WARDLE, F.C.S., F.G.S., ETC., AND J. CARTER BELL, A.R.S.M., ETC.

The history and present practice of weighting silk presents some very interesting features, both from industrial and scientific aspects. I propose to speak of the industrial weighting of silk by chemical means, as well as of the chemistry of the subject, whilst to my friend Mr. Carter Bell (who with me, since he was appointed to the honorary office of chemist to the Silk Association, has given considerable attention to it) I shall leave the analytical part, promising you that his investigations will possess much interest, both in subject-matter and methods. I have to speak of deception the most refined; unspeakable fraudulent intent; the application of chemical science (from the modern rage for cheapness) to the opposite of economical purchasing; and a procedure and practice of adulteration now so universal in all continental silk centres as admittedly to threaten the industry with destruction, if much longer continued in its present excessive state.

If this paper can help forward the laudable effort now being made on the continent by the best manufacturers and dyers to expose the chicanery and sophistication of the excessively weighted and spurious fabrics miscalled silk, and mendaciously sold as such, its object will be attained. The treatment of my paper may be thus epitomised. 1. The history and development of silk adulteration. 2. The nature of silk and its adulterants. 3. The present state of silk weighting. 4. The simple detection of the presence of this sophistication. 5. And Mr. Carter Bell's more abstract treatment of the chemistry and analyses of silk so weighted.

The First Sinner.

If we must begin at the beginning, we have to select what was doubtless an object-lesson to the industry in the silkworm itself, as being the first silk loader, for amongst the countless millions of silkworms hatched into the world, not one of them has appeared without the power of weighting, or as the French say "charging" its silken fibre with at least 25 per cent. of weighting matter, and this in the shape of a substance, called in England, gum, and in France, grès, the composition and nature of which I will describe farther on.

This gummy substance, called in chemistry, sericine, is seriposited on the fibre at the moment the fibre itself is seriposited, from what are erroneously termed, the spinnarets of the silkworm, in the act of forming its cocoon. As is well known, the silk is never seen in its natural beauty and lustre, until this weighting matter or sericine has been removed by boiling soap baths or by the vapour of steam.

The instinctive object of the silkworm is apparent enough, namely, by a coating of varnish to protect the delicate fibre or bave which it seriposits, from climatic and other dangers, from and during the time it passes from the larval and pupal state, and until it emerges from its cocoon envelope as an imago or moth, then to begin to perpetuate its race.

Now if we can fix upon the silkworm as the first sinner in the idea of weighting silk, it is not surprising that this example should have been followed by those intent on gain. I mean, that the silkworm itself was the first to reveal the fact of a possible attachment of a substance to the fibre, foreign to the nature and properties of the fibre itself. It is true that this gummy attachment is almost, if not entirely, mechanical and not chemical; but it was in this way that the earliest dyers began to add weight, I won't say to the fibre of silk, because that is scarcely correct. I have just said that before the beauty of the silk is revealed and before its lustre can be manifested in fabrics or in the skein, this gum must be dissolved from the fibre; but a textile fabric consists of two parts, warp and weft. The weft being generally concealed between the warp threads, or so woven as to be seen only on the back of the fabric, need not have this gum removed, that is, need not be lustrous. I think the practice of weighting was wholly of European origin, for I have never been able to find any ancient silken stuffs of China, Japan, India, Persia, or even the coptic grave-cloths of the early Egyptian Christians, which were not bright both ways, or both warp and weft "boiled-off" as we dyers say, that is, the gum first discharged from the fibre.

I think the ancient dyer, no doubt, first began to weight the weft and in the gum. I don't know how ancient the practice was, history does not tell us, but it probably commenced in France through competition with the brocade weaving in Italy.

Here are some specimens of Italian and French brocades of the century, when the silk designing and colouring and dyeing of the rennaissance had reached almost the highest pitch of artistic excellence and of the artistic periods of Louis quatorze, quinze, and seize. Also a specimen of the period of the Empire when artistic decline had set in. All these silks are made of pure boiled-off silk, both warp and shute.

The specimens are—

Italian brocade of the 15th, 16th, and 17th centuries.
French brocade of Louis 14th, 15th, and 16th.
French brocade of the Empire about 1800.

The weft, better known as shute, when handed to the dyer by the manufacturer to be dyed, was accompanied by instruction from him to dye the warp with the gum removed, and the weft or shute with the gum left on the fibre. In this latter state it is generally termed, souple, or hard, and instead of the dyer returning it 12 ozs. per lb., as in the case of the warp, he returns the weft if unweighted, 15 to 16 ozs. to the lb., *i.e.*, close upon its original weight, thus having dyed both the gum and the silk.

Developments gradually crept in. It was found that by immersing the silk, whether boiled off or in the gum, in vegetable extracts containing tannic acid, an affinity of one towards the other was set up; a chemical union took place between the warp and the tannic acid, bringing up the silk to nearly its original weight before it was boiled off, or replacing nearly the 25 per cent. of gum it had lost in that process.

The case of the non-boiled-off shute or weft was different. The tannic acid had two missions, one to unite with the fibre, and the other to the gum, forming both a chemical and mechanical attachment; but its weight in this operation increased in proportion to the amount of tannin and the nature of the process generally, say, from 2 to 3 ozs. per lb. on the original undischarged weight. But something else occurred, and it is this which constitutes the chief reason for the adoption and recent developments in the processes for charging or weighting silk. Silk fabrics are not sold by weight, but by the yard, therefore it is obviously of but little use to weight the silk when sold by measure, merely for the purpose of increasing its specific gravity, unless in those cases where silk is sold by weight.

What does take place is that the silken fibre, in absorbing the tannic acid, expands, as a dry sponge does with water; the fibre thus becomes thickened, and generally in proportion to its amount of absorption. This rule has, however, exceptions, which need not be discussed here. This expansion of the fibre therefore renders it unnecessary to use as much silk as would be required if the silk were woven in the gum-discharged state.

The economy or cheapening by this property in silk, of increasing its bulk by absorption of chemical substances, lies in the fact that these substances and their application are all much less costly than silk, and, so long as the natural beauty of the silk can be preserved, modern demands for cheapening prove insatiable, as I shall presently show. The cheating, or cheapening, if you like, of the manufacturers of the latter half of the 17th century is innocent as compared with the relentless demand of the wholesale merchant of to-day, who is not content unless the fabric is cheapened to its utmost limit, and, consequently, adulterated to maxima truly alarming.

Early Weighting.

Originally, in fact, down probably to the 18th century, the weighting of silk was confined to the weft and upon the gum; but other chemicals, being found to possess affinities for silk, gradually came to be applied also to the warp, chiefly metallic salts having basic properties. First, alum, which always adds a little weight to silk, although only used as a mordant, then salts of iron, lead, and, lastly, tin, for which silk has a most surprising affinity. There are few subjects in modern chemistry which have occupied the attention and close study of chemists to a greater degree, in conjunction with dyers, than the creation of affinities in the processes of weighting silk; so much so now, that no dyehouse is complete without its laboratory, and no dyer's education is complete without a large command of the domain of both organic and inorganic chemistry, not so much for tinctorial purposes, but for swelling the fibre of silk by chemical means. Unhappily, the better chemist the dyer is, the more he is sought after by the manufacturer and his customer, the wholesale dealer, whose cry ever is cheaper, cheaper, and cheaper still, until at last silk is now manufactured into dress and other materials, the average maximum of adulteration of which is but little below 100 per cent., and often to a much higher degree.

As to maxima of weighting silk in the gum and boiled-off conditions, I will expose a few sophistications as examples:—

A piece of black ribbon, the warp of which is 24 ozs. per lb. on boiled-off silk, and the weft 76 ozs. per lb. on silk in the gum, or containing, respectively, 50 per cent. and 375 per cent. of adulteration.

Samples of coloured silk in the hank, weighted before dyeing in the boiled-off state respectively to 50 per cent., or 24 ozs. per lb.; also others weighted as heavily as 40 oz. per lb., or 150 per cent.; that is, each 12 oz. of silk containing 12 oz. and 18 oz. of weighting matter.

Samples of black silk in the skein, weighted on the undischarged silk to 100 per cent., or 32 oz., and 40 oz. per lb.

A sample of spun silk, that is, silk spun in short fibres or drafts from silk waste. It was termed cordonnet, and has been sold largely for fringe and tassel purposes. It was given to me by a Lyons dyer, who had weighted it to the incredible extent of 920 per cent., or 160 oz. per lb., or 144 oz. of weighting matter added to each 16 oz. of silk. It is sold at about 2*s*. 6*d*. per lb. in its present state, whereas the yarn in the grey cost 8*s*. per lb.

A sample, such as is used in the elastic web trade, in which the weighting has been carried on a boiled-off silk to 30 oz. per lb. Weighting in this branch of trade has had two unfortunate results, a greatly diminished use of elastic webs for boots, and the substitution of wool for silk, which wears better than silk when so heavily weighted.

Also a skein of black souple tram, weighted to 400 per cent., or 80 oz. per lb.; and samples of old Admiralty handkerchief silk, weighted before the reform, of which I shall speak presently, the warp 26 oz. dye on boiled-off silk, the shute 50 oz. souple.

The Cause of Decline of English Silk Industry.

The excessive weighting of silk is one of the causes of the decline of the English silk industry, because it is to Continental chemistry that the adoption and practice of weighting methods are due. Large fortunes have been made by Continental dyers or, as I ought more properly to say, weighters and by manufacturers by their skill in silk sophistication, and never so much as at the present moment is the weighting of silk a Continental industry.

English manufacturers have been obliged to send their silk abroad—not indeed to be dyed, for that is unnecessary, but to be weighted. English manufacturers and dyers, with a few exceptions, have been slow, as well as unwilling, to adopt the pernicious practice. It is sad to have to state that if the British industry is to be brought back it must be by British dyers and manufacturers weighting the silk exactly to the same extent as it is practised on the Continent, unless (which is more desirable) the large wholesale distributors of silk return to a purer state of trade. This, in the absence of a public demand for pure silk, does not, I am grieved to say, appear likely. As a case in point, one of the most important modistes in London, on my pointing out to her that the Paris silks she was making up were heavily weighted with tin, replied, Well, we must have the cheapest silks; why don't the English manufacturers put tin in theirs?

I do not find any mention of silk weighting in books on silk dyeing up to the middle of the 18th century, that is, before the time of such chemists as Macquer, Berthollet, Pileur d'Apligny, and Hellot. I have an Italian book of processes of the early part of the 16th century. Weighting does not there appear to have been dreamt of. Neither Pileur d'Apligny, who wrote in 1776, but on cotton dyeing, nor Hellet on wool dyeing in 1750, make any mention of substances used for weighting. But Macquer, who brought to a focus the state of silk dyeing in France of his time, in 1763 wrote his comprehensive and interesting contribution to those magnificent volumes issued by the French Academy, at the instance of the Government in that century, known as "Les Arts et Metiers." He entitled his work, "Art de la Teinture en Soie."

In the process of dyeing black on silk, Macquer describes the use of nutgalls and sumac, also antimony, litharge, plumbago, orpiment, corrosive sublimate, arsenic, sal ammonine, copperas, and sugar, all needlessly confused together in the same operation for dyeing black, accompanied by this remark: "For the black dye the silk is boiled as usual, having been washed and beetled according to custom; you then give the gall liquor for heavy blacks twice, but for light blacks only once. These two blacks are alike in beauty and shade, differing only in the weight of the silk; the light black has, however, rather more lustre"; and with regard to black souple or "black in the raw" (cru), he says, "the silk should be galled in a cold liquor of fresh galls."

These methods indicate a weighting on soft and souple, but of not more than 3 to 4 oz. per lb., which, although termed heavy blacks ("noirs pesants" as distinguished from "noir leger"), were innocent as compared with the present state of things.

Silk Weighting in Charles I.'s Time.

The advance of the silk industry in England under beneficial restrictions appears from a proclamation issued by King Charles I. about 1630; this proves that a quantity of raw silk was worked in this kingdom, sufficient to cause the issue of a proclamation, which stated as follows:—"Deceptions in the dyeing of silk have been discovered consisting of an addition to the dyeing mixture, by means of which it is intended to increase the weight of the silk, and owing to this frudulent combination not only is the dye inferior, but the silk itself also becomes tender. We therefore most strictly command that henceforth no silk dyer shall make use of, in the dyeing of silk, such injurious additions as, for instance, alder-back, iron-filings, or other fraudulent substances, and not only for black but for coloured silk; further, that no silk be dyed with other than 'Spanish black,' and the use of the so-called 'London black,' or what is called 'light weight,' is to be entirely avoided. Also, before the raw silk is dyed the gum must previously be altogether extracted by boiling."

Some years later the King partly withdrew the prohibition contained in his proclamation by allowing some silk to be dyed in the gum—that which is generally called 'hand silk, intended for rough taffeta, patterned Atlas, fine thin ribbons, and both black and coloured Floret or Ferret ribbons." The King, with commendable straightforwardness, gave us his reason for this retreat from his first pronouncement that he had since become better acquainted with the subject.

I have already stated that the older methods of weighting were upon silk with its gum not discharged. In this case the weighting matter added had the effect of combining with the gum, which surrounded the silk fibre as a thin bark does a branch. By weighting, this bark becomes swollen in proportion to the weight added. I will show you on the screen the actual appearance upon Italian silk several black souples, weighted from 16 oz. to 96 oz. per lb. You see the silk fibre remains of its original size, whilst the gum has been increased to a considerable thickness, and where it is broken off, because of its brittleness, it resembles bobbins on a thin rod, at intervals from each other, or a branch of a cork tree with its thick bark removed at intervals.

Now let me show you the difference on a fibre of silk weighted after the gum has been removed. This is an original fibre weighted to 150 per cent.; or 40 oz. per lb., and afterwards dyed. The first or gum-weighted silk is to some extent mechanical, and is easily rubbed off, whilst the second is a chemical incorporation of metallic salts with the fibroin of which silk is composed. The following formulæ of silk and gum will be of interest:—

The composition of raw silk or fibroin is—

Fibroin, Cramer.

Carbon	48·60
Hydrogen	6·40
Nitrogen	18·89
Oxygen	26·11
	100·00

giving the formula $C_{15}H_{23}N_5O_6$.

As to the composition of the sericin or gum, no two chemists agree in their analyses. Silk has been examined by Cramer, Mulder, Bolley, Schützenberger, Roard, Rosa, Vogel, Staedeler, Bourgeois, Persoz. Bolley shows the following as its composition:—

Sericin or Silk Gum.

Carbon	44·32
Hydrogen	6·18
Nitrogen	18·30
Oxygen	31·20
	100·00

giving the formula $C_{15}H_{25}N_5O_8$.

It approaches in composition very closely to albumen and fish-glue, as the following tables show:—

—	Egg Albumen.	Fish-Glue.
Carbon	53·40	50·10
Hydrogen	7·00	6·60
Nitrogen	15·70	18·30
Sulphur	0·40	25·00
Oxygen	23·50	
	100·00	100·00

I may here remark, in passing, the curious and unique property possessed by this insect-fibroin for uniting with inorganic salts, which materially add weight to it. It is not possessed by the fibre of cotton (which, as we know, is cellulose), by linen, or any other vegetable fibre, nor do animal fibres partake of the affinities possessed by fibroin. It is true wool unites with alum, chromic and other salts, as cotton with tannin and other mordants, but not in a weight-taking capacity. It is said it is possible to weight wool, but I have not met in commerce any wool so weighted. It may be safely said that woollen manufacturers are fortunately free practically from chemical weighting. If woollen fabrics require to be cheapened, recourse is had to mixing cotton with them; or, if cotton has to be weighted, mechanical sizing is used. How strange then that it is reserved for a product of the insect world to have a monopoly of combining in this way with inorganic salts and tannin.

The silk of spiders I have proved to possess the same property. I have found on the silk of a species of Indian spider as much as 50 per cent. of gum, which, after being discharged, I have dyed and weighted like the silk of Bombyx Mori. Here are several samples of spider silk, both raw and woven, from Madagascar, India, and other countries.

Gradual Inroad of Weighting Silks.

The gradual development of weighting silk, "in gum," as it is termed, by the employment at first simply of the tannic acid extracted from such vegetable matters as sumach, valonia, galls, catechu, the cutch plant, &c., and afterwards in the dyeing of blacks, a gradual increase of weighting by double mordanting with iron salts, notably persulphate of of iron, and ferrocyanide of potassium, which, combining with the iron on the silk, formed a base of Prussian blue, helping the formation of the tone of black, and enabling the tannin afterwards employed to combine in larger proportions with the silk, and so increase its weight, until the weighting, at first only amounting to an increase of 2 oz. per lb., has gradually risen in practice to 100 per cent., or 80 oz. per lb., on souples for textile goods, or even more, and 40 to 50 oz. on boiled-off silk.

Here is a German dyed sample of silk weighted to 400 per cent. You will notice that the silk possesses no lustre, but, as it would be covered by a warp which has lustre, that does not signify. This excessive weighting swells up the silk, or rather the gum, and pads the fabric like stuffing a cushion, giving it fictitious feel, appearance, and substance.

An excellent example of this occurred about 10 years ago in the black silk neckerchiefs supplied to the sailors by the Admiralty. Jack is privileged to have a new black neckerchief annually, but for which, however, he has to pay.

The Admiralty, ordering the goods by tender, generally accepted the lowest, and, not knowing the nefarious ways of contractors, and not being patriotic, gradually became their victims, until the weighting of the silk of which the neckerchiefs were made became so abnormal that even Jack rebelled; for after a day's wear in summer he came home to his wife with a neck as black as his kerchief.

Several years ago the Director of Admiralty Contracts consulted me on the subject; he said things had got to such a pass that Jack threatened to refuse to wear his neckerchief. I said, "The fault is yours for accepting the lowest tender and encouraging foreign competition," either the goods having been made in Germany, or the English manufacturers having no resource but to order their silks, warp and weft, to be weighted, but generally sending their silk in the skein to Germany to be so weighted and dyed. Here are specimens, the warp a 26-oz. boiled-off black, *i.e.*, 12 oz. of silk weighted to 26 oz., or 14 oz. of increase on 12 oz., and the weft weighted on the gum to 50 oz., or 300 per cent.; no wonder the black weighting matter rubbed into Jack's neck. I at once suggested unweighted silk, but the Director of Contracts, in fear of too great an increase in the annual estimates, not daring to accept my recommendation not to weight the silk at all, I recommended a 16-oz. dye for the warp and a 20-oz. dye for the weft. These dyes were adopted, and the following words were at once inserted in the Admiralty specifications:—"The dye of the warp to be of a sound, permanent black on boiled-off silk, and not to exceed 16 to 17 oz. per lb.; and that of the shute to be a boiled-off black, not to exceed 20 oz. per lb. An inspecting officer shall be permitted to see both the spinning of the yarn and the manufacture of the handkerchiefs to be supplied."

There was then no chance of escape, for I gave, by request, to the Admiralty chemist methods for analysing the weighting of both warp and shute, and the standard has been observed by the manufacturer down to the present time, with two distinct and surprising results: first, that a handkerchief after a year's wear is now looked forward to by Jack's wife as none the worse, whereas under the old régime they were worn out often before the year had passed; second (and this is singular and interesting), that the handkerchiefs since the reform have been all made in England—pointing to this, that silk can be made as cheaply here as abroad, providing the weighting is forbidden, or where equal conditions are observed.

The History of the Weighting of Boiled-off Silks.

In course of time, and competitive trade—which, I take it, meant money-making for its own sake, versus the making of artistic goods for the sake of the pleasure thereof and reputation—the weighing of boiled-off silk began, as I have said, with tannic acid to the extent of 15 to 20 per cent. its limit by this method; then sugar weighting (a purely mechanical operation) came in the latter part of the last century, and stayed until more chemical methods were discovered.

A syrupy solution of sugar can be added as a coating to silk fibre to nearly the extent of tannic acid, sometimes employed on the top of tannin-weighted silk in the case of souples where lustre is not the desideratum. But sugar, being deliquescent, subjects the silk to several fatal objections: destruction of silky feel; substituting for the natural silky touch, a clammy feel; silk so weighted is also liable to mildew and other fungoid growths; also it is easily detected when present by its sweet taste. Its use is now practically abandoned on the Continent and almost so in England. It is at best a clumsy artifice. I need not mention the many chemicals which have been from time to time experimented with, like barium salt for example, which from its high specific gravity, was once thought to be a great find, but like several others only introduced to be abandoned.

I will at once come to the modern methods of adulteration, the subject of Tin weighting, stannous chloride, $SnCl_2$, also known as tinsalt, tin crystals, muriate of tin. This salt has for a long time been used in the dyehouse as a mordant for colouring matters, chiefly for scarlets and crimsons from cochineal, and for violets and reds from dye-woods, notably logwood and Brazil woods. Various forms of solution of tin were employed, according to the ideas of both dyer and manufacturing chemist, but they may be classed, for the purpose of this paper, under the generic term of tinsalt or muriate of tin.

About 40 years ago, a blue called Napoleon blue was for several years the prevailing fashionable blue, being less cold in tone than indigo. It was dyed in the following way, being, in fact, a modified form of Prussian blue. In one bath was a solution of tinsalt and ferricyanide of potassium, and in another a solution of muriate of iron. The silk was worked in these baths alternately, and a rich blue was the result, the tinsalt being used for mordanting with no intention of silk weighting, although a little weighting resulted. The reason I mention this is to show how tinsalt began to be employed in the dyeing of black silks for weighting purposes. It was a discovery of a lamentable nature and purely accidental. It has formed the groundwork of all the subsequent discoveries, not accidental, of inordinate and abnormal silk weighting.

A well-known dyer at Crefeld about 40 years ago, whom I knew personally, was occupied chiefly in dyeing Napoleon blue and ordinary 16-oz. blacks on boiled-off silks. One day one of the men working in the blue-dyeing department, larking with another close at hand in the black department, threw at him a large hank of silk which he had just been wringing out of the tinsalt bath. The silk missed its mark and fell into a bath of catechu ready for being used to weight some silk for black. It was of course ruined for blue, turning at once green; and the men, to conceal knowledge of the lark, allowed the hank to pass along with the parcel for black. When the silk was dried at the end of its processes, a hank was found in the parcel more bulky and heavier than the rest, and considerably brighter and bluer in shade. This was enquired into and found to be the one missing from the blue parcel. A small parcel of silk was then put to the test of experiment, being mordanted with the tinsalt as for Napoleon blue, and it was found to result in an improved colour and increase of from 12 oz. of boiled-off silk to 18 oz., or a return of 18 oz. per lb. This accidental discovery caused a great sensation in the silk-dyeing world, and I remember going over to Crefeld to consult my friend Mr. Adolf Kottgen, a silk dyer there, since dead, on the subject. He told me the story I have stated, and when I visited him, he was dyeing large quantities of silk black in this weighted way.

Shortly after, the French dyers took up the process, and by their cleverness and ability in dyeing soon increased the weighting to much heavier degrees. The way was led, at first, singularly enough, by a French dyer in London, named Drevon, who, finding not much room for his genius in this direction in England, the English manufacturers being adverse to the introduction of such sophistication, went to Lyons, and in a short time became one of the largest dyers there. His increment of addition did not exceed 26 to 30 oz. per lb. on boiled-off silk; but since his time, now 20 years ago, the Lyons, Swiss, and German dyers have discovered methods to enable them to carry into very extensive practice the weighting of black silk to at least 40 oz. per lb.; that is, 12 oz. pure silk made to return in black 40 oz. without in any way impairing the lustre of the silk or its solidity of colour. Here is an example. In fact, we have the curious phenomenon of weighted black silks being more permanent in colour than unweighted ones, mainly from the tinsalt forming both mordant and weighting media. With time and chemical development, another important change was introduced. Unavailing were the efforts to give weight to silk by immersion in tinsalt solution alone, without the intervention of tannic acid or catechu tannin. Stannous chloride, although an excellent mordant, has in itself little or no practical weight-giving property. It combines with fibroin very much as alum does, but, unlike alum, in the presence of tannin, especially the Catechu tannin obtained from Catechu, affinities between the fibroin, tinsalt, and tannin become quickened, with a result of a considerable expansion of fibre and increase of weight. These affinities are increased by the previous treatment of the silk with persulphate of iron and ferrocyanide of potassium, without which good

blacks or sufficiently weighted could not be obtained. Of course, these processes were inapplicable to the weighting of silk intended to be dyed into colours, because of the groundwork of first the iron salt, then the ferrocyanide of potassium, which gave a dark blue, and afterwards the tin and tannin, which left the silk so dark in shade and so dirty a green, that to dye it into colours was of course out of the question.

The difficulty of giving abnormal weight to fibroin was at length solved by the discovery that a strong solution of Pinksalt gave the desired result without imparting any colour to the silk, and made it equally applicable to silk in the gum or boiled-off. Here was the commencement, more than 10 years ago, of a new era in silk weighting by the introduction of pinksalt, which, as you know, is a double chloride of tin and ammonia, having the formula $SnCl_4 2NH_4Cl$. By passing the silk before being dyed into a solution of pinksalt, and then into a soap bath, the silk increased in bulk and weight, according to the number of repetitions of these processes, but not equally. I find the bulk does not increase so much after the silk has increased to 100 per cent., or 12 oz. of boiled-off silk to 24 oz. After these operations the silk can be dyed any colour, or white, without much difficulty. This process was found to be too costly, and other developments caused a cheapening.

The price charged at first was 6*d*. per ounce for each ounce of increase of weight. At the present time the German dyers are engaged in a fierce price-competition, and the cost has been reduced one half. The methods by which this has been accomplished are very interesting, and show the importance of a profound acquisition of chemical science in any dyer intending to maintain his position in his trade.

After the silk had received its tin it was soaped. The effect of this was to precipitate the tin into the fibre, probably as an oleate of tin. Many chemicals were searched for and tried as substitutes for soap, and at length phosphate of soda was introduced and found to do its work better. But this change did not sufficiently lessen the cost, and at length the Germans introduced silicate of soda as an additional process after the phosphate of soda. This caused the tin to combine with the fibroin with leaps and bounds, until now, as the latest achievement, 167 per cent. in colours, or 53 oz. per lb., has been reached, as you see here, in the samples recently sent me by one of the largest Crefeld dyers. To the unassisted eye, or to the touch and taste, it is scarcely possible for even an expert to tell that the silk is weighted at all. The weighting matter (unlike sugar) is in direct chemical union with the fibroin, and is part and parcel of its substance, becoming a new and another product. The defect inherent to this method of weighting is, that by exposure to light, especially in sunlight, decomposition sets in, and the fabric soon becomes rotten, and what at first seems a cheap dress is soon useless.

Although I have described the *rationale* of this method of causing tin to combine with the silk, it is only right for me to say that I have only stated the broad lines on which the methods are based; developments occurring as each month succeeds another, having for their results lessened cost, the modification of former defects, and the discovery and use of other salts which quicken the affinities of the metals with the fibre, none of which, however, are yet, and it is hoped never may be stable. (See page 326.)

I will now show you the comparative results of burning pure and adulterated silk after the manner of the incandescent light, making a similarly shaped mantle. There would be much incandescence with the weighted silk if the heat were great enough. The tin glows like lanthania and other rare earths, but it will be seen how indestructible the weighted mantle is and how immediately the pure silk is consumed.

Reasons for Weighting Silk.

A few words on the reasons for weighting silk. Two causes only, in my opinion, have led to this disastrous state of things: the insatiable pressure of the merchant's buyer for cheapness, and the baneful and degenerate spirit of commercial competition amongst the manufacturers themselves, which in too many cases has degraded itself into mere underselling without due regard to, or, in too many instances, knowledge of the differential and integral totality which constitutes the cost of production and the necessary margin of profit.

With regard to the first of these two causes, I may illustrate the truth of what I state by what I was told by one of the most important of the Lyons manufacturers—one of my colleagues at the Paris Exhibition of 1889. He said the English buyers who periodically came to Lyons had become very pressing in the matter of price. He said they went to Crefeld and got quotations, with which they squeezed the Zurich manufacturers, and then came down to Lyons saying, we can buy these goods at such and such prices at Crefeld and Zurich, and unless you are prepared to compete we cannot place our orders with you. So much so was this the case that my informant told me there were several English buyers whom they preferred never to call on them. No wonder, then, that under this severe pressure hidden means are taken to cheapen the fibre artificially by extending it by chemical methods and so economising the employment of silk.

To such an extent is this now practised at each of the three great silk centres I have mentioned, that the very existence of their staple industry is most seriously threatened, and great alarm occasioned amongst the manufacturers and dyers themselves, the latter being the first to be roused by the danger.

At a meeting held in Crefeld of the dyers of that town, a memorial was drawn up asking the Chamber of Commerce to make an enquiry into the excessive adulteration they were compelled to apply to the silk sent them to be dyed and weighted by the manufacturers, requesting them to call a conference of manufacturers and dyers with a view to fix a maximum limit to this scourge; and further giving notice in this memorial that they, the dyers, would not be responsible for the decomposition and rottenness of the fabrics so adulterated, and would refuse to weight the silk to a higher degree than 50 per cent., or 24 oz. to the lb., which on boiled-off silk means 12 oz. of pure silk weighted to 24 ozs. Their report, addressed to the Crefeld Chamber of Commerce, runs as follows:—

"The undersigned dyers beg to bring the following report conjointly with the meeting of silk merchants, makers of silk stuffs, and silk dyers, held by them on the 22nd December 1896, to the knowledge of the Chamber of Commerce, and to all those whose interests are bound up or closely connected with the successes of the silk trade."

"On account of the disappointing results which the silk industry has had to witness during the past year concerning the solidity of dyed and very heavily filled stuffs, a speedy change is urgently required, if the general desire is not to endanger the existence of the whole market. The absolute necessity for a remedy to produce a better state of things, has caused the undersigned dyers to call the general attention to the following points:—

"Until the introduction of the phosphate silicate method all makers used the so-called 'charge mixte,' and that to a very limited extent only as regards its strength. Makers did not go beyond 40 per cent. to 50 per cent. The apparent insufficiency of this mixture was, for a long time, passed over by the buyers. The effect of this insufficiency was observable when dyeing light and dark shades causing the self-same quality of cloth, taken for both shades to have the appearance of two different qualities; further, that this so-called mixture in a greater strength did not produce the strong and desirable grip or feel in the goods; and, still further, that when dyeing repeat shipping orders, very often a great difference appeared as regards this grip, and this without the least fault on the part of the dyers. It is also a fact that the solidity of light tints remains far behind in comparison with that of dark tints. To overcome this want of grip and feel, the phosphate-silicate method is used. This method has been condemned as producing unresisting dyes, but this is not justified.

"The unresisting nature of dyes showed itself simply on account of the overcharge of mixture used; and the following comparisons are instructive. It is a fact that, by applying the phosphate-silicate method the silk thread swells enormously, and it can be taken for granted that by

using, for instance, mixtures 40 to 50 per cent. above par, the thread gets stronger and more grippy than when using 20 to 30 per cent. of the phosphate-silicate charge.

"However, we do not pretend to say that when using the phosphate-silicate method the swelling is doubled; that when applying the 'charge mixte,' the fact remains that it comes very near that proportion. This shows that when applying this 'charge mixte' to all those stuffs for which, up till the present day, 80 per cent. of the Ph. S. method is still used; and it is here apparent that, in proportion, such an addition is too great. On account of this overstepping of limits lies the danger and the chief cause of the brittleness shown by dyed stuffs; it is necessary to remember that as soon as the quantity added of the 'C.M.' is out of proportion to the silk weight, the consequence must be the destruction and breaking of threads. The dyer cannot fix these proportions, because it is of great importance for him to know where the raw silk came from and how it has to be woven. It has been shown, for instance, that of one lot of weft, part was used for weaving 'surah' (or rather long reeded-stuff), which proved sufficiently strong as to wear and tear, whereas the other part of that same lot, used for weaving a so-called 'rustling taffeta,' came up in a brittle and broken state. Further, it has been proved that even heavier weighting used for weaving surahs brought them out sufficiently strong, whereas less weighting showed them to come up brittle—a proof, this, that the manufacturer must step in here and try to succeed in fixing the proportion of weighting necessary to employ for each article. A great factor in the effect is light. The metallic powders contained in the threads follow physical laws when they are extended or contracted. The thread itself has, of course, to follow these changes. If too much metallic matter is contained in a thin thread, it will be forced to break without any chemical process going on besides. For this reason, the maker should be asked to use only a limited amount of weighting for certain tissues.

"But this is not so easily done, on account of the different processes adopted, and the maker must try to find out what is the right thing to do in all these different cases. Should the manufacturers content themselves in future, not to insist in getting better results than formerly obtained with the adding of 40 per cent. to 50 per cent. of 'C.M.,' then it would be preferable to go on with the 'Ph. S. method,' and not to introduce again the 'C.M.,' for reasons above mentioned. It would also have to be taken in account that the 'C.M.' is, firstly, a dear process; and, secondly, it would have to be used in larger quantities, which, of itself, would in consequence cause an increase of cost. For the establishment of a healthy market generally, the use of heavy threads from good spinners combined with lighter weighting would have to be the rule.

"The undersigned dyers therefore have decided not to execute any orders in future where the weighting material is asked to exceed 50 to 60 per cent.

"The makers will, on the other hand, receive the thanks of the various trades if they, as mentioned above, use more of the better and stronger silks, and less of sizing material.

"As, firstly, dyers cannot lay down a hard-and-fast rule in that direction; and, secondly, it being a fact that ready-made goods suffered by being heavily sized, against which the dyers had no remedy, although the silk used was originally of good quality; and as, thirdly, the buying parties are generally inclined to ask for a certain guarantee from the manufacturers, and these demands being, in the end, thrown upon the dyer, who, naturally, is not in a position to give such guarantee—then the consequence must be that the undersigned dyers must feel themselves compelled to decline giving such guarantee for goods when manufactured.

"Leaving aside the pecuniary guarantee, it must be clear that the dyers are not in a position to give a moral one; but they are persuaded that, by the introduction of the above-mentioned methods, general confidence will return within a short period as regards the position of the silk market.

"The annexed treaty is recommended to the Swiss dyers for their special consideration. The meetings of the Swiss dyers have already had the effect of bringing forward special recommendations, as regards weighting systems, but these as yet have not gone deep enough into the subject, to our thinking, as there is especially a want of comparative methods used for producing strong and effective results for putting weight into goods, such as 'C.M.,' and for heavy black weighting.

"According to natural laws, any methods to produce heaviness in silkstuffs by artificial means must injure and reduce the strength and durability of the silk thread. The question only is, how far one can go, and which method can be proved to be the right one. Up till now, only a deteriorating effect on all points has shown itself.

"It is a well-known fact that silk goods well covered up, or kept in well-closed boxes, have shown their faults in the same degree as freshly-made goods for robes, which proves clearly that this result is not only caused by the influence of light rays, but is also caused by other still unknown influences which play a part in the deterioration of silk-stuffs. To find within a short period an unfailing remedy to produce a harmless and useful weighting method would form a highly important theme for a scientific treatise. As the manufacturers and the buyers must have the same interests at heart as the dyer, the undersigned believe that it is the duty of the Chamber of Commerce to cause such scientific treatise to be written and distributed. Finally, requesting the calling of an early meeting for an exhaustive exchange of opinion on this matter, we remain," &c.

[*Here follow the signatures.*]

The Zürich manufacturers and dyers, taking fright, met; and it appears, from a proclamation which appeared in the papers a fortnight ago, they have signed a convention, with heavy penalties for infringement, the clauses of which are as follows:—

Proceedings towards the Attainment of Uniformity between Silk Manufacturers and Dyers regarding the Weighting of Boiled-off Colours on Silk.

A convention has been appointed for the purpose, in the interest of the trade. The terms agreed upon are as follows:—

"1. The undersigned firms of dyers bind themselves from the 1st April 1897 not to weight boiled-off colours under any circumstances, and for whomever it may concern, more than 60 to 80 per cent. for tram, 20 to 30 per cent. for organzine, excepting seal-brown for lining stuffs.

"2. These maximum weightings shall undergo a further reduction to 50—60 per cent. for tram, *viz.*, (*a*) for white, (*b*) for colours, in case the tram is intended for taffetas.

"3. The undersigned silk manufacturers on their part bind themselves not to have any heavier weightings produced, not even by local or foreign dyers, whether members of the conference or not.

"4. It shall be declared with every order the uses of the coloured silk, whether the silk is intended for taffetas or other textures, and, in case of a dispute, the manufacturer has to prove that the silk in question has in reality been used for the indicated purpose.

"For every disposition of colours, the dyer has to be supplied with the correct titres and particulars.

"5. There will be a committee chosen whose task it will be to watch that the agreement will be rigidly carried out by both parties. This committee will consist of three confidential persons, the first of whom to be elected by the manufacturers, the second by the dyers; the director of the Silk Conditioning House for the time being is to be the third member. In cases where preventive measures have to be adopted, two additional members are named, one a manufacturer and one a dyer. The director of the Silk Conditioning House will be salaried with 500 to 1,000 francs per annum, according to the claims upon his time. The committee is pledged to the utmost discretion.

"6. Each of the undersigned dyers pledges himself to a security, the amount of which will be fixed in each separate case by the committee. The securities will be deposited by

the committee in the archives of the valuable writings of the Swiss Institute of Credit. For the withdrawal of them, all the three signatures will be required. The security will be wholly or partly forfeited when the majority of the committee find that the present agreement has been broken by the dyer in question. The disposal of the amount will be in favour of the Institute of the Silk Industry School of weaving, and textile drawing school; and will be decided upon by the majority of committeemen.

"7. Manufacturers acting in contravention to this agreement are subject to a fine of ten times the amount of the cost of dyeing.

"8. The circulars of the dyers issued January 18th and February 10th, as well as that of the silk manufacturers dated February 1st of this year, are withdrawn, concerning the responsibility for their method of dyeing, and matters remain as before. In disputed cases the committee shall decide.

"9. Exhaustive investigations are to be at once undertaken, based on scientific and practical foundations, as well as with regard to the different methods of weighting, and their effect on the silk, as concerning the thickness, strength, durability, &c. of the textures. The expenses incurred thereby are to be borne in equal shares by the dyers and manufacturers, as well as the other expenses of the committee.

"10. This agreement to be in force for the term of one year, *viz.*, until March 31, 1898, and, in case there shall be no notice given to the contrary by January 1st at the least by either side, it will continue to be valid for another year."

Notwithstanding this bane of weighting silk, there are several English houses which, on principle, offer to the public only pure and unadulterated silks. It is not a little singular and encouraging that these houses are doing, not only the best trade, but export to France to customers who are content to pay an extra price for a pure article. Of the great majority of British manufacturers, I know that they have their silks weighted only to the most moderate degree that foreign competition permits, and in so doing act unwillingly.

SILK ADULTERATION.

BY J. CARTER BELL, A.R.S.M., ETC., COUNTY ANALYST.

During the last few years I have worked with Sir Thomas Wardle on this question: "The Adulteration of Silk," and perhaps it will not be out of place if I give a short account of the analysis of the various samples. The question is to ascertain the real amount of silk in these heavily weighted samples: to attempt to estimate all the weighting materials would be to court failure, therefore it is better to use various solvents which will not act upon the silk. If we analyse the raw silk we shall find that it contains about 25 per cent. of a gummy substance, which must be removed before dyeing operations can take place. This, I may say, is not always the case, for just within the last few days I have analysed a sample of Japan silk, which had evidently been dyed in the raw state, that is, with the gummy matter left in the silk, for on estimating the moisture and the amount soluble in water there was a loss of over 25 per cent., the ash being as low as 0·464. The ash of a pure white silk was 0·349. While a Chinese salmon-coloured silk only contained 0·246 of ash. Therefore it is plainly seen that the Chinese and the Japanese have not yet learnt the fraudulent art of selling mineral matter for silk; it has been left to the French, Germans, and Swiss to carry this to perfection, and the heaviest weighted silk I have as yet examined is where 100 pounds of silk were sent to the dyer with a request that it should be made into 1,000 pounds; this particular silk gave 43 per cent. of ash, there was 9 per cent. of moisture, and the amount of nitrogen was under 2 per cent.: this would give less than 10 per cent. of silk in the sample. The only advantage I can see in such a compound (for it is a misnomer to call it silk) is to make an incombustible dress for a lady, for it would be an impossibility for her to be burnt to death in such a dress. When such a compound is raised to a high temperature it does not flare up, but simply smoulders away and leaves the original form intact, and in many cases with a beautiful silky lustre. This compound is not a tin-weighted silk.

In the analyses of these weighted compounds it is better to take the moisture from using 5 grms. of silk, then to extract with ether, alcohol, and water weighing between each solvent. This will give the quantities of soap, oil, sugar, and such like compounds added for weighting purposes. Half of the sample may now be treated with hydrochloric acid diluted with one or two volumes of water; the silk is then dried at about 230° F. It can then be reduced to powder, which can be mixed with the soda lime and the combustion for nitrogen carried out in the usual way. One of the metals which is largely used for weighting silks is tin; this can be detected by heating the silk with strong hydrochloric acid, added in quantity to effect solution, then diluted with water and a current of H_2S passed; the tin is precipitated, and may be recognised by the usual tests.

As I have already said, silk weighted to 800 and 900 per cent. is not easy to burn; but certain black silks which have been weighted up to 300 or 400 per cent. have been known to take fire spontaneously, and for dyeing blacks the substances usually employed are catechu, gall nuts, and certain salts, specially sulphates of iron, basic and non-basic. A silk of this dyeing was found to contain over 20 per cent. of moisture and from 12 to 15 per cent. of mineral matter. When dried at about 230° F. it took fire spontaneously as soon as air got free access to it. This may be owing to the rapid absorption of moisture, during which oxidation occurs. It is not so many years since that a fire broke out in the warehouse of a large silk mercer in Paris. The fire was soon discovered, and this gave rise to the discovery that it originated inside a large parcel of black dyed silk which had been returned from the dye-house only 24 hours previously. I believe that it is a well-known fact that black silk, under certain conditions, is liable to spontaneous combustion, but the real explanation of the cause is yet to be discovered. It is very advisable not to keep large quantities of black dyed silk together, and also to prevent it getting very dry, and above all, care should be taken to prevent it being exposed to any great degree of heat. In 1871 a fire occurred in a silk-dyeing establishment in America, and was traced to the heavily weighted goods, and it is considered unsafe to pack them in deep boxes for transportation.

Astringent matter like tannin, which is used for blacks and greys, and catechu which is used for brown, in combination with sulphate of iron, take up oxygen very readily, and it only needs a little oily matter with heat to have the whole mass smouldering to ashes. I have never myself been able to obtain actual flame from these experimental spontaneous combustions, but the mass, as a rule, smoulders away to ashes.

Some think that silk waste and silk rags are as dangerous in regard to spontaneous combustion as cotton waste. This question of heavily weighted silks is worth the attention of the insurance companies.

The ash of a genuine silk may vary according to the district in which it is produced, but I think one may take as an average 0·5 per cent.; in the raw state the ash varies very much. Some years since a new silk was introduced into Europe from China, a native worm feeding on oak leaves instead of mulberry, it was called the "Tussur silk," it was found to contain over 8 per cent. of ash; while good Italian silk only contains about 1 per cent. But when this Tussur silk is first treated with some alcohol, next with dilute H_2SO_4 and then washed in a soap bath, the quantity of ash is brought down to about 0·5 per cent. I believe one of the reasons why this silk is not more widely known is, that it has a less affinity for mordants than the ordinary Italian silk.

In estimating the real amount of silk in a sample I prefer to estimate the nitrogen, either by soda lime or Kjeldahl's process, and I prefer the latter with certain modifications of Mr. Stock.

The following are a few analyses of weighted and unweighted silks :—

——	Moisture.	Ash.	Silk.
	Per Cent.	Per Cent.	Per Cent.
1. White silk	5·1	0·349	100
2. Salmon-coloured Shanghai	5·5	0·246	100
3. Black	7·9	18·7	25
4. White	8·2	50·0	25
5. Shot silk	6·2	34·7	39
6. —	10·2	33·7	32
7. Pink silk	8·5	43·2	54
8. „	9·4	52·4	43
9. „	8·1	49·5	46
10. Blue	6·5	35·4	60
11. Gold colour	5·8	41·7	52
12. Cream colour	9·1	45·7	46
13. Pink and green	8·6	34·2	50
14. Pink	8·6	28·2	60
15. Cream	9·3	8·9	87
16. Yellow	8·6	9·1	89
17. Black	8·5	42·9	12
18. Sky Blue	9·1	48·9	37
19.	8·1	45·9	36

The standard for silk, which I have taken in these analyses, is what is called pure boiled-off silk and dried at the ordinary temperature; such silk as this will contain about 18 per cent. of nitrogen.

Tussah silk, which one may say is a child of Sir Thomas Wardle's, as he has done so much work to introduce it into this country, differs a little in composition from the ordinary silk. Tussah silk may be easily distinguished from mulberry silk by the action of zinc chloride. Tussah does not dissolve so readily as mulberry silk, which dissolves at 45° B., while for tussah it has to be at least 60° B. In case of a mixture of the two silks, zinc chloride at 45° B. will dissolve the mulberry silk after boiling for a minute, leaving the tussah silk unattacked.

I think one may venture to say that the original cause for weighting was a desire to make up the original weight of the raw silk, the loss being occasioned by the boiling off, which amounts to about 25 per cent., that is, a lb. of 16 oz. is reduced to 12 oz. In 1875 it was stated that weighting was then carried on to the extent of 100, 200, and 300 per cent., but through fierce competition these numbers are now left far behind, for, as you will see from the analyses, it has reached 900 and 1,000 per cent. until it is no longer silk, but a mere conglomeration of mineral matter.

Newcastle Section.

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Hon. Local Secretary and Treasurer:
Saville Shaw, Durham College of Science, Newcastle-on-Tyne.

The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Committee:* G. Sisson and J. E. Stead.

SESSION 1896–97.

Meeting held in the Chemical Theatre, Durham College of Science, on Thursday, March 25th, 1897.

MR. W. W. PROCTOR IN THE CHAIR.

THE SAMPLING AND ANALYSIS OF COKE, AND VALUATION OF COAL FOR COKING PURPOSES.

BY W. F. KEATING STOCK, F.I.C.

Introduction.—I have ventured to bring the laboratory aspects of the coke trade before this Section for several reasons, chief among which is my desire to draw the attention of members to a growing necessity for greater uniformity of method in dealing with matters which are now inseparable from this very important branch of commerce. Another circumstance which has guided me in the choice of my subject is the scattered and fragmentary nature of its literature. It would be difficult to say where, outside of private reports and laboratory note books, one could find detailed information regarding such points as the proper method of sampling coke, its chemical analysis, its suitability for special purposes, or the improvement of coal from inferior seams, to fit it for coking. All these are matters which can be decided in the laboratory, probably to the satisfaction of the individual analyst. but it is a lamentable fact that occasionally upon one or more of these issues serious discrepancies arise; and it seemed to me that it would not be out of place to introduce the whole question as a subject for discussion before an influential society of industrial chemists, sitting in the very metropolis of the coal and coke trade. It would be too much to expect ones own ideas to meet with universal favour, I should be very well satisfied if they eventuated in the adoption of methods of working of established accuracy.

Sampling of Coke.—I think it will be generally conceded by analysts that, when dealing with material which, in the nature of things, is variable in composition, there is no more fertile source of discrepancies in analyses than insufficient and inefficient sampling. No matter how perfect the analytical methods may be, no matter how scrupulously they may be carried out, a faulty sample renders them of no avail, and I have found by long experience that the taking of a sample of coke from bulk is the rock upon which analysts most often split. I have abundant evidence that the time has come when vendor and purchaser alike would welcome any plan which would place this matter upon a fair and systematic footing. I have elsewhere pointed out, as a matter of actual observation (Iron and Coal Trades Review, 1887) that a sample of coke may be taken in at least six different ways. (1.) A few small pieces of coke may be broken off as many different columns. (2.) The whole of a column may be taken. (3.) The top or bottom "lift" of a column may be taken. (4.) The sampler may proceed to break small pieces of coke off a few columns, holding the latter, as he strikes them with his hammer, over the pail or other utensil in which he intends to carry the sample. All these methods are improper, the latter extremely so. Of the first three, it may be said that not in one case in a hundred would a representative sample be obtained, and of the fourth, that all the fine particles of shale, which are non-adherent to the column, would be started, out of the interstices of the coke. and would cause the ash to mount up quite abnormally. A fifth method, which is fairly reliable, and which is a convenient "works" method, consists in the sampler going over the surface of a train of coke, and breaking at least 10 columns on each truck, from each of which he takes a few pieces as they lie and transfers them by hand to his pail or bag and so proceeds until he has collected at least 20 lb. weight, the further treatment of which will be described later. The sixth method, which I do not hesitate to say is the one which should always be followed in case of dispute, is carried out in this way:—If the coke is in trucks at the ironworks, the trucks are run up on to the gantry over the storage bunks and two doors on one side of each truck are let down. This allows half the load to be seen from top to bottom, and enables the sampler to judge of the relative proportions of "large," "small," and "breeze." Portions of the coke are taken at bottom, middle, and top, and this is repeated for, say, 10 trucks, resulting in a bulk sample of from 3 cwt. to 5 cwt. If the coke is at the ovens, the sampling is simplified, as a barrow can be taken along the draw-bench, and filled

according to the judgment of the sampler. The bulk sample so obtained is then knocked down on clean iron plates (which are always available at an ironworks or coke ovens), until it is reduced to about macadam size. It is then thrown up into a heap and thoroughly mixed, after which it is spread out into a good sized square, and divided by running a shovel through it twice at right angles thus :

A.	B.
C.	D.

The corners A and D are now thrown out and all the small and dust belonging to them is swept away. The whole process is now repeated on the portions B and C, alternately pounding, mixing, squaring, and dividing until the sample will pass a $\frac{1}{2}$-in. sieve. The sample is well mixed at this stage, and about 20 lb. is taken for further reduction. This and the 20 lb. sample obtained by method No. 5 may now be treated alike. The sample in either case is taken down on a bucking plate until it will pass a $\frac{3}{8}$-in. sieve. The whole of it must pass through the sieve. It is again mixed, and 100 grms. are weighed off for moisture. A further portion of 500 grms. is put through a 20-mesh sieve, and after again thoroughly mixing, 20 grms. are taken and dried for analysis, after which this 20 grms. are finely triturated in about 2 grm. lots in a hard Wedgwood mortar. Finally, the whole of the fine powder is returned to the mortar and thoroughly mixed with the pestle. It is of vital importance that this final detail be not neglected. I attribute to its non-observance, several cases of serious discrepancy, which have been from time to time referred to me for settlement. It may be thought that such elaboration is uncalled for, but when I mention that differences of 3 or 4 per cent. in the ash, and of 3 or 4 tenths per cent. in the sulphur are not uncommon in samples of coke taken by different samplers from the same bulk, it seems clear that some standard mode of sampling is much wanted.

Chemical Analysis of Coke.—Coming now to the chemical analysis of coke, it will be necessary to revert for a moment to the 100 grms. of coke, which was taken for moisture when the sampling had reached the $\frac{3}{8}$-in. stage. This portion is dried for three hours at a temperature not exceeding 105° C. I myself prefer the heat of a water-oven in test cases. It is quite sufficient to weigh on a balance sensitive to one centigram. The loss is recorded as moisture. For the ash, 1 or 2 grms. of the finely-powdered coke are ignited in a platinum capsule at a bright red heat, until no further loss of weight is observed. A muffle furnace is indispensable where a large number of analysis are carried out simultaneously. The ash determination is always done in duplicate, and the greatest allowable difference between the duplicates is 2 mgrms., such a difference is rare where care is exercised. For the estimation of sulphur in coke, many methods have been proposed. I have used one method exclusively since 1874, in which year it was devised and published ("Chem. News," 1874, page 211) by myself. I mean what is known as the "lime method." It has undergone some important modifications in my hands, and as I often meet with singular "sports" from this method, I take the opportunity of describing it as now used in my laboratory. One grm. of coke in fine powder is mixed in a round bottomed platinum capsule with approximately 1 grm. of half hydrated caustic lime (see foot-note), moistened with 1 c.c. of water, rapidly dried, and heated in a muffle at bright redness, till all carbon has disappeared. The assay is then cooled, transferred to a 6-oz. conical beaker, 20 c.c. of cold distilled water added, and then 5 c.c. saturated bromine water. Solution is then effected by the addition of $7\frac{1}{2}$ c.c. pure strong HCl, and boiling for five minutes. The solution is filtered through a 9 c.m. Swedish filter paper. The filtrate must be bright. If not, it is passed a second time through the filter. The empty beaker and the filter are then washed three times respectively, the beaker washings being passed through the filter before the latter receives its three washes. The filtered solution is boiled, and whilst boiling, 10 c.c. of 5 per cent. barium chloride solution are run in from a pipette, in such a way that the boiling is not sensibly checked. The boiling is continued for half an hour, and the beaker is then kept on the hot plate, so that its contents are nearly at boil for at least three hours. Finally, the resulting precipitate of $BaSO_4$ is filtered off, washed very thoroughly with hot water, the filter and its contents is loosely folded whilst wet, and put into a platinum capsule. The filter is then slowly charred, the $BaSO_4$ is heated to redness for five minutes, weighed, and calculated to sulphur. It must be noted here that the sulphur assays are never, on any account, to be fired in the same muffle simultaneously with ash assays. The latter give rise to free SO_2, which is absorbed by the lime of the sulphur assays and converted into SO_3.

The carbon is taken by difference, and the analysis is stated thus :—

	Per Cent.
Carbon	..
Ash	..
Sulphur	..
Moisture	..
	100·00

Since the introduction of the manufacture of hæmatite iron into the Cleveland district, a good deal of attention has naturally been attracted to the question of phosphorus in coke. According to my experience, the proportion of this element in different cokes varies from 0·005 per cent. to 0·017 per cent., and it is a point which ironmasters cannot afford to neglect if they are to undertake to guarantee low phosphorus irons for any acidic method of steel making. I have found the following method of determining phosphorus in coke to be convenient, and to yield check results on duplicate assays of any given sample:—Ten grms. of the coke to be assayed are spread out on flat trays of platinum and heated in a muffle until burnt to an ash. This is finely ground in an agate mortar, transferred to an ordinary 8-oz. beaker, and treated with 20 c.c. of brominised HCl. The mixture is digested at near boiling for one hour, the beaker being covered with a watch glass to prevent too rapid evaporation of the HCl. The watch glass is then drawn aside, and the greater part of the HCl is evaporated as quickly as is safely possible. The solution is now diluted with 20 c.c. of water. It is filtered into a conical flask, and the residue and filter are washed with distilled water. Filtrate and washings should not exceed a bulk of 45 c.c. Fifteen c.c. of strong ammonia are now added to the contents of the flask, and this is neutralised by cautious addition of nitric acid from a *small* pipette. When resolution takes place, dilute ammonia is dropped in till a faint opalescence appears, and this is taken up with a few drops of nitric acid. All these operations are carried out at near boiling. The faintly acid solution now receives a further addition of $2\frac{1}{2}$ to 3 c.c. of strong nitric acid, and then 5 c.c. of a 10 per cent. solution of ammonium molybdate is suddenly added from a measure (not a pipette), whilst the contents of the flask are being briskly agitated. The solution should remain quite clear for a second or two, and then the yellow ammonia-phosphomolybdate makes its appearance in a granular form which rapidly settles out. When this takes place as described, the experiment is almost certain to succeed. The flask is heated to about 90° C. with constant shaking for five minutes, and then the precipitate is filtered off on a hardened filter paper, washed three times with 1 to 50 nitric acid, dried in the water-oven for $\frac{1}{2}$ to $\frac{3}{4}$ hour, brushed off the filter into the balance scoop and weighed. The weight in parts of a grm. multiplied by 0·163 gives the percentage of phosphorus in the coke.

Mechanical and Chemical Analysis connected with Coal Washing for Coking Purposes.—I have already alluded to

NOTE.—The half hydrated lime is made by calcining marble, and dividing the calcic oxide so obtained into two equal portions. One of these has added to it exactly the weight of water to form $Ca(HO)_2$, and is then mixed with the other half. This leaves a mixture to which more water may be added without fear of loss by spattering.

the utilisation of inferior seams of coal for coking. It will be unnecessary to remind members of this Section of this Society of Chemical Industry that for at least a quarter of a century efforts have been made to improve the condition of certain qualities of coal by various washing processes, differing in elaboration from the ordinary long wood trough with its screens and settling ponds, to the wonderfully complete system of the Lührig Company of most recent introduction. As the construction of washing plant varies, so does the cost, which may be from 250*l.* to 10,000*l.* or more, but, whatever the system employed, it has recently become the practice for coal owners to enquire to what extent the coal they propose to wash will benefit from the treatment. Coals differ very greatly in their amenability to washing processes, especially those processes depending upon gravity. A coal containing a hard stoney waste is readily cleaned, whereas coals containing light carbonaceous shales, whose ash runs to 18 or 20 per cent., whilst their gravity is only a few points above that of the clean coal, will resist all ordinary treatment.

Again, the question may be one in which the sulphur only is involved. There are certain coals with a low ash and a very high sulphur, and it is often a matter for enquiry whether the proportion of sulphur can be reduced by washing. These questions are seldom directed to the whole produce of a mine. In the majority of cases it is what is known as "rough small," that is, the coal which remains when the screens have separated the more valuable descriptions, which is subject to washing. This is not a paper on coal-washing but in many cases it is imperative that coal should be washed before coking, and I have introduced the subject in order to indicate how it may be shown in the laboratory to what extent a given coal may be improved for coking purposes by a given method of washing. I can best illustrate the lines on which the procedure runs by giving one or two actual examples —

A sample of "rough small" coal sent for analysis will probably be divisible into five different portions, viz:— "Nuts," "Peas and Pearl," "Pearl and Duff," "Washer Waste" and "Coal lost in Washer Waste." In order to separate quantitatively these different portions, two sieves are required, a ½-in. mesh and a ⅜-in. mesh, a good supply of calcium chloride solution of about 1·35 specific gravity, and a tall glass cylinder which will hold 3 litres. For the analysis, 500 to 1,000 grms. of the air-dried sample are taken according to the proportion of "small" it contains. This is thrown on to the ½-in. sieve. The "nuts" are left on the sieve and are weighed. The portion passing the ½-in. mesh is then put on to the ⅜-in. sieve. "Peas and pearl" remain on the sieve and are weighed. These two portions are now carefully examined for shale and stone, which are picked out, the weight deducted in each case and the combined weight is added to that of the washer waste. The portion passing the ⅜-in. sieve is now treated with the calcium chloride solution in the glass cylinder. This must be done by half filling the cylinder with the solution, and then carefully shooting in the coal from a smooth scoop. The mixture is then very thoroughly stirred with a smooth wooden rod. It is absolutely necessary that every particle of the solid should be wetted. The cylinder is now filled up to within about 2 ins. of the top and the mixture stirred repeatedly during two or three hours; it may then be allowed to stand at rest for 18 or 24 hours. At the end of this time the glass jar is examined and the clean coal is found at the top, a clear portion in the middle, and all the separable earthy impurities at the bottom. A large porcelain basin is now filled about two-thirds with clean water and the cylinder is tilted over it, when, with a little careful management the whole of the coal can be made to fall into the water. The rest of the manipulation is only a matter of thorough washing with the aid of fine cloth filters for the smaller particles of coal. When the washed coal is dried and weighed, it stands as "pearl and duff." It only remains now to wash, dry, and weigh the waste, and the mechanical analysis is complete. What has been done so far shows the amount of waste the coal will give on washing. But the cleaned and washed coal is then analysed for ash and sulphur, and the complete analysis stands as follows in the example selected :—

Composition of the Air-Dried Sample:—

	Per Cent.
"Nuts," from 1 in. down to ½ in.	34·40
"Peas and pearl," from ½ in. down to ⅜ in.	9·15
"Pearl and duff," below ⅜ in.	47·00
"Washer waste"	6·52
"Coal lost in washing"	2·93
	100·00

The washed coal yields—

Coke	65·95
Volatile matter	34·05
	100·00
Ash in washed coal	4·67
Sulphur in washed coal	1·62

Assuming that the coal contains ⅘ths of the sulphur contained in the coal, the composition of the dry coke would be—

Carbon	91·54
Ash	7·06
Sulphur	1·40
	100·00

It now only needs comparison with the analysis of coke from the same coal untreated, to show what the gain in quality (if any) is.

Before leaving this part of my subject I will ask permission to quote another example where the problem is much more complicated than in the one just given. A sample of "peas and duff" had been washed, and it was required to know how it differed from the unwashed article, and what proportion of stony waste it retains, also what proportion of coal was passing off with the stony waste. Upon my answer to these questions depended largely the erection of a very expensive plant. I asked to be supplied with samples of the original "peas and duff," the same washed by the process in use, and a sample of the stony waste. In this case the sieves were not used as the samples were too fine. The calcium chloride method was adopted, and the following results were obtained :—

——	"Original."	"Washed."	"Washer Waste."
Mechanical analysis :—	Per Cent.	Per Cent.	Per Cent.
Good coal down to "pearl."	67·40	89·20	4·00
"Fine duff"	4·80	2·90	3·55
"Stony waste"	27·80	7·90	92·45
	100·00	100·00	100·00
Chemical analysis :—			
Fixed carbon	48·90	60·64	Omitted.
Volatile hydrocarbons	29·50	31·97	
Ash	18·30	5·00	
Sulphur	1·47	0·97	
Moisture	1·83	1·42	
	100·00	100·00	
Yield of coke	67·20	65·65	
Ash in coke	27·30	7·60	
Sulphur in coke	1·09	0·73	
Fixed ash in coal	2·75	2·22	
Free ash in coal	15·55	2·78	

Shortly summarised, the foregoing analyses prove that the washing process in use is capable of removing 82·23 per cent. of the free ash from the coal along with 33·03 per cent. of the sulphur, resulting in a diminution of 19·70 per cent. of ash in the coke, and 0·36 per cent. of sulphur, whilst in effecting this improvement, there has been a loss of 7·55 per cent. of coal with the "washer waste." It is, however, necessary to carry the enquiry a step further in order to ascertain what would be the gain in quality, and what the ultimate loss of coal when using the most perfect type of washing plant. In order to arrive at this information, the two grades of coal, namely, "pearl" and "fine duff,"

recovered in the foregoing mechanical analyses, were separately subjected to chemical analysis. The following are the results obtained in the examples quoted :—

——	"Original."	"Washed."	"Washer Waste."
Good coal down to "pearl":—	Per Cent.	Per Cent.	Per Cent.
Coke	64·65	64·55	67·55
Volatile matter	35·35	35·45	32·45
	100·00	100·00	100·00
Ash in coal	2·60	2·10	14·55
Sulphur in coal	1·09	1·02	1·50
Ash in coke	4·00	3·25	21·50
Sulphur in coke	0·84	0·79	1·11
"Fine duff":—			
Coke	67·40	68·10	68·90
Volatile matter	32·60	31·90	31·10
	100·00	100·00	100·00
Ash in coal	5·00	6·60	16·48
Sulphur in coal	1·07	0·95	1·90
Ash in coke	7·40	9·70	23·80
Sulphur in coke	0·79	0·70	1·38

In order to avoid confusion, the whole of the foregoing analyses are rendered on the air-dried samples.

The general conclusions to be drawn from the example just quoted are :—

1st. That the sample in the original is not fit for coking purposes.

2nd. That by perfect washing it yields 72·20 per cent. of coal, and that the whole of this is well adapted for coking.

3rd. That the sample which is washed at the colliery is of very fair quality for coking purposes, the ash being 7·60 per cent. and the sulphur 0·73 per cent., although the presence of practically 8 per cent. of stony waste shows that it is imperfectly washed. It is shown also that it is susceptible of great improvement by perfect washing, for when perfectly cleaned, the coke it yields contains only 3·50 per cent. of ash and 0·77 per cent. of sulphur.

4th. That with regard to the sulphur the colliery washer brings it down to as low a figure as can be got by perfect washing.

5th. That the total loss of coal resulting at the colliery washer is only 2·00 per cent. That this coal is of very inferior quality. It averages 15·50 per cent. of ash and 1·70 per cent. of sulphur. That if recovered (which could only be done by perfect washing) and spread over the perfectly washed coal, it would raise the ash in the coke from 3·50 per cent. to 5·00 per cent., and the sulphur in the coke from 0·77 per cent. to 0·83 per cent. It was quite clear that in this case the erection of a more perfect washer could not have improved the quality of the coke whilst saving the coal, and that the coal was not worth saving. It is probable that, had the proposed scheme been carried out, it would have entailed an annual loss to the colliery of nearly 500*l*.

Discrimination of the Origin of Coke.—It is sometimes required to know whether two or more cokes have a common origin. The matter is simple enough when the proportions of ash and sulphur in each are widely different. When these are closely in agreement, it is best to resort to complete analysis of the ash of the samples. Here is a case in point :—It was required to know whether three samples of coke were from one and the same seam of coal in the same pit. They were dried and the ash and sulphur were determined in each with the following results :—

——	No. 1.	No. 2.	No. 3.
	Per Cent.	Per Cent.	Per Cent.
Ash	9·15	8·20	9·60
Sulphur	0·87	1·37	0·98

Considering the variations found in these constituents in samples from bulk, I thought it unsafe to rely upon these indications, as the matter was a very important one, and therefore had recourse to the complete analysis of the ash, when the following results were obtained :—

——	No. 1.	No. 2.	No. 3.
	Per Cent.	Per Cent.	Per Cent.
SiO_2	47·80	38·32	46·87
Al_2O_3	33·82	26·52	33·45
Fe_2O_3	10·15	22·96	11·01
Mn_3O_4	Trace.	Trace.	Trace.
CaO	3·47	6·46	4·43
MgO	1·60	2·51	1·40
K_2O	0·38	0·23	0·25
Na_2O	0·56	0·23	0·64
P_2O_5	Trace.	Trace.	Trace.
SO_3	2·21	2·80	1·92
	99·97	100·06	99·97

On the strength of these analyses, I felt justified in reporting that Nos. 1 and 3 had the same origin, whilst No. 2 had a totally different origin.

Excess of Moisture in Coke.—Disputes occasionally arise as to the proportion of water in coke. As coke is drawn from the ovens in an incandescent state, large volumes of water quickly applied are necessary to prevent loss by oxidation, and very considerable judgment has to be exercised to prevent excessive quantities remaining in the coke when slacked. I have known a sample come off the bench with over 11 per cent. of water; but as a rule, samples received for analysis do not exceed 1 per cent., and in the majority of cases 0·70 per cent. is the maximum limit. As to the highest possible proportion of water a sample of coke can contain, I have the following results from a recent experiment. A number of pieces of hard blast-furnace coke, dried at 212° F., weighed 399 grms. After soaking in cold distilled water for 24 hours, and freeing from extraneous water by dabbing with a sponge, they weighed 475·5 grms., giving a gain of 76·5 grms., or 19·17 per cent. I have met with no such proportion in practice. In 1894 I followed 16 trains of coke from various localities to their destination after a heavy rainfall of some hours duration. All these cokes were analysed at the ironworks laboratories by myself, after sampling by the method I have described as No. 6. The lowest moisture was 2·30 per cent., and the highest 12·36 per cent. Average, 5·25 per cent.

Mechanical Condition of Coke.—This is a matter upon which the analyst is often asked to report. The most he can fairly say is whether the sample is hard, dense, and of good colour. I have tried to arrive at a standard of mechanical condition by dropping columns of coke of nearly approximate weight from measured heights. I failed to get consistent results from different samples of the same coke from the same delivery; and my opinion is that it is not a laboratory question, save in so far that the analyst is perfectly justified in giving his opinion of the respective value, for example, of "black ends" and "bright coke," when he can base that opinion on chemical analysis and practical knowledge.

In bringing this paper to a close I may refer to some curiosities of coke analysis.

Coke-Oven Flue Dust.—The analysis of coal or coke-ash serves to show that its composition is fairly complex. It has often occurred to me that metals capable of volatilisation might reasonably be expected to occur in coal, and that the flue dust of coke-ovens would be a substance which would repay investigation. One would not, for example, expect to find zinc in the ash of either coal or coke, but that it exists in coal in some cases is beyond doubt. I have myself detected zinc oxide in coke-oven flue dust in two

instances. The samples I examined had the following percentage composition :—

——	No. 1.	No. 2.
	Per Cent.	Per Cent.
SiO_2	23·37	34·67
Al_2O_3	18·06	29·08
Fe_2O_3	6·57	8·85
Mn_3O_4	Trace	Trace
PbO	Trace	Trace
ZnO	43·51	24·04
CaO	1·37	1·37
MgO	0·99	0·96
KCl	0·59	Omitted
NaCl	1·11	Omitted
H_2O	3·80	0·15
SO_3	None	None
	99·37	99·12
Metallic zinc	34·91	19·29

State of Existence of Sulphur in Coke.—The question of the occurrence of sulphur in forms other than that of sulphides of iron has been investigated by quite a number of observers, and it has been sought to prove that such of the sulphur as existed in combination with alkalis or alkaline earths did not combine with iron in the blast furnace. This no doubt is the case; but although it is quite a quarter of a century since this question first attracted attention, blast-furnace managers are as shy as ever of sulphury cokes. I am not at all clear that we possess a reliable method for the separation of oxidised sulphur compounds or soluble sulphides from sulphides of iron when they exist together in coke. I have never been able to find more than very insignificant amounts of sulphur which could be got out of north-country cokes by boiling with sodium carbonate solution. However, this is a debatable point. I have found one compound of sulphur in certain cokes, which is very embarrassing to the analyst unless great precaution is taken in grinding the sample for analysis. This is a magnetic iron sulphide, which may readily be sorted out of a roughish sample with the aid of a magnet. It contains, according to my analysis, as much as 16 per cent. of sulphur and is difficult to reduce in a mortar. It occurs in cokes containing a low and ferruginous ash. A good plan of securing its proper diffusion throughout the sample is to reduce to fine powder, pass this through a 70-mesh brass bolting cloth, and stamp the coarse particles in a steel diamond mortar until all will pass the 70 mesh, when the whole is mixed carefully in the Wedgwood mortar once more.

Occurrence of Soluble Salts in Coke.—On treating a considerable weight of powdered coke with distilled water, and agitating frequently for four or five days, varying proportions of matter dissolve out of different samples.

In the case of three varieties of West Durham coke the following results have recently been obtained in my laboratory :—

——	No. 1.	No. 2.	No. 3.
Percentage of soluble matter yielded by the coke.	0·08	0·27	0·124

In all these cases calcium and magnesium were the only metals found in weighable quantity in solution, and the acid radicles were thiosulphuric, sulphurous, and sulphuric. There were no soluble sulphides. It is exceedingly probable that the salts dissolved out of coke by even air-free water, are oxygen derivatives of the calcium and magnesium sulphides, which must exist in the coke before it is slacked. The addition of water to incandescent carbonaceous matter containing an admixture of calcium and magnesium sulphides, would, of necessity, lead to complicated reactions, and, given free access afterwards to air and moisture, it is not surprising that soluble sulphides are no longer to be traced.

Discussion.

Dr. H. S. Pattinson had been much interested in Mr. Stock's paper. He agreed with Mr. Stock that the importance of proper sampling could not be over-rated, and he thought that the method adopted by the author of the paper of estimating the whole pieces and the small breeze was a very good one, but a method which could not always be carried out. On Tyneside, where samples had often to be taken as the coke was being loaded into ships, the practice of his firm had been to take long sections of the coke as it was on the spouts, and in that way probably a column from each truck would be secured, the sample being then broken down as described by Mr. Stock. With regard to the breaking down of the sample, if this were done on an iron plate and the crushed sample then ground fine on the same plate the ash of the coke would be very considerably increased, the sharp hard particles of coke causing very appreciable wear of the iron surfaces. Of methods of sulphur testing he (Dr. Pattinson) had not much experience, as he invariably used the method described by Mr. Stock. With regard to phosphorus in coke, that was a constituent they were not often asked to determine, but they usually found the amount of phosphorus in Durham coke to run from 0·01 to 0·03 per cent. The presence of more than 0·03 per cent. would cause an iron maker to seriously consider whether he ought not to change his coke.

Mr. W. W. Proctor said he always used Mr. Stock's method of estimating sulphur, and had found it very satisfactory. He would like to ask Mr. Stock whether he had found that the insoluble portion contained phosphorus? Lately in the *Comptes Rendus* a writer had stated that no wet method gave the whole of the phosphorus actually present, and that in all cases the fusion method gave a higher result than the wet processes.

Dr. H. S. Pattinson said that their experience had been that in some cokes all the phosphorus could be got into solution, but that in some cases where, for instance, titanic acid was present this was not possible. On the whole, their experience led them to believe that there was not very much difference between the two methods.

Dr. Bedson asked whether the apparent discrepancy between Dr. Crace Calvert's results and those of other analysts with regard to the extraction of sulphur with sodium carbonate solutions, might not be owing to the fact that Calvert worked with Lancashire coke.

Mr. W. F. K. Stock, in reply to Dr. Pattinson's remarks concerning sampling, agreed that the rubbing fine of the sample on the bucking plate was quite inadmissible. He used a chilled steel bucking plate, but even with that it was quite easy to increase the ash of the coke by 0·2 per cent. owing to the steel particles rubbed off. With regard to Mr. Proctor's question, he (Mr. Stock) thought that the writer in the *Comptes Rendus* was at fault in that he attempted to extract his phosphorus with *aqua regia*, but it was well known that if much phosphorus was present the nitric acid would have little effect upon it until the acid was largely broken up. For his own part, he had never found any difficulty in extracting the phosphorus with strong hydrochloric acid. In fireclays with, perhaps, 3 per cent. of titanic acid it was often very difficult to extract the phosphorus, but this would again depend upon the condition in which the phosphorus was present—if the phosphorus and titanium were in combination, it was very difficult to separate them. There was another point with regard to the fusion method for estimating phosphorus, and that was that he had never met with a sample of sodium carbonate free from phosphorus. In reply to Dr. Bedson's suggestion it seemed probable that Dr. Calvert's results might be due to the cause he mentioned. The whole practice of coke-making had changed in the last 30 years, and one had to deal with varieties of patent oven cokes and others never met with formerly. If Dr. Calvert's experiments were carried out with soft coke, he probably would get out the sulphur by his method, but with modern hard cokes it seemed too intimately held by the carbon. He had never been able to extract more than a trace of sulphur by Dr. Calvert's process.

SUPPLEMENTARY NOTE. April 17th, 1897.

At the close of the paper the Chairman drew attention to a statement by Louis Campredon (Comptes Rend. 1896, **123**, 1000—1003) to the effect that when very finely powdered coal-ash is heated with concentrated HCl for 15 or 20 hours the whole of the phosphoric acid is not dissolved, and that as much as 10 per cent. or even 25 per cent. of the total phosphorus present may be left in the insoluble residue.

Dr. H. S. Pattinson spoke of the probable presence of titanium in coke-ash, and of its ascertained action in retarding the complete separation of phosphorus. (This Journal, 1895, p. 433.)

I have since analysed several samples of coke for phosphorus, and I have in four cases tested the soluble and insoluble portions for that element. My results, as shown in the subjoined table, substantially confirm those of L. Campredon as to the inability of HCl to dissolve all the phosphorus. But I did not find, as mentioned by him, that the temperature at which the incineration took place had any influence on the result, nor did I find any titanium in these samples.

—	High-Phosphorus Coke.		Low-Phosphorus Coke.	
	No. 1.	No. 2.	No. 3.	No. 4.
Percentage of P dissolved in one hour by brominised HCl.	0·064	0·069	0·0047	0·0044
Percentage of P recovered by fusing residue with pure Na_2CO_3.	0·006	0·003	0·0006	0·0013
Total percentage of P ...	0·070	0·072	0·0053	0·0057
Ratio of soluble to insoluble P.	100—9·37	100—4·34	100—12·76	100—29·54
Percentage of titanium in ash.	None	None	None	None

According to these results, fusion with alkaline carbonate must be resorted to when the total phosphorus is required; but the method given in the body of my paper still remains a rapid means of sorting high- and low-phosphorus cokes.

MICRO-CHEMICAL EXAMINATION OF ALLOYS.

BY J. E. STEAD.

(*This Journal*, 1897, 200.)

DISCUSSION.

Prof. H. LOUIS wished to draw attention to two points. In the first place, if he had understood Mr. Stead correctly, the latter had stated that the result of Prof. Arnold's work was to show that the gold in the gold-bismuth alloy was not in an allotropic condition. He (Prof. Louis) had attacked the problem some time ago in a totally different way, namely, by making various gold alloys, and then dissolving out the alloying metal, a typical example being the parting of gold cornets. In every case he obtained the gold in a brown, spongy, and sometimes highly voluminous, mass, the specific gravity of which was always markedly higher than that of ordinary gold.* As the gold was thus liberated in an allotropic condition he concluded that it existed in this state in the alloy itself. At the same time he so far agreed with Mr. Stead's view that he did not hold that the brittleness of the alloy was due to an allotropic modification of the gold, because he had obtained similar results in his own experiments, whether the alloy treated were brittle (gold and lead, &c.) or quite malleable (gold and silver, &c.).

With respect to the tin alloys, he had seen something of the arborescent figures produced by etching surfaces of commercially pure tin, just as shown by Mr. Stead; he wished to know whether Mr. Stead had investigated microscopically the structure of tin with just sufficient impurity in it to destroy the "cry." It was well known that very small amounts of certain metals (antimony, &c.) would prevent tin from giving the characteristic "cry," and he would like to know whether the loss of this property was accompanied by any marked change in the micro-structure of the metal.

* Trans. Amer. Inst. Min. Eng., XXII., p. 117; XXIV., pp. 182 and 705.

Mr. T. W. HOGG said that he was much interested in Mr. Stead's account of the defects in steel rails. In his own experiments on segregated steels he etched the surface of the section of the entire ingot, and in that way the segregated portions could be observed standing out in sharp lines running through the normal structure of the steel. Though he had worked a good deal with etched specimens, he was somewhat sceptical as to the value of the mere examination of the etched surface. Mr. Stead's method of supplementing this examination by a treatment with acid and the extraction in this way of definite compounds was however capable of yielding much information. He had never seen crystals of cyano-nitride of titanium on an etched surface, yet he had shown that something like 2,000,000 crystals of this substance were present in every cubic inch of the alloy examined, and that they could be extracted by suitable treatment with acids.

Nottingham Section.

UNIVERSITY COLLEGE, NOTTINGHAM.

Chairman: F. J. R. Carulla.
Vice-Chairman: J. M. C. Paton.

Committee:

L. Archbutt.	F. E. Lott.
R. M. Caven.	*T. Mason.*
F. Clowes.	J. J. Sudborough.
H. Forth.	*C. Taylor.*
W. G. Johnston.	G. J. Ward.
J. F. Kempson.	

Treasurer: S. J. Pentecost.
Hon. Local Secretary:
J. T. Wood, 29, Musters Road, West Bridgford, Nottingham.

The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Chairman:* J. O'Sullivan. *Committee:* S. F. Burford and F. J. R. Carulla.

SESSION 1896–97.

May 26th (Nottingham).

Meeting held on Wednesday, March 24th, 1897.

MR. F. J. R. CARULLA IN THE CHAIR.

ON HEHNER'S BROMINE THERMAL TEST FOR OILS.

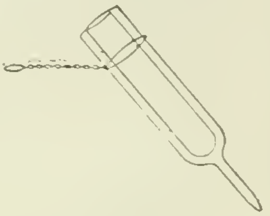
BY L. ARCHBUTT, F.I.C.

THE heat of combination of oils and fats with bromine has been made the basis of a new thermal test by Hehner and Mitchell (Analyst, XX. (1895), 146), the value of which depends upon the fact that there is in the case of most oils and fats, a nearly constant ratio between the bromine thermal value and the Hübl iodine value. The correct determination of the iodine value takes, as is well known, several hours, and involves the use of a special reagent which cannot be prepared for use at short notice; but the bromine thermal value needs no special reagent for its determination, can be ascertained within half an hour or less, and when multiplied by a constant gives a number which, in general, closely approximates to the iodine value. As an auxiliary test it is therefore most useful.

In my experiments I have found no difficulty in following the method of working described by the authors of the process, and I have not seen the necessity for the modifications introduced by Wiley (Jour. Amer. Chem. Soc. XVIII. (1896), 378), which detract from the simplicity which is one

charm of the process. Wiley does not give any figures showing that the results obtained by his method of working are any more concordant than those obtained by the author's method.

The reagents required are liquid bromine and methylated chloroform. The test is made, preferably, in the vacuum jacketed (Dewar's) tube suggested by the authors, which can be suspended obliquely, by means of a stiff platinum wire loop, as shown in the figure, from the right-hand



VACUUM JACKETED TUBE.

arm of the balance, a counterpoise made from a piece of thick sheet lead being placed in the other pan. By opening the side door of the balance case, the oil or fat can be dropped straight into the tube, and the exact weight required easily and quickly adjusted. The tube is then slipped out of the wire loop and fixed firmly, by means of its short stalk, in a suitable stand, and 10 c.c. of chloroform are introduced from a pipette. The temperature of the bromine is next ascertained to the nearest 0·1 degree, by stirring with a centigrade thermometer divided into fifths of a degree; and the same thermometer, freed from adhering bromine, is then immersed in the chloroform solution of the oil, which is brought to exactly the same temperature as the bromine. In adjusting the temperature, the outer tube may be warmed by the hand or cooled by immersion in cold water: but the warming or cooling of the solution in the inner tube can take place only by radiation, and is the slowest part of the process. Immediately the temperature has been correctly adjusted, 1 c.c. of the bromine is withdrawn from the bottle, by means of a pipette having a short soda-lime tube fixed to the upper end, with a flexible rubber tube for mouthpiece, and is added to the chloroform solution of the oil, which is immediately stirred with the thermometer until the mercury has risen to the highest point and begun to fall again. This takes only about 15 seconds, and the rise of temperature is the "*bromine thermal value.*" The vacuum tube, when emptied, rinsed with chloroform, and wiped with a cloth, is ready for the next test. Five or six tests may thus be made in an hour.

The numerical value of the "constant" for converting the thermal values into iodine values is liable to vary in the hands of different operators, depending upon the heat capacity of the particular tube and thermometer used, and doubtless to some extent upon the exact method of manipulation. Hehner and Mitchell found it to be about 5·5, and the same for most oils: I have found it rather higher, and not quite the same for different kinds of oil. Thus, in experiments with tallow, olive oil, rape oil, and linseed oil, which are the only oils of which I have tested more than single samples, the factor has varied from 5·7 to 6·2; this variation is, however, partly due to the fact that I have found it advisable not to use the same weight of all oils, for reasons explained further on. The results of my experiments with the four descriptions of oil above mentioned are arranged in Tables 1 to 4, and it will be seen that the agreement between the observed and the calculated iodine values is, in general, very satisfactory. Thus, with the seven samples of tallow, the maximum difference is 1·7; in the case of the olive oils it is 2·5; eleven samples of rape oil gave a maximum difference of 1·5; and the agreement in the case of raw linseed oil is nearly as good. The greatest difference observed in testing ten samples of the latter oil was 4·2, but if we omit the first two samples the difference does not exceed 2·5.

In the case of rape oil, a fairly constant ratio exists between the Maumené figure (the rise of temperature with sulphuric acid) and the Hübl value—a fact which has already been called attention to in the case of some other oils; but although, in my experiments, the Maumené ratio was only one-third as great as the bromine ratio, the agreement between the observed and calculated iodine values was not so close. Thus, in experiments with eight samples of rape oil, the differences between the Hübl values and the numbers calculated from the Maumené figures varied from 0·4 to 2·7, whereas the numbers calculated from the bromine thermal values gave differences varying from 0·1 to only 1·5. The sulphuric acid thermal test has, no doubt, a special value in certain cases, and gives information not afforded by the iodine number; but as a basis for calculating the latter it cannot compare either for accuracy or simplicity with the bromine test.

The use of liquid bromine is not a serious objection to this process, if one has a good fume cupboard; neither is there any real difficulty in measuring the bromine with a pipette, provided the connections between the pipette, soda-lime tube, and flexible tube are perfectly tight. Wiley's plan of forcing the bromine up into the pipette is a good one, but the action of the bromine vapour would soon harden the rubber stopper in the neck of the flask, and the simpler plan of taking the bromine with a pipette direct from the stoppered bottle in which it is kept seems preferable. An error of 1·5 per cent. in measuring the bromine makes no appreciable difference in the result, and such an error would be a large one with a narrow pipette.

After adding the bromine, the rise of temperature is very rapid, and a sharp eye is needed to read the thermometer, as the mercury, having reached the highest point, quickly begins to fall; yet, in many consecutive experiments with the same oil, the extreme difference I have observed has been 0·3°. Duplicate experiments, if carefully made, usually agree within 0·1°.

Bromine as purchased, the same fully saturated by shaking in a separator with water and drawing off the bromine, and the same dehydrated by shaking with strong sulphuric acid and redistilling, gave practically the same results, as the following figures, obtained with the same sample of rape oil, show:—

Experiment	Thermal Values (Centigrade Degrees).		
	With Bromine as purchased.	With Bromine saturated with Water.	With Anhydrous Bromine.
1	20·4	20·2	20·1
2	20·3	20·4	20·2
3	20·1	..	..
4	20·2	..	..

In my experiments with this process, the only modification I have found it desirable to make has been in regard to the quantity of oil used. Experiments with tallow, which develops comparatively little heat, showed that by working on 2 grms. and dividing the result by two, gave nearly the same result as working on 1 grm. Therefore, for the sake of greater accuracy, I prefer to take 2 grms. of tallow or any other fat consisting mainly of saturated glycerides, dissolve in 10 c.c. of chloroform, add 1 c.c. of bromine, and divide the rise of temperature by two, to obtain the thermal value. The results in Table 1 were so obtained. Of olive, rape, and most oils, 1 grm. is a suitable quantity; but of linseed oil, which reacts very violently with bromine, 0·5 grm. is best taken and the result multiplied by two. As this doubles the error of observation, a single experiment should not be relied upon in the latter case. The results in Table 4 are the means of two or three experiments made in this way.

The results recorded in this paper prove conclusively that the thermal test with bromine is a most valuable auxiliary method for the valuation of oils, and in laboratories where a large number of oils of the same kind have to be examined, a very great saving of time and labour may be effected by it. The test should also prove useful in the examination of single samples, provided that the apparatus used in making the test has been previously standardised by making with it

comparative determinations of the Hübl iodine values and the bromine thermal values of about half a dozen samples of the same description of oil.

My thanks are due to Mr. Hehner for the vacuum-jacketed tube, and to Mr. Rowland Williams for most of the samples of linseed oil used in making these experiments.

1. Tallow. 7 samples (2 grms. used; the rise of temperature ÷ 2 = β).

Description.	Bromine Thermal Value (β).	Hubl Iodine Value.	β × 6·2.
American beef	6·1	39·1	37·8
Home refined	6·7	41·3	41·5
"	6·7	41·8	41·5
"	7·05	44·3	43·7
"	7·2	44·9	44·6
Australian mutton	7·55	46·3	46·8
Home refined	8·9	53·5	55·2

2. Olive Oil. 10 samples (1 grm. used; the rise of temperature = β).

Description.	Bromine Thermal Value (β).	Hubl Iodine Value.	β × 5·7.
	13·55	78·7	77·2
	13·8	78·9	78·7
Malaga	13·8	79·3	78·7
"	14·2	78·1	80·9
Unknown	14·2	81·4	80·9
"	14·35	81·4	81·8
Gallipoli	14·4	82·0	82·1
Unknown	14·45	84·2	82·4
Gallipoli	14·5	82·5	82·6
"	14·5	82·1	82·6

3. Rape Oil. 11 samples (1 grm. used; rise of temperature = β).

Description.	Bromine Thermal Value (β).	Hubl Iodine Value.	β × 5·92.	H_2SO_4 (96·3 per Cent.) Thermal Value.	β × 3·55.
Stettin	17·0	99·6	100·6	..	60·4
Unknown	17·35	102·7	102·7	59·5	61·1
"	17·4	103·3	103·0	60·7	61·4
"	17·3	103·1	102·4	61·0	61·4
"	17·7	105·7	104·8	63·8	62·8
"	17·8	104·6	105·4	62·5	63·2
"	17·9	106·2	105·9	63·5	63·5
"	18·25	108·1	108·0	65·8	64·8
"	18·6	109·4	110·1	66·7	66·0
Black Sea	19·1	113·5	113·1	..	67·8
"	20·3	121·7	120·2	..	..

4. Raw Linseed Oil. 10 samples (0·5 grm. used; the rise of temperature × 2 = β).

Description.	Bromine Thermal Value (β).	Hübl Iodine Value.	β × 6·0.
Old sample	28·5	167·1	171·0
Unknown	28·8	177·0	172·8
American (?)	29·6	177·0	177·6
Unknown	29·7	177·8	178·2
Calcutta	29·8	178·7	178·8
"	30·45	183·3	182·7
Baltic	31·35	188·5	188·1
"	31·4	188·8	188·4
"	31·75	188·8	190·5
"	32·5	192·5	195·0

Discussion.

Mr. Otto Hehner said that he was pleased that Mr. Archbutt had had the advantage of working with other oils than he himself was accustomed to, and had got such good results. There was a lowering of temperature when bromine was dissolved in chloroform. If 1 per cent. of bromine was added to 10 c.c. of chloroform there was a falling of temperature, which, under certain circumstances, might influence the result. This had been pointed out by Wiley, who first dissolved his bromine in chloroform, and used the chloroform solution of bromine.

Mr. F. E. Lott asked whether the purity of the chloroform had any effect on the process, and how far the varying temperature would affect the results.

Prof. F. Clowes asked how far the process applied to oils generally. He did not know whether it was a general process, available for all oils and fats.

Mr. J. T. Wood asked whether any experiments had been made with fish oils. Some years ago they tested, by Hübl's process, in his firm's laboratory, a good many samples of fish oil, and found the iodine values varied very much, and gave no information as to the quality of the oils for leather dressing—the purpose for which they used them. If they had been able to use this test, it would have been a great saving of time.

Mr. Archbutt, in reply, thought that it was desirable that a separate factor should be used for each oil. Every chemist who used the test frequently with oils of the same kind would find that he could, by practice, work out a factor with, perhaps, two decimal places, which would give the iodine value very nearly. With regard to the slight change of temperature when bromine was mixed with chloroform, he did not know whether there was such a change when it was mixed with tetrachloride of carbon, and, if not, it might be advisable to use the latter solvent in some cases. The more experience he had of Mr. Hehner's test the more highly he thought of it. In reply to Mr. Lott, he had only used pure methylated chloroform in his experiments, and he had not observed that any difference in the results was caused by the varying initial temperature, which was that of the atmosphere. In reply to Dr. Clowes, Mr. Hehner had tested a large number of oils, and in nearly every case the results came out very well. In a paper recently read before the London Section, blown oils and Japanese wood oil had been shown to be exceptions. Mr. Wood had asked about fish oils. With these he had not made any experiments, but he did not see why the process should not be applicable.

Mr. Hehner, in reply to a question as to whether all that was necessary was to have excess of bromine, said that this factor was undoubtedly an arbitrary one, and the whole process must be worked out under the same conditions. If the proportions were varied, although the amount of heat produced might be the same, the amount of measurable heat produced was not the same. He had tried pretty well every oil he could lay hands on. He had not had Japanese wood oil, and should like the opportunity of trying it. He had had a few samples of fish oil, and found them come out very well. Neither was that nor any other process capable of saying what the adulterants in fats were. It could only say that if a certain fat gave abnormal values with the test, they indicated some adulteration. Some fats, as was well known, gave a definite reaction. If two fats, neither of which had a definite reaction, came together, one could at most form some vague inference what were there.

Scottish Section.

Chairman: John Clark.
Vice-Chairman: J. B. Readman.

Committee:

A. C. J. Charlier.	A. Macdonald.
R. Cox.	R. C. Menzies.
C. A. Fawsitt.	E. Ostlere.
J. Gray.	Sir R. Pullar.
D. Harris.	H. Rose.
G. G. Henderson.	P. Rottenburg.
R. A. Inglis.	D. R. Steuart.
W. I. Macadam.	R. T. Thomson.

Hon. Secretary and Treasurer:
Thomas Gray, Technical College, 204, George Street, Glasgow.

The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Chairman*: G. T. Beilby. *Vice-Chairman*: G. G. Henderson. *Committee*: John Clark, Robt. Irvine, J. B. Readman, and E. C. C. Stanford.

SESSION 1896–97.

Meeting held at Glasgow on Tuesday, March 30th, 1897.

DR. JOHN CLARK IN THE CHAIR.

THE ANALYSIS OF WORTS, BEERS, AND OTHER BREWING MATERIALS: 1. ESTIMATION OF THE SOLID EXTRACT OF WORTS AND BEERS. 2. ESTIMATION OF THE SUGARS.

BY WILLIAM FREW, PH.D. (MUNICH).

The necessity of adopting certain standard methods for the analysis of the raw materials and of the finished products of any manufacture is being day by day more generally recognised. Unless two distinct methods can give absolutely identical figures, it is useless comparing the results obtained by those methods. In all statements of results, chemists should indicate clearly what methods have been used, as by this means many misconceptions would be prevented, and the analyses rendered of much greater value.

In no department of analytical chemistry is this more necessary than in that of worts and beers, and, I regret to say, in no country is a reform in the methods of stating results more urgently required than in Britain. It is often impossible to tell from the reports of brewing chemists how the various determinations have been carried out. The necessity for uniformity in methods and the statement of analytical results has long been recognised on the Continent, so that it is possible in most cases to compare results of various workers.

There is at present one set of standard methods which is generally recognised by the brewing chemists of Austria-Hungary and Germany, and which has also been adopted by the majority of brewing chemists other than those of Great Britain. In regretting that our chemists do not accept this very general standard, I do not for one moment insinuate that the methods they prefer are less accurate; but, in a case of this kind, we are less concerned with *absolute* accuracy as with *comparable results, i.e.*, we do not wish to know absolutely how much maltose, for example, there is in a certain wort, but, rather, how the amount found by a given method compares with that in another wort determined in an exactly similar manner.

The standards which I have adopted in my own practice are those proposed by the convention of brewing experts at the International Exhibition of Forestry and Agriculture held at Vienna in 1890. As I have said before, these are adopted by all the brewing chemists in Germany and Austria, and as the results published in those countries so enormously exceed those recorded in this country, it is much more valuable to me to be able to compare my own figures with the former than with the latter.

I do not intend to enter into the details of those standard methods at present. I would only remark, in passing, that both of the following short notes have a certain bearing upon what I propose to communicate in the future.

1. *The Estimation of the Solid Extract of Worts and Beers.*

The great difficulty which has up to the present been experienced by chemists in the direct estimation of the solid extract of such saccharine liquids as wort and beer, is that of rendering the solids left on evaporation anhydrous. It is practically impossible to get rid of the last traces of moisture in the sticky mass obtained by evaporating a wort or beer when one attempts to do this in the ordinary way by heating in a water- or air-bath, and even when one dries *in vacuo* in presence of a dehydrating agent the process is a tedious one. Owing to this circumstance the indirect method of deducing the dissolved extract from the specific gravity of the liquid in question has been generally adopted, and various factors and tables are in use for converting the specific gravity formed into percentage of solid extract present. In our own country O'Sullivan adopted the divisor 3·85 for the purpose of converting specific gravity into grammes of extract per 100 c.c. Thus water being taken as possessing a gravity of 1,000 at 15·5° C., he assumed that the specific gravity of a solution of starch-conversion products increased 3·85 for every gramme of solid extract per 100 c.c. of the solution. Thus a liquid containing 10 grms. of solid extract in 100 c.c. at 15·5° C. was assumed to have a density of 1038·5, so that the weight of solid extract present in 100 c.c. of any such solution could be found by dividing its weight above water by 3·85 (C. O'Sullivan, Trans. Chem. Soc. 1876, ii., 130).

Brown and Heron (Trans. Chem. Soc. 1879, **35**, 596) used a constant divisor 3·86 in a similar manner; this was derived from the relation between the density of a cane-sugar solution of sp. gr. 1,055, and the number of grammes of that substance present in 100 c.c. of the solution.

The last-mentioned factor has been, I believe, generally used by British brewing chemists since that time, for the purpose of converting solution density into grammes of extract per 100 c.c. On the Continent a similar method has been generally adopted, and the specific gravity tables of Balling are used to convert solution density into percentage of solid extract present in worts and beers.

Both of those methods are, however, merely arbitrary, as they express the solid extract in terms of cane-sugar—not in terms of actual extract present.

Various tables have been drawn up to show the relationship between the real extract present in worts and beers and the specific gravity. The Schultze-Ostermann tables for the estimation of the solid extract in clear decoction and infusion worts and in de-alcoholised beers were obtained by determinations of the total solids dried at 70°—75° C. under ordinary pressure. The figures thus obtained, however, are too high, as maltose does not part with all its water of crystallisation under such conditions. H. Ellion (Zeits. f. angew. Chem. 1890, 291—298 and 321—326) got over this objection by drying the solids left behind on evaporation at 97° C. in a vacuum. Under these circumstances the extract may be rendered anhydrous, although the process of drying is a very lengthy one.

It will be seen from what follows that the last-mentioned tables represent, with a very considerable degree of accuracy, the amount of solid extract present in worts and beers of widely varying origin. My attention was, however, drawn to the *direct* method of estimating solid extract by the extremely interesting and valuable series of papers on the products of the conversion of starch by diastase recently published by Brown, Morris, and Millar (Trans. Chem. Soc., **71**, 72). In the first of these papers the authors describe the apparatus used by them for the determination of the solid extract of saccharine fluids. This consisted of "two small flasks united by a tube furnished with a stop-cock and a side tube leading to a good air-pump. The substance to be dried is introduced into one of the flasks, the other being partially filled with phosphoric anhydride. After exhausting the apparatus, the flask containing the substance is immersed in a water, salt-water, or oil bath, according to the final temperature required, and is slowly heated up to a point at which it ceases to lose weight." The authors state that by means of this apparatus crystallised hydrated maltose may be rendered completely anhydrous in a few hours at 105°—106°. The dextrins, maltodextrins, and mixed starch-transformation products require much longer drying and temperatures as high as 120°—130° C. before losing the whole of their water.

It struck me that, if one could by a similar process obtain the desired results more quickly, such a method would prove of great value to the commercial analyst in general as well as to the brewing chemist in particular. I have been fortunate enough to devise a very rapid method of estimating the anhydrous extract present in worts, beers, and solutions of starch-transformation products, which also lends itself to the estimation of the dry matter in such substances as glucose syrup and the other preparations of

glucose used by brewers, as well as for the estimation of moisture in samples of cane and other sugars.*

This process is a modification of that described by Brown, Morris, and Millar, in the paper previously referred to. The drying apparatus consists of two large wide-mouthed bottles, provided with double-bored rubber stoppers, and communicating with one another by a tube. One of these bottles is immersed in a glycerine bath, so that it may be heated up to any desired temperature, and is connected by means of a short length of rubber tube to a wash bottle containing concentrated sulphuric acid. The other bottle contains a small quantity of concentrated sulphuric acid, and is connected with an efficient filter pump. In carrying out an estimation of the solid extract in a wort or beer, the following is the procedure:—

Wide-mouthed stoppered weighing bottles are provided with coils of filter paper, such as are used for the estimation of fat in milk, and are then heated in an air-bath at 110°—120° C. till constant. Into these are then run suitable quantities of the worts or beers, after which they are at once weighed, the increase of weight being the amount of liquid taken. Of course, no more liquid should be taken than can be totally absorbed by the paper coil, the limit being conditioned by the size of the coil. After weighing, the open bottles are placed for an hour in a drying oven at 100° C., after which they are transferred to the drying bottle of the apparatus described above, the temperature of the glycerine bath having been previously raised to about 110°—120° C. The filter pump is now set in action and a slow current of dried air is allowed to pass through the apparatus, its rate being so regulated that a considerable degree of exhaustion is maintained in the apparatus. After an hour the supply of air is cut off and the apparatus is thoroughly exhausted. The rubber tube connecting the apparatus with the filter-pump is now closed by means of a screw-clip, and the whole is allowed to stand for another hour. Dry air is then admitted, the bottles are removed, allowed to cool in a desiccator, and weighed. They are then once more transferred to the drying apparatus which is again exhausted, and the whole is allowed to stand for an additional hour. The bottles are again weighed, and this process is repeated till no further loss of weight occurs.

In the case of glucose syrups, treacle, and golden syrup, and cane sugars, about 20 grms. of the sample are dissolved in distilled water and made up to 100 c.c. Of this solution an aliquot portion is withdrawn and the dry substance determined in the manner described.

I find that by this means one is able to render the solid extract from beers, worts, solutions of Lintner's soluble starch, solutions of glucose syrups, &c., anhydrous after two, or at the most three, hours' heating. There is, even in the case of solutions of soluble starch, practically no further loss of weight after two hours' treatment. The great rapidity with which such substances lose the last traces of moisture in this method, is due to the extremely fine film of extract distributed over the large area of filter-paper.

This method will, I think, prove useful in other ways. Thus, to give one example, it is sometimes necessary to determine the amount of yeast formed during the fermentation of some saccharine liquid, or to determine the percentage of dry matter in a given sample of moist yeast. In the first case this may be done by making the liquid (after fermentation) up to a definite volume, and determining the dry matter in an aliquot portion by the above method. A portion of the liquid is then filtered clear and the dry matter determined in the filtrate, when the difference between the two results will give the weight of dry yeast present. If we know how much yeast we originally added, we can thus determine the amount formed during the fermentation. In the case of moist yeast a weighed portion may be thoroughly mixed with sufficient water in a drying bottle, the paper coil dropped in, and the whole dried as above at a suitable temperature until of constant weight.

* Since this paper was read, the author's attention has been drawn to the description by C. N. Rieber (Zeits. f. d. ges. Brauw. **13**, 271) of a similar method. As the latter, however, used a temperature of only 70° C. constancy in weight was only reached after a very prolonged drying, in some cases 66 hours being required.

Before concluding this note, I should like to give a few determinations of solid extract in worts and beers, to show how closely the figures given in Ellion's tables correspond with the real extract present:—

Description.	Specific Gravity $\frac{15\cdot5°}{15\cdot5°}$	Extract, per Cent.		
		Ellion.	*Brown, Morris, and Millar.	Found.
Stout wort	1·08724	20·18	20·29	19·89
All malt wort	1·06398	15·08	15·17	14·89
Malt-rice wort	1·03526	8·51	8·59	8·42
Light all-malt wort	1·01516	3·72	3·72	3·77
Last runnings of all-malt mash.	1·00578	1·41	1·45	1·22
Dealcoholised beer	1·0094	2·32	2·34	2·11

* These figures were obtained by using the appropriate divisor for a corresponding starch conversion. This, of course, is not strictly correct, as Brown, Morris, and Millar's divisors refer to the products of pure starch conversions; but, as can be seen from this table, they give closely approximating results in the case of hopped malt extracts as well.

To return now for one moment to the indirect method of estimating the solid extract in worts and beers.

As I have already pointed out, both Balling's tables and the use of the 3·86 divisor give, not the real extract, but the weight of cane-sugar present in a solution of that substance of a similar density; of these two methods, the use of Balling's tables is preferable, as they are computed from the amounts of cane-sugar present in solutions of varying gravity, whereas the 3·86 divisor only expresses the relation between what we may call excess gravity (water being taken as = 1,000) and weight of cane-sugar present in a solution of sp. gr. 1,055.

Ellion's tables, on the other hand, express, with a very considerable degree of accuracy, the real dry extract present in a wort—especially when this is an ordinary hopped or unhopped wort which has not been fermented. The most serious discrepancies between my results and the figures obtained by reference to Ellion's tables are in the cases of the last runnings and of the de-alcoholised beer. In both of these, however, the dissolved extract is of an abnormal character, consisting chiefly of dextrins, albumin, and mineral matter.

The low figure obtained in the case of the stout wort is probably due to the volatile matter present in black malt.

2. *The Estimation of the Sugars.*

In no department of analytical chemistry is uniformity of method more to be desired than in the estimation of the sugars by means of Fehling's solution.

Of the two methods of estimating sugars by Fehling's solution, the volumetric cannot be considered so far as scientific accuracy is concerned; if even approximately correct and comparable results are wished, it must be employed under strictly defined conditions, to which I will refer later on. In the same way the results obtained by weighing the precipitated cuprous oxide are only comparable and really accurate when the precipitation is carried out under certain fixed conditions, and the results interpreted by means of tables constructed for this purpose. These two facts were first pointed out by Soxhlet (Journ. f. prakt. Chemie, **21**, 227), who showed that the reduction factor between copper and sugar is not constant but variable, and is dependent upon (*a*) the concentration of the two reacting fluids, (*b*) the amount of copper present. There is, in fact, no stoechiometric regularity in the reaction, and this, of course, condemns any volumetric method where varying quantities of Fehling's solution are added to a sugar solution of varying strength. Soxhlet's results also show the inaccuracy of using a fixed factor for the conversion of precipitated copper into sugar present.

Maercker (Chem. Centralblatt, 1878, 584) showed that the alteration in the reduction factors, caused by the relative excess of copper, is quite regular, and so, by allowing

varying quantities of the sugar in question to act upon equal quantities of Fehling's solution under certain fixed conditions, definite factors might be obtained, from which tables can be constructed allowing the corresponding quantity of sugar to be read off from the weight of copper found.

Such factors have been determined by several Continental chemists for the various sugars, and their results have been collected by Dr. Ernst Wein, of Munich, whose "Tabellen zur Quantitativen Bestimmung der Zuckerarten" are almost universally used by brewing chemists in Germany and Austria. The English edition of these tables, published by Spon, represents my small effort in the direction of uniformity of method in this important estimation.*

The following is a short description of Wein's method :—

The specified quantity of Fehling's solution is heated in a porcelain basin (holding about 250 c.c.) to boiling, when the prescribed volume of the sugar solution is added to the vigorously boiling liquid, and the whole is allowed to boil for the specified time. In no case should the sugar solution exceed 1 per cent. in strength. The Fehling's solution used in most of the determinations contains 34·639 grms. of crystallised copper sulphate, 173 grms. of pure Rochelle salts, and 51·6 grms. of sodium hydrate per litre ; no other Fehling's solution than that specified for the sugar in question should be used.

The precipitated cuprous oxide is then filtered off through a previously tared Soxhlet's asbestos filter-tube fitted on to a filter-flask connected with a vacuum-pump. The filtration and washing with hot water are done as quickly as possible, in order to prevent any cuprous oxide redissolving. After washing sufficiently, alcohol and ether are passed through the filter, in order to facilitate drying. The filter-tube (still in connection with the vacuum-pump) is heated in the Bunsen flame until the cuprous oxide is oxidised to cupric, after which the layer of oxide is reduced to metallic copper in a stream of dry and pure hydrogen. The tube is then weighed, and from the weight of copper found its equivalent in sugar may be ascertained by referring to the table constructed for the special sugar.

The filter tubes are prepared by first of all pushing down a plug of glass wool to the bottom for the asbestos layer to rest on. On to this plug finely-divided asbestos, which has been specially prepared by boiling respectively with nitric acid, strong caustic soda solution, and water (in which it is kept suspended for use), is poured until a sufficient layer is formed. This layer is pressed down by means of a glass rod flattened at the end, and is then washed with hot water. Alcohol and ether are passed through to facilitate drying, and the tube is heated for some time in a stream of hydrogen, cooled and weighed. An alternative method of estimating the copper precipitated is to filter off the cuprous oxide through a double filter-paper, wash with boiling water, alcohol and ether as before, and dry. The precipitate is now detached as completely as possible from the filter and shaken on to a piece of glazed paper. The filter papers are then incinerated in a crucible, the ash being reduced to a fine powder by stirring with a platinum wire. After this the cuprous oxide is transferred to the crucible and oxidised to cupric oxide at a low heat, the flame being increased for a few minutes at the close of the operation. It is necessary by means of a few blank experiments, to ascertain how much copper is absorbed by the filter papers—this, for an ordinary "ash-free" filter paper of 9 cm. in diameter, amounts to about 3 mgms. of cupric oxide for 50 c.c. of boiling Fehling's solution passed through. This method gives concordant results with the Soxhlet tube method when carefully carried out, and may prove useful where it is found difficult to obtain suitable asbestos. The Soxhlet method is, however, much more expeditious.

Brown, Morris, and Millar (Jour. Chem. Soc. 1897, p. 105), and Ling and Baker, have found that Wein's maltose table gives results which are about 5 per cent. too low. As the factors from which this table was calculated were determined in 1885, it is quite possible that the maltose used was not so pure as that prepared by the English workers. It is my intention, when my other work permits of it, to redetermine the factors for the various sugars, taking all possible precautions to obtain pure samples.

It is nevertheless well to remember that this alleged error does not affect the comparison of results in which Wein's tables have been used, and, as I have said before, that is the more important consideration for the analytical chemist. Owing to the method employed in constructing the tables, they are correct within themselves, and if there is an error due to the use of an impure sample of the sugar in question, it will be a constant one throughout the table.

As I said before, the volumetric method as usually carried out is valueless in point of accuracy. There is, however, one mode of working by which approximately accurate results can be obtained.

A solution of the sugar to be determined is made up containing, in the case of dextrose, not more than 0·58 grm. per 100 c.c., in that of maltose not exceeding 0·88 grm.

Into each of a series of small test tubes, 5 c.c. of the solution is run, and to these varying quantities of Fehling's solution are added. The tubes are placed in boiling water for a specified time, after which they are examined to see in which tube all the copper has been reduced. We thus obtain two limits between which we once more arrange a series of tests, until at last we arrive at a point where the difference between two tubes does not exceed 0·01 c.c. From the volume of Fehling's solution used, we can ascertain from the tables what quantity of sugar is present in 5 c.c. of the solution with which we are working.

I have given this short description of a comparatively old method, firstly, because so little seems to be known about it in this country, and, secondly, because it is the standard method recommended by the Vienna Congress in 1890. The gravimetric method as described by Wein, and the tables calculated by him, are used in practically every scientific station for brewing in Germany and Austria, and it is according to them that the maltose figures of any analyses in my future communications will be obtained.

My earnest desire is that British brewing chemists may soon adopt the Vienna standards, so that analyses, so important to the brewing scientist, may be comparable from whatever part of the civilised world they come.

* "Tables for the Quantitative Estimation of the Sugars." (Wein.) E. and F. N. Spon, London and New York.

Discussion.

Mr. Beilby asked the author how the results obtained by drawing a current of hot air over the residue, spread in a thin layer over filter paper, compared with those obtained by drying *in vacuo*. He remarked that Dr. Frew had supplemented his drying operation in a vacuum, by passing a current of hot air over the residue, and asked whether the author had found that necessary.

Mr. Gray remarked that he thought the author had placed the volumetric method for estimating the sugar in a somewhat unfavourable light, and asked if Dr. Frew had not found it possible to obtain reliable and comparable results, by carrying out the estimation of the sugar under precisely the same conditions as had been observed in standardising the Fehling's solution. He stated that the fact was well known that varying results were obtained, depending on the concentration of the Fehling's solution, the rate of addition, the time of boiling, and the concentration of the sugar solution ; but that the first of these conditions did not require to be considered in practice, as a Fehling's solution of a definite strength was always used, while the only one of the other factors which was of necessity variable was the last named, and that within comparatively narrow limits. He thought that, with strict adherence to rules laid down in connection with the rate of addition and the time of heating between each addition, comparable results might be obtained by different brewing chemists.

Dr. Frew acknowledged that it was possible to get fairly approximate and comparable results under the conditions laid down by Mr. Gray. His objections to the volumetric method employed in this way, however, were, that it was extremely difficult always to add the sugar solution to the

Fehling's solution at exactly the same rate, and that it was necessary for each chemist to determine the factors for his own particular method and make up a table therefrom. He regretted that he had not himself compared the relative speed of drying in vacuo, and by passing a stream of hot air over the substance in question, but he believed that Ellion in his paper had stated that by passing a current of dry hot air through his apparatus the desiccation was rendered more rapid. Brown, Morris, and Millar said that by their method of simply evaporating and drying the sticky mass in vacuum a very considerable time was required to expel the last traces of moisture. There was one advantage in using this modification of their method. Several determinations could be made simultaneously by using a drying bottle large enough to contain three or four weighing bottles, whereas in theirs only one could be made at one time. His modification, therefore, made the process as convenient as the ordinary hot-air oven.

Obituary.

HUGO KUNHEIM, Ph.D.

COMMERZIENRATH OF THE CITY OF BERLIN, MEMBER OF THE SOCIETY OF CHEMICAL INDUSTRY, &c.

In the removal by death of Dr. Hugo Kunheim, Germany has lost one of the foremost among those who have made the German chemical industry what it is. About 65 years ago the father of Dr. Kunheim opened a chemical factory, the business of which grew and prospered remarkably. This factory eventually came under the proprietorship of Dr. Kunheim, who succeeded his father, and under his management enormous extensions were made. He was interested in almost every department of chemical industry, and his firm was the first to establish the manufacture of many important chemicals on German soil. Dr. Kunheim was, however, more specially associated with the manufacture of sulphuric acid, ammonia, borax, liquid carbonic acid, oxalic acid, and potassium salts. He was the first German manufacturer of "yellow prussiate of potash" from gas-liquor, and was Chairman of the Convention of Yellow Prussiate Manufacturers. Dr. Kunheim was also the pioneer of the Sicilian citrate of lime industry. In the year 1873 the city of Berlin purchased his factory on the Kreuzberg for six million marks, the site being required for the extension of the city. Dr. Kunheim was a prominent figure in Berlin society, and in Continental chemical circles generally. It is said that he died very wealthy, and he is known to have been financially interested in about a score of large chemical and electrical undertakings on the Continent. Having suffered for a considerable time from a gradual breakdown of the nervous system, the end at last came on the 22nd ult. Dr. Kunheim was about 62 years of age, and he leaves two sons.

Journal and Patent* Literature.

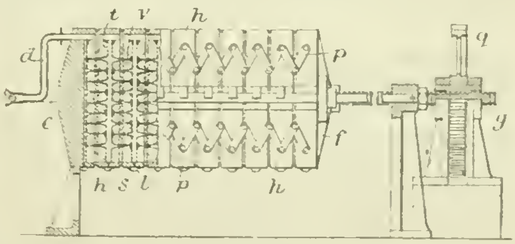
* Any of these specifications may be obtained by post by remitting 8d.—the price now fixed for all specifications, postage included—to Sir Henry Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

I.—PLANT, APPARATUS, AND MACHINERY.

PATENTS.

Filter Presses, Impts. in. W. E. Heys, Market Street, Manchester. From J. Hundhausen, Hamm, Westphalia, Germany. Eng. Pat. 6873, March 30, 1896.

The press is built up of a number of frames *h*, mounted on lazy-tongs *p*, and connected at the joints by india-rubber packing. Each frame is provided with perforated metal filter plates *t*, which are formed with projecting arms *v* and are covered with filter cloth, the arms of adjacent frames being arranged to break pitch, as shown.



The press ram *f* is operated by the pinion *q* mounted on the screwed spindle *g* of the ram. The liquid is admitted through the pipes *d*, *c*, and, after passing through the filter cloth and plates *t*, it flows off through the passages *s* and gutters *l*. The reversing gear of the driving mechanism is controlled automatically by the movement of a safety-valve lever.—R. A.

Filtering Material, and Process for Making the Same, An Improved. F. Jurschina, L. Schnitzer, and F. Hirsch, all of Vienna. Eng. Pat. 6934, March 30, 1896.

The material consists of ground quartz cemented together by insoluble silicates and silicic acid, so as to leave pores between the granules. The process consists in "wetting with water-glass a mixture of ground quartz and wood ashes, pouring or pressing the mass into moulds, where it is allowed to harden, drying the moulded hardened bodies, heating them to a temperature of from 1,000° to 1,200° Celsius, and finally boiling them in water."—V. C.

Mixed Gases, Separating by Refrigeration, Apparatus for: especially applicable to the Separation of Oxygen from Air. W. Hampson, Mold, North Wales. Eng. Pat. 7559, April 9, 1896.

The refrigerating apparatus described in this Journal, 1896, 340 (in which compressed gas is allowed to expand and produce cold, which is intensified by interchange of heat between the compressed and expanded gases), is employed in conjunction with additional apparatus, in which the mixed gases, supplied under pressure, are passed through a reducing valve, and are liquefied at the reduced pressure by the cold generated in the apparatus. The liquefied gases are allowed to boil, thereby cooling the chamber in which the liquefaction takes place, so that a further liquefaction occurs, and simultaneously a separation of the gases is obtained by boiling off one of

them from the mixture. The gas boiled off is also employed to reduce the temperature of the incoming gas.
—R. A.

Producing Cold by Means of Volatile Liquids, Impts. in Apparatus for. M. Douane, Paris. Eng. Pat. 9127, April 30, 1896.

To prevent leakage of the volatile liquid from the compressing pump, the piston-rod works through a double stuffing-box, within which the rod is surrounded by a liquid enclosed in a chamber, this liquid forming a hydraulic joint with the rod and also serving to lubricate it. "In a single-acting compressing pump having its crank revolving in a closed chamber, a double stuffing-box for the crank shaft," is also claimed. To provide a greater refrigerating surface in the refrigerating vessel in which the liquid is volatilised, the vessel is made of annular form, by means of two concentric tubes. In another arrangement, the vessel consists of a number of horizontal superposed tubes communicating with each other by vertical branches at the ends. "For obtaining ice directly on the surface of the refrigerating vessel without the use of brine, a vertical tubular vessel is employed"; the volatile liquid is introduced through a small tube against the inner surface of the refrigerating vessel, so that it flows down in a thin film over the same and is evaporated before it reaches the bottom. For detaching the ice from the vessel, warm water is introduced into a tank surrounding the coil through which the volatile liquid is delivered, the heat being conveyed by the volatile liquid to the vessel, so as to warm the latter and detach the ice. — R. A.

Liquids, Impts. in and relating to Apparatus for Heating, Cooling, and Vaporising. V. C. J. Ortmans, Brussels. Eng. Pat. 10,338, May 14, 1896.

The liquid to be heated, &c., is passed through a series or cluster of tubular elements, each element consisting of tubes having concentric sides, and constructed so that the heating or cooling medium may circulate around these sides. The orifices connecting the interior surfaces with the exterior may extend nearly the entire length of each element. In one form of element, two tubes are arranged concentrically, the inner tube being connected at its ends with the exterior surface of the outer tube. In another form, the tube is single at its ends, and is bifurcated along the greater part of its length, so as to form a passage between the branches for the circulation of the heating or cooling medium.—R. A.

Gas Vessels or High-Pressure Gas Reservoirs. W. C. Tyler, Waterloo Road, London. Eng. Pat. 24,483, Nov. 3, 1896.

The sides of the vessels or reservoirs are corrugated, to render them capable of withstanding a higher pressure without materially increasing their weight or diminishing their capacity.—R. A.

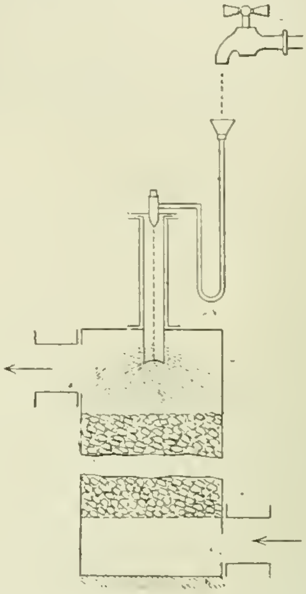
Ammonia and other Volatile Liquids [Refrigerating Apparatus], Safety and Regulating Appliance for Apparatus for Compressing Vapours of. T. B. Lightfoot, 35, Queen Victoria Street, E.C. From The Gesellschaft für Linde's Eismaschinen, Wiesbaden, Germany. Eng. Pat. 853, Jan. 12, 1897.

The compressing cylinder is connected to a closed vessel or chamber containing a safety valve, which is covered by a non-volatile liquid, such as oil, so that when the valve is forced open, a quantity of the liquid escapes through it, thereby preventing the escape of the volatile vapour which is being compressed. The liquid which escapes is returned to the chamber by the action of the compressing piston. The "dead space" of the compressor can be eliminated by introducing liquid into the chamber. The connection between the cylinder and chamber is provided with a valve, so that when the chamber is empty, the apparatus can be thrown in or out of work.—R. A.

II.—FUEL, GAS, AND LIGHT.

Scrubber Sprinkling. F. Burgemeister. J. für Gasbeleucht, **40**, [6], 85—86.

The device used by the author for distributing water uniformly over the packing material of gas scrubbers is shown in the accompanying figure. To an opening in the middle of the scrubber top is connected a pipe about 1 m. long and 100 to 150 mm. wide. This supports, by means of two rods, a convex disc about 80 mm. wide. The disc has its



convex side uppermost and is about 250 mm. below the top of the scrubber. The water falls from the usual funnel and siphon pipe on to the disc, and is thereby broken into very fine spray. A scrubber packed for three-fourths of its height with pebbles showed no accumulations of tar after seven years' use under this system of water-distribution. One spraying disc serves for a scrubber 2 metres in diameter.—J. A. B.

Illuminating Gas, Combustion of, in contact with Cooled Surfaces. F. Haber and A. Weber. Ber. **29**, 3000—3006.

The combustion of illuminating gas in flames, which neither smoke nor flicker, or against white hot surfaces, as in the Welsbach burner, is complete, *i.e.* the combustion products either contain no combustible gases, or contain them only in mere traces. On the other hand, Lewes has shown that the combustion products of gas burning against cooled surfaces (as in gas cooking apparatus, water-baths, &c.), contain considerable quantities of combustible gases. The authors are of opinion that Lewes' experiments were not suitably arranged, and they have therefore studied the question afresh. They examined the combustion products obtained from a large Bunsen burner (burner tube 16 mm. wide, modified as described by Teclu) when working with varying proportions of primary air, and with its mouth placed 18 mm. from the bottom of a tin vessel kept cool by flowing water. Around this vessel a metal strip was soldered to form a V-shaped groove, which acted as a constantly refilling gas-reservoir and from which the combustion products could be collected without prejudicing the admission of air to the flame. The gas drawn from this space, after drying by means of sulphuric acid and phosphorus pentoxide, was led through a weighed carbonic acid absorption apparatus. Thence the gas passed through a white-hot capillary tube (Drehschmidt's method), and then into a series of absorption vessels, in which the newly-formed carbonic acid and water were separately retained. The residue of gas was collected and measured.

The results are given in Table I. (Experiments I., II., and III.), from which it is seen that the determinative factor is the primary air content. The latter part of the table gives the corresponding figures obtained with cooking-stoves of various makes. Of these results, those obtained with the Bouchier et Cie.'s hearth are noteworthy. The construction of the latter is such as to prevent the admission of a sufficient supply of secondary air to the flame, and this causes a considerable alteration in the degree of incompleteness of the combustion and the atomic relation of C : H. The conditions here appear to be the same as those under which Lewes obtained his results. This deviation vanished when the distance between the burner and cooling vessel was increased. From an analysis dependent chiefly on fractional combustion, the author finds the composition of the combustible portion of the combustion products, to be—

CO.	H.	C_2H_2	CH_4	Content of Primary Air.
Per Cent.	Per Cent.	Per Cent.	Per Cent.	
0·3	0·04	0·01	0·015	31 to 32 per cent.

Direct estimation of the acetylene in the gases passing away from a cooled Bunsen flame, led to the following values :—

Primary Air.	Combustion Products.	
	CO_2	C_2H_2
Per Cent.	Per Cent.	Per Cent.
33·49	5·8	0·11
30·62	5·8	0·10
19·14	6·2	0·19
20·57	5·0	0·23

The presence of a cooled surface in the Bunsen flame therefore causes the combustion to be incomplete ; the cold surface reduces the temperature of certain of the combustible gas molecules to such an extent that oxidation will not occur when these molecules come into contact with the secondary air supply. The smaller and hotter the flame, the less proportionately is the influence of the cooled surface. The combustion zone is small and the motion of the gas molecules so energetic that after striking the cold surface they rebound into the flame ; the result of this is that the flame boundary is sufficiently hot to allow of complete combustion with the surrounding atmospheric oxygen.

With a decreasing primary air supply and a larger flame, the reactionless zone increases in depth and also spreads horizontally. The gas particles do not rebound so readily into the flame, and the number reaching the flame surface too cold to burn, increases.

Thus, appreciable quantities of combustible constituents appear amongst the products of combustion ; even completely unaltered gas constituents, such as methane, being found under these unfavourable conditions. There is always present, even before cooling below the combustion temperature, sufficient oxygen to convert this hydrocarbon into carbonic oxide and hydrogen ; this agrees with facts as to combustion proceeding by steps, as explained by Smithells and others. The combustible constituents of the flame gases contain much carbonic oxide and little hydrogen ; this is due to the fact that hydrogen burns at a much lower temperature than carbonic oxide, as shown by Mallard and Le Chatelier.

From the practical and hygienic standpoint, the following are the results obtained :—

Flames striking a cold surface give rise to hygienically important quantities of carbonic oxide only when the primary air supply is small ; when this is high, only minute traces are produced. The loss in heating power due to incomplete combustion with small primary air supply is trifling: even with a high degree of incompleteness of combustion

TABLE I.

No.	1. Gas Consumption in Litres per Hour.	2. Gas Pressure in mm. Water.	3. Primary Air Content.	4. CO_2 in the Combustion Products estimated by the burette.	5. CO_2 before the Combustion Apparatus.	6. CO_2 after passing the Combustion Apparatus.	7. Carbon.
			Per Cent.	Per Cent.	Grms.	Grm.	Grm.
Bunsen burner with regulated air supply. (Teclu) I.	278	30	79·57	..	2·8411	0·0189	0·0013
II.	275	29	66·50	5·8	2·7894	0·0966	0·0263
III.	277	29	21·77	6·7	3·2481	0·2573	0·0702
Friedr. Siemens and Co., D.R.P. 78,156.	152	30	76·12	7·4	2·8510	0·0256	0·0070
Junker and Ruh, D.R.G.M. 25,681 .	232	20	74·36	6·2	2·7810	0·0113	0·0031
Leclerq, Fontenau, et Cie, Paris ...	..	19	58·28	..	2·8050	0·1766	0·0460
Boucher et Cie., Fumay............	225	..	51·63	..	2·9729	0·6316	0·1722
" "	225	..	51·63	..	3·1656	0·2209	0·0602
Blank experiment with air.........	..	..	..	..	0·0420	0·0043	..

TABLE II.

No.	8. H_2O after the Combustion Tube.	9. Hydrogen. H.	10. Volume of Residual Gas at 0° and 760° Mm.	11. CO_2 in Combustion Products calculated from 5 and 10.	12. Incompleteness of the Combustion.	13. Atomic Ratio. C : H.	14. Percentage of Unburnt, calculated from CO_2(6)=CO.
	Grm.	Grm.	C.c.	Per Cent.			
Bunsen burner with regulated air supply. (Teclu) I.	0·0061	0·0007	21,595	6·3	0·66	..	0·4
II.	0·0091	0·0010	21,413	6·2	3·35	1 : 0·4601	2·3
III.	0·0432	0·0048	25,580	7·6	7·64	1 : 0·8206	7·3
Friedr. Siemens and Co., D.R.P. 78,156.	0·0032	0·0004	18,099	7·4	0·89	..	0·7
Junker and Ruh, D.R.G.M. 25,681 .	0·0052	0·0006	19,770	6·7	0·40	..	0·3
Leclerq, Fontenau, et Cie, Paris	0·0224	0·0025	19,184	7·0	5·92	1 : 0·6228	4·7
Boucher et Cie, Fumay	0·2100	0·0233	20,458	7·8	17·52	1 : 1·6254	15·7
" "	0·0330	0·0037	20,819	7·7	6·52	1 : 0·7323	5·5
Blank experiment with air.........	0·0050	..	24,912	..	..	..	..

By the degree of incompleteness (12) is understood the percentage by weight of CO_2 behind the Drehschmidt capillary calculated on the total carbonic acid = 100. The values in the last column are obtained by considering the CO_2 behind the capillary as being formed from CO.

(= 7) (see note to Table II.), in an illuminating gas giving 600 litres of carbonic anhydride and having a heating value of 5,000 cal. per 1 cb. m., the loss due to this cause is under 3 per cent. The poor economic effect of such combustion is due to the lower flame temperature and to the deposition of soot and tar on the cold surface and the consequent decrease of heat-conducting power.
—J. T. C.

Gas, Carburetting by means of Benzene. Hoyer. J. de l'Eclair. au Gaz, **45**, [3], 51—53.

Continuing the experiments detailed by Pettré (J. de l'Eclair. au Gaz, 1896, 27) in order to determine the cause of the variations in the rate of enrichment obtained by the use of benzene, it was found that the quantities of that substance required to produce a saving in consumption of 1 litre of gas, commencing with a 109-litre gas, were as follow :—

Lighting power............	109	108	107	106	105	104	103
No. of grms. of benzene required per cb. m. to gain 1 litre.	0·9	0·9	1·0	1·0	1·1	1·1	1·2
Lighting power............	102	101	100	99	98	97	96
No. of grms. of benzene required per cb. m. to gain 1 litre.	1·3	1·4	1·6	1·7	1·9	2·2	2·7

i.e., to reduce the consumption from 108 to 103 litres, 5·1 grms. of benzene were needed. The reason for the decreased effect recorded below 104 litres, is due to the inability of the Bengel burner, which was made for consuming 105 litres, to develop the true power of the gas under conditions of slower delivery. The results cited cannot be applied for comparison with those obtained in other forms of photometer than the one (that of Dumas and Regnault) employed.—C. S.

Coal-Gas, Purification of; The Use of Atmospheric Air with Lime for. S. Carpenter. J. of Gas Lighting, 1897, **69**, [1765], 527.

The author states that :—(1.) Air should not be allowed to pass up the ascension pipes or go into any part of the hydraulic main. (2.) It should not be admitted at the inlet of the condensers. (3.) Nor should it be allowed to get into the lime purifiers, as it interferes with the chemical action of the lime and the gas. The air oxidises the sulphuretted hydrogen and at the same time fixes the sulphur in the lime. If air be admitted into the purifiers to the extent of (say) 1·5 to 2 per cent., the lime is converted into calcium sulphide, which has very little affinity for bisulphide of carbon. The lime should be converted into "hydrogenated" sulphide of calcium (calcium hydrosulphide), which has, in a high degree, the power of absorbing carbon bisulphide from coal-gas. If air, or any furnace gas, or the gas at the end of the charge where anti-dips are used, be admitted into the lime purifiers just before a box is thrown out of action, the impurities which the lime has already extracted from the gas will be evolved and driven forward with the gas which is being made.

If care be taken that no atmospheric air is admitted either directly or indirectly into the lime purifiers, there will be no trouble with them, and the carbon bisulphide in the gas will keep to about 10 to 15 grains per 100 cb. ft.

Atmospheric air, to the extent of 1 to 1·5 per cent., should be admitted at the inlet of the first oxide of iron purifier, and if sulphuretted hydrogen make its appearance at the top of the first oxide purifier, a small quantity of air should be passed in at the inlet of the second purifier. In this manner, the oxide will last in the purifiers for months instead of weeks.—A. S.

Calcium Carbide, Manufacture of; A Furnace for the. L. Bullier. Rev. de Chim. Ind. 1896, **7**, [83], 340—341.

A rectangular structure of brick, three of the walls (upright) being permanently fixed, whilst the fourth is built up as required from loose bricks held in place by an iron plate. An iron grating, covered with a layer of coke, constitutes the sole (as also the negative electrode) of the furnace. The positive electrode consists of a bundle of carbon rods supported from, and sliding up and down, an iron pillar. The furnace is worked on interrupted circuit, *i.e.*, the positive electrode is raised (after the current has been turned on) until an arc of suitable length is obtained. Subsequently the furnace is charged at intervals with raw material as required, and its open side bricked up to keep the mass in place. In this system the production of carbide takes place within a comparatively limited area (*i.e.*, the confines of the arc), whereby, it is claimed, the action of the current is greatly intensified ; whilst the loss of heat is reduced to a minimum on account of the thick non-conducting layer of material, unacted upon, intervening, between the scene of action and the furnace walls. For the same reason the furnace need not necessarily be constructed of highly refractory materials.—H. T. P.

Calcium Carbide, The Cost of Production of. D. Korda. Rev. de Chim. Ind. 1896, **7**, [83], 341—343.

In Europe, the sale price of calcium carbide has, hitherto, not fallen below 300—500 francs per ton, and for smaller quantities the price is at least twice as high. As a consequence, the use of acetylene as an illuminant is restricted to one or two special purposes. The more favourable estimates which have been published from time to time as to the cost of illumination by acetylene, for the most part appear to be based on certain calculations furnished by the Willson Aluminium Co., Spray, N.C., as also by Mr. Willson personally, according to which the cost of production of calcium carbide is variously estimated at 75—100 francs per ton. These estimates, the author considers to be far too optimistic, and he draws attention to the high yield of carbide therein reckoned on (20 lb. per electrical horsepower per 24 hours). Indeed, Willson and Sukert say: . . . . "with improved furnaces . . . . utilising the waste heat of the furnace gases to the fullest extent . . . . &c., it is hoped to raise the yield, on the large scale, to 30 lb. per H.P.-day." Now, in no European installation has a yield of 4 kilos. (= 9 lb.) per H.P. been sensibly exceeded. Adopting this figure as substantially correct, the author estimates the cost of manufacture to be, at least, 250 francs per ton of carbide. The remaining data—which conform closely to the conditions obtaining in an existing installation—are subjoined. The available H.P. (hydro-electrical) is assumed to be 2,000, corresponding to a daily output of 8 tons of carbide.

	Francs.
Raw Materials:—	
5 tons of coke	62·50
7 tons of lime	112·00
Motive Power.—Assuming 75 fr. to be the annual (360 days) cost of 1 H.P., then 2,000 H.P. per diem	500·00
Electrodes.—10 fr. per ton..................	80·00
Salaries:—	
75 labourers at 3·50 fr. .... }	
25 „ „ 5·00 fr. .... }	425·00
6 foremen at 6·25 fr. .... }	
Repairs......................................	400·00
Packing, in barrels, at 1·75 fr. per 100 kilos.	140·00
General Expenses, about 100,000 fr. per annum—a low estimate	260·00
Daily expenses..........	1,979·50

or, in round numbers, 250 fr. per ton.

Under present conditions, the above figures are by no means exaggerated ; although it is probable that, as progress is made, the expenses relating to repairs, and especially in respect to the number of workmen required, will be reduced.—H. T. P.

Acetylene in 1896, Summarised Report on. Rev. de Chim. Ind. **8**, 8—12.

Whilst acetylene, in common with ordinary illuminating gas, will burn without exploding, and may be exploded by the combustion of detonating mixtures, it is also capable of explosion on decomposition; but in the gaseous state this decomposition has to be effected by mercury fulminate or a similar detonator. As regards its behaviour in the compressed and liquid conditions, there are as yet no reliable data to go upon.

The calcium carbide at present obtainable contains impurities of two kinds: those attacked by water and those—such as graphite, boride and silicide of carbon, and metallic silicides and carbides—unacted upon by that liquid, but reducing by their presence the gas-yielding capacity of the carbide, sometimes by as much as two-thirds. The active impurities—phosphides, nitrides, sulphides, &c.—yield products which, besides lowering the intensity of the acetylene flame, facilitate the action of the gas on copper.

Liquefied acetylene for lighting purposes is purer than the gas generated direct, but is more expensive, and not without danger. Berthelot and Vieille recently found that an incandescent metallic wire would decompose liquid acetylene, and in so doing develop a force approximating to that of guncotton, but that the shock due to a fall from a height of 6 metres caused an explosion of the liquid rather of the nature of combustion than of decomposition, whilst acetylene gas under 10 atmospheres pressure did not explode at all. Decomposition of the gas may also be caused by the heat generated when carbide is acted on by very small quantities of water, and local overheating may result in polymerisation. Compression and sudden checks may also generate sufficient heat to induce dangerous incipient decomposition, but the fracture of the cylinder containing the liquid is only likely to have serious results in case sparks are caused by the friction of the metallic fragments. These dangers, however, may be avoided with care, and should not detract from the practical value of the gas.

It has also been found that acetylene forms no stable compounds with the blood, and is therefore without any toxic action, though capable of producing asphyxiation; and in view of the completeness of the combustion of the flame, no toxic action may be expected from the resulting products.

The high illuminating power and degree of whiteness of the flame are due to the high temperature of the solid particles of carbon, which play the same *rôle* as the rare earths in the incandescent mantle. The consumption of acetylene per candle-hour varies between 0·643 and 0·8 litre, whilst that of coal-gas in different burners is as follows:—Butterfly burner, 11·5; Argand, 10; Siemens, 3·7; Auer, 2·7 litres.

Probably one of the best results to be obtained from this gas is the enrichment of existing kinds of lighting gas, but the results already obtained are discordant; and benzene may prove a formidable competitor, in view of its lower cost. At present, acetylene is too expensive for motor work, but when the carbide can be produced at a cheap enough rate it may be found advantageous for special purposes where lightness of fuel is a consideration, such as for marine engines, automotors, &c.—C. S.

Acetylene: Its Dangers and their Prevention. J. für Gasbeleucht. 1897, **40**, 149—153.

A CONFERENCE was held under the auspices of the Berlin Section of the "Berufsgenossenschaft der Chem. Ind.," Dec. 29, 1896, for the discussion of the manner in which it was desirable that the manufacture and use of acetylene should be controlled. Dr. C. A. Martius, who presided, feared that the new industry might be crippled by prohibitive legislation, such as the stringent police regulations in force in Berlin. A careful study of the properties of acetylene, for which the researches of Berthelot and Vieille (Comptes Rend. **123**, 523; this Journal, 1896, 793) might serve as a starting point, would indicate to what extent legislation was required.

Dr. Frank thought the dangers of gaseous acetylene had been much exaggerated. With regard to its toxic properties, he and Dr. Weyl had shown that air containing 9 per cent. of acetylene could be breathed for a long period without serious results. The phosphuretted hydrogen in the gas evolved from calcium carbide promoted its combination with copper and increased its explosiveness. This impurity could be completely removed by washing with acid solutions of metallic salts. The explosive metallic compounds were only formed in ammoniacal solutions. When acetylene was stored in gas holders, the confining liquid should be brine, in which it was practically insoluble. The impurities of low-grade carbide were generally carbon and quicklime, and it did not necessarily give an impure acetylene. No explosive decomposition of acetylene took place when it was passed, under atmospheric pressure, through a tube heated to 600°—1,000°. Large pieces of carbide decomposed slowly in water, as a protecting layer of calcium hydrate formed upon them. Carbide could be safely transported in light air-tight drums, whereas liquefied acetylene required heavy cylinders. Acetylene was inferior to benzene for enriching coal-gas. Dealing in carbide should come under similar restrictions as traffic in explosives and poisons, but the manufacturer should not be unduly hampered.

Prof. O. N. Witt thought that accidents with acetylene had always been associated with its compression. Its critical point and temperature did not render its compression difficult, and the danger must be due to its endothermic character. In this respect it was comparable to modern explosives.

Dr. Th. Elkan considered the dangers were confined to the liquefying of the gas. The so-called "condenser" should be emptied when the work stopped, otherwise a rise in temperature was caused and irresistible pressure within it, owing to the very high coefficient of expansion of liquefied gases. The steel bottles should not be overcharged, and the limit of 1 kilo. of acetylene per 3 litres capacity, which had been laid down by the German railways, might well be observed. Bottles and parts which might come in contact with liquid acetylene should be quite free from copper. He thought compression of acetylene need not be prohibited, provided precautions were enforced.

Dr. Krämer advocated brine instead of water for seals.

Dr. Tiefrunck pointed out that it was necessary to keep the pressure low in generating apparatus. Decomposition of liquid acetylene produced a pressure of 5,561 atmospheres. Special precautions were therefore necessary in its production and storage.

Prof. R. Pictet recommended the introduction of the carbide into a relatively large volume of well-cooled water. Ice sufficed for the cooling in a small plant. Absolute purification before storage was essential. It could be carried out by means of solution of calcium chloride, lead compounds, and sulphuric acid, all cooled to − 16° C., at which temperature these liquids did not combine with acetylene. For compression he cooled the gas to − 20° C., and then pumped it, under a pressure of about 8 atmospheres, into a long tube kept at − 80° C. by the evaporation in a vacuum of SO_2 and CO_2. The liquid so obtained was charged into steel bottles at the same temperature. Bottles thus charged with the pure liquid had been subjected to the most violent shocks, and no explosion had ensued. The explosion at his Paris works, which was attended with fatal results, was caused by mechanical injury to a bottle while in a vice for alteration of the valve. The bottle was a full one, accidentally returned to the works with empties. Acetylene in a holder and in bottles in the room where the explosion occurred had been unaffected by it.

Dr. M. Altschul thought purification absolutely essential, whatever the purpose to which the acetylene was to be applied. The steel bottles should be tested at 250 atmospheres, and only two-thirds filled.—J. A. B.

Thoria in Thorite, Estimation of. E. Hintz and H. Weber. Zeits. anal. Chem. **36**, [1], 27.

See under XXIII., *page* 357.

PATENTS.

Acetylene Gas, Impts. in Means or Apparatus for Use in the Production and Application of. T. Holliday, Huddersfield. Eng. Pat. 5813, March 16, 1896.

THE improved apparatus consists of a generator in which calcium carbide is put, and a displacement gasholder with superimposed cistern. The gas passes normally through a cooling coil in the cistern direct to the services, but in the event of over-production, the water, and thereafter the surplus gas, escape through a pipe near the base of the generator into the upper part of the gasholder, by forcing

water therefrom into the cistern; and the gas, and thereafter water, return by the same course when the pressure in the generator falls.—J. A. B.

Acetylene, Improved Apparatus for Generating, and Utilising the same for Lighting and Heating Purposes. R. Reitmeyer, London. From A. Cerckel, Paris. Eng. Pat. 6719, March 27, 1896.

The apparatus consists of a generator containing carbide on trays, having at its base a pipe leading to a water reservoir, and at its top a gas-outlet pipe to a bell gasometer. The bell, as it rises, closes a tap on this pipe, and thus causes the pressure to increase in the generator, and force the water therefrom to the reservoir. So soon as all the water has thus escaped, a ball which was floating on the water drops into the mouth of the pipe and prevents the escape of gas thereby. The bell, as it falls, re-opens the gas tap, and as the gas escapes from the generator the water returns thereto.—J. A. B.

Acetylene Gas, Impts. in Apparatus for Generating and Storing; especially suitable for Use in Portable Installations. J. C. Bayley, Bournemouth. Eng. Pat. 6739, March 27, 1896.

The object of the invention is to avoid the loss of acetylene due to its absorption by the water in contact with which it is usually stored. It is attained by means of an expansible storage chamber of "concertina" pattern, set within a closed water tank, which communicates with an upper cistern supported by springs, so that this cistern rises or falls, according to the amount of water driven out of, or drawn into, the lower tank by the expansion or contraction of the flexible storage chamber, and thus maintains a constant head of water and uniform pressure on this chamber. It may also be attained by means of a float covering the surface of the water in a storage chamber, and preventing the gas coming in contact therewith. The water taken from the tanks to the generator is replaced from a water main, provided with an automatically operated cock. A double valve operated by a single handle serves to open and shut inlet and outlet water pipes, and gas exit of a generator simultaneously, as required in practice.—J. A. B.

Acetylene Gas, Apparatus for the Automatic Generation and Distribution of. A. Tobler, Garenne-Colombes, France. Eng. Pat. 12,662, June 9, 1896.

This consists of a series of generators connected at top to a collecting pipe leading to a bell gasholder, and of a water reservoir closed by a weighted piston. The generators being charged with carbide, water flows from the reservoir through a non-return ball valve into the lower part of the first of the series, and when the carbide therein is exhausted, the water flows on through another valve to the next generator, and so throughout the series.—J. A. B.

Acetylene Gas or Acetylene and Carbonic Acid Gas combined, A Gas Plant and System to Make and Produce. R. Goodwin, Dublin. Eng. Pat. 17,644, Aug. 10, 1896.

The plant consists of one or more lead-lined generating chambers, to be charged with carbide or a mixture of carbide with some substance which produces carbonic acid when treated with acidulated water; a reservoir for such water, with feed pipes to the generators and a purifying vessel containing in one chamber lead wire filling, and in another chamber copper sulphate. The vessels are provided with gas-tight lids, and the feed and exit pipes with suitable cocks and pressure gauges. It is stated that, admixed with carbonic acid, a more perfect combustion of the acetylene is secured, without the deposit formed under ordinary conditions.—J. A. B.

Acetylene Gas, Impts. in Apparatus for Generating. E. Quelle, Paris. Eng. Pat. 29,500, Dec. 22, 1896.

Several patterns of apparatus are described, consisting either of a fixed or movable bell with vertical partitions, or of two or more concentric or contiguous bells: some of the compartments or bells contain a carbide receptacle, and serve as generators; others serve as gas reservoirs. The generators and reservoirs communicate by a pipe furnished with a tap, which may be actuated by the rise and fall of a gasometer. The carbide receptacle has several vertical compartments, with bars or grids supporting the carbide at different levels, so that the contents of one compartment only, are brought into contact with the water at one time. Several modifications of parts of the apparatus are described. —J. A. B.

Generating [Acetylene] Gas, Impts. in Apparatus for Use in. J. Schumacher, Chicago. Eng. Pat. 30,134, Dec. 31, 1896.

The invention relates to apparatus for producing acetylene gas from calcium carbide and water, or other gas generated by the action of a liquid on a solid, the object being to produce the gas continuously and in practically constant quantity throughout the period of generation and use. The diagram and description given, show the invention embodied in a lamp adapted for use on vehicles. The carbide is contained in an upright cylinder, and is confined therein by two horizontal diaphragms; the upper diaphragm is fixed, and the lower one, which is held in position by means of a spring ring, is capable of moving downwards in the manner of a piston, the sides of the cylinder being continued downwards some distance below the lower surface of the carbide. Above the upper diaphragm is situated a gas-tight water reservoir, from which water flows through an opening (the size of which may be regulated by a valve operated by a milled head outside the lamp) in the diaphragm on to the upper surface of the carbide. The gas liberated, passes down through the unaltered carbide, becoming dried, passes through the lower diaphragm and then to the burner by way of an upright tube which rises from the lowest part of the interior of the apparatus. A branch tube communicates with the upright tube and with the interior of the water reservoir, thus equalising the pressure in the latter and in the carbide chamber. The carbide becomes exhausted at its upper part, and if the quantity of water flowing on to it from the reservoir were kept constant, the amount actually filtering down to the unaltered carbide would become less and less, and the gas would be more and more slowly generated. But the mass swells on being acted upon by the water; this causes the downward depression of the lower diaphragm, which communicates by means of a central rod with the valve through which the water passes from the reservoir to the carbide cylinder. Four forms of the valve are shown, each of which is employed for the same object, namely, to admit a gradually increased amount of water to the carbide as the lower diaphragm is depressed. The rate of generation of the gas is thus kept constant.—H. B.

Acetylene Gas, Process and Apparatus for Producing. [Calcium Carbide Cartridges.] F. Trendel and J. Mücke, Berlin. Eng. Pat. 139, Jan. 2, 1897.

Uniform generation of gas is said to be secured by the employment of a cartridge containing carbide in compartments, with separate water inlets at different levels. The water enters the cartridge by a pipe at top, but passes to the bottom before coming in contact with the carbide, and thus cools the compartments before they come into operation. An increase of gas pressure in the generator depresses the water below the inlet to the cartridge. A siphon with water closure serves to remove air from the generator prior to the development of the gas. The cartridges have their openings sealed to preserve the carbide from the air and moisture until required for use. Special valves and systems of pipes for regulating the water-feed from the reserve cistern, the gas bell, and the generator are described for one form of the apparatus. Another form has a day flame to burn when the burner proper is extinguished. A safety-pipe ends near it, so that in the event of over-production the surplus gas is consumed.—J. A. B.

Acetylene and Similar Gases, Impts. in Processes of and Apparatus for Forming and Burning. J. Zimmerman, Chicago, U.S.A. Eng. Pat. 328, Jan. 5, 1897.

The apparatus consists of generating, gas-containing, and water-holding chambers superimposed in the order named. Above these is a chamber with diaphragm partition; the

diaphragm is attached to a spindle, which actuates the valve admitting water to the generator, and the lower side of the diaphragm is subject to the pressure of the gas in the generator, so that the admission of water is automatically controlled by this pressure. The gas passes from the generating to the gas-containing chamber through a purifier or dryer and cooling-pipe, the outlet of which is closed or opened by a valve automatically actuated by the pressure of the gas in the containing chamber. The burners are supplied from the latter.—I. A. B.

Reducing Metals from their Ores, and for Obtaining Carbides, Acetylene, Cyanogen, Cyanides, Nitrides, and for fixing Nitrogen; Impts. in Processes and Apparatus for. [*Electrical Blast Furnace.*] H. Maxim, New York, U.S.A. Eng. Pat. 1905, Jan. 27, 1896.

See under XI. B., *page* 338.

Calcium Carbide, An Improved Method of and Apparatus for the Production of. [*Preliminary Heating.*] F. H. Haviland, A. Holloway, J. B. Collier, and W. H. Murch, Bournemouth. Eng. Pat. 15,482, July 16, 1896.

In this process the mixture of carbon and lime is heated by means of a furnace before or whilst being fed into the electric arc, which is struck between a plumbago pot and a movable carbon electrode. This pot closes the upper part of the furnace, from which it is insulated, and through one side of it passes an inclined feed, which is provided with a jacket which is open to the furnace flue, so that the hot gases surround the tube and also the receptacle into which the mixture of coke and lime is fed. This tube may be provided with a rotating worm to feed the mixture into the arc, or the worm may be dispensed with and the tube itself be rotated. The pot is provided with a lid, which is insulated from the carbon electrode passing through it, and from it a flue carries off the gases to a chamber where they may be treated, which chamber is also connected by a flue with the feed receptacle. The outlet pipe discharges the molten calcium carbide on to two grids placed over each other transversely, these by their motion producing granules of any desired size.—G. H. R.

Incandescent Gas Lighting, Impts. in. [*Use of Rotary Mixers for Gas and Air.*] L. Denayrouze, Neuilly. Eng. Pat. 7463, April 8, 1896.

Referring to his Eng. Pat. 4494 of 1895 (this Journal, 1897, 130), the patentee considers that the mixture of gas and air should arrive at the point where it burns under these conditions: physically, there should be complete interpenetration of the two fluids; chemically, the proportion should be 4 or 5 parts of air to 1 part of gas; "the mechanical condition is determined by a pressure scarcely sensible (about 2 mm.) and an average speed of issue from the gauze at the top of the burner of about 4 metres per second." The patentee claims—

1. In the production of incandescent gas light by Welsbach and like mantles, employing the gas and air in their ordinary conditions, or, after having modified these conditions, utilising, if necessary, the gas pressure as the power for effecting intimate interpenetration of the molecules of the gas and air.

2. Effecting the intimate interpenetration of the gas and air by rotary mixers, or by passing the fluids through gauzes or successive diffusion chambers, or through crossing passages, or between blades or baffles which cause the fluids to become broken up, subdivided, and intermingled, while the filamental streams of air and gas may be still further broken up or triturated by rotary movements produced by the simplest mechanism, including chimney draught.

3. The application to incandescent gas lighting of the principles above set forth, employing, instead of ordinary gas, combustible vapours of more or less volatile oils, spirits, and other liquids, or acetylene or other combustible gases.—H. B.

Incandescent Gas Lights, Impts. relating to. De R. Hollins, New York. Eng. Pat. 27,269, Dec. 1, 1896.

This invention relates to a composition for incandescent devices made of a mineral substance without a cotton fabric or the like. The incandescent material consists of magnesia (to give incandescence), powdered porcelain and asbestos (to impart durability), lime and sulphate of chromium (to cause diffusion of the light), sulphate of barium or minium (to give the device "a good weight in proportion to its size"), thoria and zirconia (to help to diffuse the light and give an agreeable white or yellowish light). These materials are mixed together as a dry powder, then rubbed into an organic binder, such as sugar solution, and shaped into incandescent devices, which are dried and baked in suitable ovens in the usual manner. The composition is claimed. —H. B.

Incandescent Lights, Impts. in and relating to. A. Rammoser and A. von Hake, Berlin. Eng. Pat. 29,613, Dec. 23, 1896.

To produce thread material for incandescent lights adapted to resist shock, falls, &c., a thin wire, composed of one or more of the platinum metals is overspun with cotton or other suitable fibre, and the material so produced is plaited or woven with a corresponding number of cotton or other threads and made up into suitable shapes. The body is then impregnated with any of the known lighting fluids; or the thread may be impregnated before spinning it on the wire.—H. B.

Carburetted Water-Gas, Impts. in Apparatus for the Production of. W. Wheatley, Liverpool, and G. Kros, Leigh. Eng. Pat. 4980, March 5, 1896.

This invention relates to improvements on Fourness' Eng. Pat. 15,169 of 1891 (this Journal, 1892, 149). Two generators are used alternately, the one being under blast for raising the fuel to incandescence and meanwhile heating up the carburetting and fixing retorts, while the incandescent fuel in the other generator is decomposing steam and producing water-gas, which is enriched with oil in the hot carburetting and fixing retorts. The improvements comprise (1) the construction of a generator having an oblong well forming the ash pit, and containing the fire grate running across the base, and having the interior sides of the wall converging towards the grate, and provided with air and steam blast-pipes; (2) the construction of a fixer for the mixed water-gas and oil vapours, consisting of a closed vertical casing fitted with an internal central conduit which communicates with the casing at one end, having chequer-work between the conduit and the casing wall, with inlet and outlet so arranged that the gases, on entering, traverse the length of the conduit inside and then throughout the length of the casing and chequer-work outside the conduit.—H. B.

Combustible Gas, Impts. [*Revolving Hearth*] *in Furnaces or Apparatus suitable for the Manufacture of.* A. Kitson, Philadelphia. Eng. Pat. 5291, March 9, 1896.

This patent describes improvements on the patentee's Eng. Pat. 19,521 of 1893 (this Journal, 1894, 243). The rotating hearth is made ring-shaped, and the opening in the centre of the ring is occupied by a stationary plate, which is provided with a number of radially-projecting wings. The ring-shaped portion of the hearth is inclined to the horizon, and its surface is diversified by elevations and depressions. The walls of the combustion chamber extend downwards below the hearth, encircling and closely fitting it, the opposing surfaces thus formed being sufficiently near to grind the ashes sifting between them. The grinding action is accelerated by cutting helical grooves in the opposing surfaces. The rotating hearth is supported on a hollow shaft resting and rotating about a vertical axis upon ball bearings, and within this shaft is a pipe, depending from the central stationary plate, up which, by suitable means, a blast of air is driven into the fuel. The hearth is rotated by means of a worm wheel attached to the hollow vertical shaft and engaging a worm. These improvements cause the body of fuel to grind continuously upon itself to a much greater degree than was attainable under the above-mentioned patent.—H. B.

Coke-Ovens, Impts. in. G. Hilgenstock, Dahlhausen, Germany. Eng. Pat. 6857, March 28, 1896.

The supply of gas for heating the ovens is admitted to the flues from conduits situated below the furnace and contained

in passages in the basement large enough to admit the attendants for the purpose of regulating the supply to any part, or for cleansing, &c. It is claimed that by this means uniformity of coking is attained, local overheating, and the resultant damage to the brickwork, and decomposition of the coke gases being prevented.—C. S.

Coke Furnaces, Impts in. F. J. Collin, Dortmund, Germany. Eng. Pat. 25,887, Nov. 17, 1896.

To ensure direct firing, without local overheating, in the middle of the furnace, longitudinal and transverse flues are arranged in the walls, and gas is supplied to the centre of the furnace through small pipes between these flues, in which the combustion is effected, the necessary air being delivered through separate pipes opening near the gas-pipe jets. By stopping the gas supply and admitting air, the soot deposited in the gas pipes can be quickly removed. —C. S.

Petroleum Regenerative Lamps, Impts. in. [Preliminary Gasification of Oil by an Under Burner.] F. Ludwigs, Berlin. Eng. Pat. 5843, March 16, 1896.

In this (inverted) lamp, the preliminary gasification of the oil is effected by an under burner with asbestos wick, fed by petroleum from the oil reservoir and heating a metal plate supporting the annular gasifier, the supply being then diverted to the latter by adjusting a two-way tap in the supply pipe, whereupon the oil is vaporised and the gas descends through an annular cylinder to the orifices of the main burner, where it is consumed, the heat thenceforward sufficing to renew the supply of gas. By a suitable method of admitting air, the flame is caused to diverge outwards in the form of an arch.—C.S.

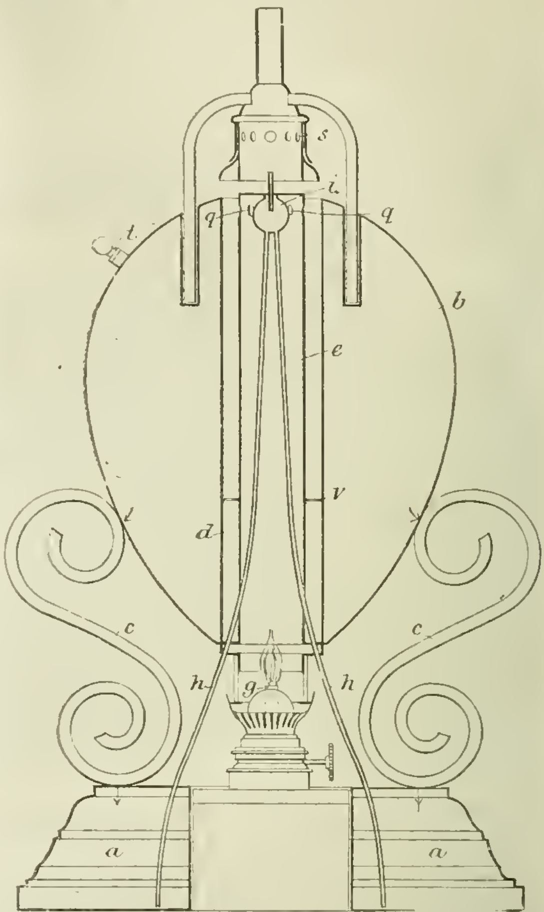
Petroleum Residues for Heating Purposes [Gasification by Aid of Hot Air], Impts. in Means for Burning. F. Grube, Hamburg. Eng. Pat. 16,574, July 27, 1896.

To gasify petroleum residuum for fuel on ship-board, where atomising is inconvenient, the inventor proposes to induce a current of heated air through the oil reservoir by means of an air pump, which also compresses the resulting gases and delivers them, by way of a reducing valve, to a burner inserted in the fire-box of the boiler. The air necessary for combustion is supplied inside the flame, through a bell-mouthed central pipe projecting beyond the nozzle of the burner and adjustable for the purpose of altering the size of the flame as desired; suitably arranged clack-valves admitting air to the outside of the flame. The flame impinges against a fire-bridge, the desired position being attained by fitting the burner at a suitable height and making it horizontally adjustable.—C. S.

Gas from Liquid Combustible Materials ["Petroleum, Spirit, or the like"], Method and Apparatus for Producing. G. Trescnreuter, Berlin. Eng. Pat. 26,516, Nov. 23, 1896.

This patent describes an apparatus suitable for producing from petroleum, &c., a flame adapted for illuminating purposes, particularly incandescence lighting. In the diagram, *a* contains the combustible liquid, and *b* contains atmospheric air, the two vessels being connected by the pipes c. Vessel *b* is air-tight, and is traversed centrally by the passage *d*, within which is a chimney, *e*, surmounting the heating lamp, *g*, which is an ordinary petroleum lamp. From *a* rise the pipes *h*, which pass over the heating flame into the chimney, and open into the vaporising chamber *i*, from which the gas passes to a burner provided with the usual air inlets, *s*, and resting, in, say, the manner shown, upon the vessel *b*. The apertures, *q*, in the chimney permit the escape of the gases of combustion of the flame *g*. Between the chimney and the wall of the vertical passage, *d*, there is inserted a metal ring *v*, which conducts the heat from the chimney to the air-vessel. In the tubes *h* there is an insertion of absorbent material, such as asbestos, for conducting the liquid upwards in a cool state, so that vaporisation does not begin until the liquid reaches the chimney, *e*.

On lighting the heating flame, the air in *b* expands and raises the combustible liquid in the tubes *h* to the vaporising chamber. The advantage claimed for the



apparatus is that only cool air comes into contact with the petroleum, &c., which can thus never become hot. At *t* is an outlet for reducing the pressure within *b* on extinguishing the lamp.—H. B.

Mineral Oils, Impts. relating to the Burning of. M. L. Ross, London. Eng. Pat. 26,962, Nov. 27, 1896.

The lamp forming the subject of this invention is intended for vaporising the oil to be burned. To this end, heat is applied to a convoluted metal tube above the lamp, and, by conduction *viâ* a central tube dipping into the receiver, expands the air in the latter and causes the oil to rise through the said tube (packed with asbestos) into the coil, where it is vaporised, and, issuing through one or more pinholes in the coil, burns, air being admitted into the coil by other pinholes situated below. For heating purposes, a metallic plate pierced by a central aperture is supported over the flame by lugs, or replaced by an incandescent mantle for lighting purposes.—C. S.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Mineral Oils of High Boiling Point, Effect of Expansion on Flashing-Point Determinations. D. Holde. Chem. Rev. Fett- u. Harz-Ind. 4, [4], 51.

The author had already shown (this Journal, 1895, 284) that even in the case of petroleum oils boiling as high as 280° C., the expansion of the oil is without any appreciable influence on flashing-point determinations in the Abel and Pensky

test cups, and he now records the results of a further series of experiments, showing that with oils flashing between 172° and 241° C., a variation of even as much as 13 c.c. below the normal filling of the instrument does not alter the flashing point by more than 1° to 1·5° C., concluding therefrom that the influence of expansion may be neglected. —C. S.

(Sulphocyanides) Thiocyanates in Gas Liquors, Conversion of, into Alkali Ferrocyanides. A Dubose. J. de l'Eclair. au Gaz, **45**, [1], 10.

See under VII., *page* 328.

Petroleum Oils, &c., Testing; Recent Experience in. Holde. Mitt. k. t. Versuchsanst. zu Berlin, **14**, [4], 229.

See under XXIII., *page* 363.

Paraffin in High Boiling Distillates from Petroleum, Determination of. Holde. Mitt. k. t. Versuchsanst. zu Berlin, **14**, [4], 211.

See under XXIII., *page* 362.

Mineral Oils, Dissociation of Petroleum Acid Salts and the Estimation of Free Acids in. R. Zaloziecki. Chem. Rev. Fett- u. Harz-Ind. 4, [2] and [3], 25—27 and 36—38.

See under XXIII., *page* 362.

American Petroleum, Some Hydrocarbons from. I. Normal and Iso-Pentane. S. Young and G. L. Thomas. Proc. Chem. Soc. 1897, [176], 58—60.

The two pentanes were separated by fractional distillation from the "pentane" supplied by Merck, of Darmstadt. This substance, which is obtained by the distillation of American petroleum, is a complex mixture of butanes, pentanes, and hexanes, with some benzene and a little hexanaphthene. A combination of a dephlegmator with a constant (or rather "regulated") temperature still-head was employed; the apparatus is fully described in the paper.

Some of the constants of isopentane were determined so as to compare them with those of the two specimens prepared synthetically; the agreement was found to be very satisfactory. The boiling points under normal pressure are: isopentane, 27·95°; normal pentane, 36·3°. The specific gravities at 0° are: isopentane, 0·63930; normal pentane, 0·64539.

The Vapour Pressures, Specific Volumes, and Critical Constants of Normal Pentane; with a Note on the Critical Point. S. Young. Proc. Chem. Soc. 1897, [176], 58—60.

The critical temperature of normal pentane is 197°·2 C., the critical pressure is 25,100 mm., and the critical volume of a grm. 4·302 c.c. The vapour pressures and specific volumes were determined from low temperatures to the critical point, and the observations were taken as near to the critical point as possible (197·15), in order to obtain more complete experimental evidence regarding the condition of a substance at and very near that point.

The ratios of the absolute temperatures (boiling points) and volumes to the critical constants, also the ratio of the actual to the theoretical density at the critical point (3·765), lead to the conclusion that, at the critical temperature and in the liquid state, the molecules are simple, like those of the gas.

PATENTS.

Ammonia, An Improved Method or Process of Treating Substances containing Nitrogen for the Manufacture of. H. H. Lake, London. From C. Pieper, Berlin, Germany. Eng. Pat. 28,199, Dec. 9, 1896.

See under VII., *page* 329.

Artificial Asphalt, Impts. in the Process of Manufacture of. A. Hannemann and G. Hoisly, both of Berlin. Eng. Pat. 3597, Feb. 17, 1896.

The inventors claim "the manufacture of artificial asphalt produced by treating pitch, or the remains of resin or petroleum distillation, first with sulphur and then with chloride of lime, or first with chloride of lime and then with sulphur, the product being ground and mixed with amorphous lime, slag or other stone- or saw-dust, and again heated to boiling point." Such a material is stated to be "superior to natural asphalt."—V. C.

IV.—COLOURING MATTERS AND DYES.

Colouring Matters [Vegetable] from Annam. M. O. Picquet. Rev. de Chim. Ind. 8, [86], 45—48.

The author describes two natural dyestuffs which he has received from Annam. The first of these is derived from the *cû-nao* or *cû-nor*, a potato-like tuber, weighing from 500—1,000 grms. It is much used in the plains of Tonkin and the provinces of Vinh and Hatinh. The flesh of the tuber is somewhat woody, and resembles a dried beetroot. The *cû-nao* is only used in its fresh state. The tubers are preserved by burying them in moist earth, so that they shall not lose their natural moisture, since by drying they lose much of their tinctorial powers. For dyeing purposes, the natives cut the *cû-nao* into thin slices, which are put into a mortar with six times their weight of water. The liquor is decanted, the cloth steeped in it for 5—6 hours, and it is then spread out on the grass, face upwards, to dry. The side exposed to the light and air is darker than the one turned towards the ground.

To increase the lustre, the cloth is subsequently steamed in a crude fashion, by placing the material on a wooden grid above a vessel of boiling water. For darker shades, these operations are repeated. Sometimes a glutinous substance, *phen-den*, imported from China, is added to the dye-bath.

An aqueous extract of *cû-nao* gives the following reactions:—

Gelatin.—Little action.

Alum and Aluminium Acetate.—Gelatinous light brown precipitate.

Iron Salts.—Greenish-black precipitate.

Iron Sulphate.—Brown precipitate.

Potassium Bichromate.—Dark yellowish-brown precipitate, slight in the cold, but abundant on heating.

Lead Acetate.—Voluminous light brown precipitate.

Tin Salts.—Drab precipitate.

Dilute Acids.—Dirty yellow precipitate.

Alkalis.—Colour much darker.

Mercury Bichloride.—Voluminous dirty white precipitate.

Potassium Ferrocyanide.—Little action.

Potassium Ferricyanide.—Yellowish-brown precipitate; liquor the same colour.

Bleaching Powder.—Dark brown precipitate, which quickly turns yellow.

The best method of application is to first dye the cloth, with or without the addition of alum, and then fix the colour with a bichromate. Prints should be steamed.

The other product, called *cay-da* or *cay-iu*, is the bark of *Brugniera gymnorrhiza*,* and resembles in appearance the cinnamon of commerce. The colour obtained from it is a reddish-brown of great density, which has a certain analogy with catechu, but the browns are much fuller and more brilliant than can be obtained from the latter. This product, the author says, is more interesting than the former, and might find a useful place among our colouring matters.

The following are some of its principal reactions:—

Gelatin.—Voluminous brown precipitate.

Alum.—Light brown precipitate.

Aluminium Acetate.—As with alum, but a more abundant precipitate.

Ferrous Acetate.—Dark greenish-grey precipitate.

Ferrous Sulphate.—Purplish-black precipitate.

Ferric Sulphate.—Deep black precipitate.

* This is one of the so-called Mangrove barks, and is already employed in the form of extract by European dyers.

Copper Sulphate.—Blackish-brown precipitate.

Potassium Bichromate.—Dark reddish-brown precipitate, slowly thrown down in the cold, but quickly when hot.

Bleaching Powder.—Dark brown precipitate, turning yellow on heating.—I. S.

Stable Diazo Compounds. C. Gassmann. Färber Zeit. **8**, 67—68.

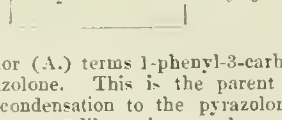
The Farbwerke vormals Meister, Lucius, and Brüning, in a recent patent (Eng. Pat. 1645 of 1896; this Journal, 1897, 40), claim the manufacture of stable zinc chloride double salts of certain diazo bodies. The author states that these double salts have been described by Feer in 1891 (Bull. Soc. Ind. Mulhouse, 1891, 221: this Journal, 1891, 695), and that similar salts were brought into the market in 1894 by Messrs. Wacker and Schmitt, of Mulhouse, but have now been withdrawn, being found insufficiently stable for long preservation. In the author's opinion, it is better to add the metallic salt, zinc chloride or stannic chloride, to the previously prepared printing colour, and for this purpose other substances besides these two salts give increased stability to the diazo compound, *viz.*, sodium picrate, and the sodium salts of dinitro-α- and β-naphthol.—R. B. B.

Tartrazine, Constitution of. R. Anschütz. Annalen, **294**, 219—243.

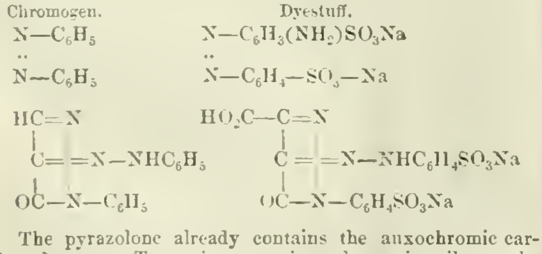
The tartrazines are dyestuffs obtained by the action of phenyl-, tolyl-, xylyl-, or naphthylhydrazine sulphonic acid on dihydroxytartaric acid, and have hitherto been supposed to have an analogous formula to the osazone (diphenylizinedihydroxytartaric acid) produced from dihydroxytartaric acid and phenylhydrazine, *viz.* :—

$$\begin{array}{l} HO_2C{-}C{=}N_2HC_6H_4[4]SO_3Na \\ \qquad\quad | \\ HO_2C{-}C{=}N_2HC_6H_4[4]SO_3Na \end{array}$$

Knorr (Ber. **21**, 1204), however, showed that diphenylizinedihydroxytartaric acid, even on crystallisation from glacial acetic acid, is very easily converted into an inner anhydride, forming a pyrazolone ring—



which the author (A.) terms 1-phenyl-3-carboxy-4-phenylhydrazonopyrazolone. This is the parent substance of tartrazine, the condensation to the pyrazolone ring taking place much more readily owing to the presence of the sulphonic acid groups. The alternative method for the preparation of tartrazine (Eng. Pat. 5693 of 1893; this Journal, 1894, 382) has also been investigated, and the following conclusions are arrived at. The dyestuffs obtained from aromatic hydrazine sulphonic acids and dihydroxytartaric acid are *hydrazonopyrazolones*, and not hydrazones or osazones. The chromogen of tartrazine is phenyl-phenylhydrazonopyrazolone, which bears the same relation to tartrazine that azobenzene does to amido-azobenzene sodium disulphonate.



The pyrazolone already contains the auxochromic carboxyl group. Tartrazine owes its colour primarily to the fact that it is derived from the osazone of an *o*-diketone. This colour is considerably increased by the formation of the pyrazolone ring.—T. A. L.

Magenta and Malachite Green, Reduction of Colouring Matters of the Type of. M. Prud'homme. Bull. Soc. Chim. 1897, **17**, [6], 376—378.

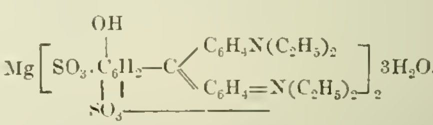
Hot solutions of colouring matters of the type of Magenta and Malachite Green, treated with zinc dust in the presence of an acid, are completely reduced, and do not regain their colour on exposure to air. If the reduction be effected rapidly and in the cold, after filtering, the solution at once begins to regain its colour; boiling for some minutes, or long standing in the air, gives a solution of which the colour intensity is about half that of the original solution. The nascent hydrogen has reduced a portion of the colouring matter to the leuco base, but in another portion two hydrogen atoms have probably united to each of the NH_2 groups, giving colourless compounds analogous to those produced by the action of mineral acids. In the case of Parafuchsine, this substance would have the formula $ClC\ (C_6H_4.NH_4)_3$. Boiling at once decomposes this compound, but by standing in the air the hydrogen of the NH_4 groups can reduce another portion of the colouring matter to the leuco base, thus explaining the observation that boiling effects a greater return of colour than long standing. An increase in colour on boiling with lead dioxide (the solution becoming partially restored to its original colour on standing) demonstrates the presence of the leuco base.

The basic colours not derived from triphenylmethane—such as the Safranines, Methylene Blue, Rhodamines,—after reduction by zinc dust in the cold, regain their original colour.—A. C. W.

Patent Blue, Constitution of. E. and H. Erdmann. Ann. 1897, **294**, 376—392.

The authors some time ago discovered a method for manufacturing Patent Blue (Ger. Pat. 64,736), which proves the position in the molecule of the latter of one of the sulphonic acid groups.

The Patent Blue of commerce is the magnesium salt, which, when purified by crystallisation from water and dried at 110°, has a composition corresponding with the formula—



It is not affected by boiling with barium hydrate solution, being deposited unchanged on precipitating the barium with carbon dioxide, filtering and evaporating.

When heated with sodium hydroxide solution, it is readily decomposed into a colouring matter of the rosolic acid group, diethylamine being evolved, and the colour of the solution changing first to violet and then to red. (This Journal, 1897, 39.)

m-Hydroxy-tetra-ethyl-p-diamidotriphenylcarbinol, $HO.C : [C_6H_4OH]\ [C_6H_4N(C_2H_5)_2]_2$, is produced when Patent Blue is heated with concentrated hydrochloric acid under an inverted condenser, being precipitated on adding an alkali to the solution. It forms, when dry, a dark green powder, which dissolves in alcohol and in very dilute hydrochloric acid with a green colour, changing to yellow on the addition of concentrated hydrochloric acid. It is not decomposed by heating with caustic soda solution.

m-Hydroxy-Brilliant Green p-sulphonic acid and its leuco compound, prepared from *m*-hydroxybenzaldehyde-*p*-sulphonic acid, are acids of so feeble a character that they may be extracted from aqueous alkaline solutions by means of chloroform. The authors were led by this circumstance previously to regard the compounds in question (and Patent Blue) as sultonic anhydrides formed by elimination of the elements of water from the *m*-hydroxyl and *p*-sulphonic acid groups. They now find that this view is incorrect, as the analytical results do not accord with it, and the acids do not become dehydrated either when heated alone or with condensing agents.

Synthesis of Patent Blue from m-nitrobenzaldehyde.—*m*-Nitrobenzaldehyde condenses with diethylaniline, giving *m*-nitro-leuco-Brilliant Green, a compound crystallising from

alcohol in yellow needles (m. pt. 95°—96°) with a yellow-green fluorescence. When reduced, it gives an amido compound, which is converted into Patent Blue by diazotising, boiling, treating the product with fuming sulphuric acid at the ordinary temperature, and finally oxidising and neutralising.

Synthesis of Patent Blue from p-chlorbenzaldehyde.—This chloro-compound gives, on nitration, *p*-chlor-*m*-nitrobenzaldehyde (m. pt. 62°), which, when boiled with sodium sulphite, forms sodium-*m*-nitrobenzaldehyde-*p*-sulphonate. From the latter, the corresponding *m*-hydroxy compound is prepared by reducing and diazotising, and this, when condensed with diethylaniline, yields *m*-hydroxy-leuco-Brilliant Green *p*-sulphonic acid, from which Patent Blue is easily obtained by sulphonating and oxidising.—E. B.

Magenta and Magenta S, Distinction between, by Schiff's Reaction. P. Cazeneuve. Bull. Soc. Chim. 1897, **17** 196—199.

See under XXIII., *page* 361.

PATENTS.

Colouring Matters [Cotton Violets, Blues], Impts. in the Manufacture of. H. H. Lake, London. From The Farbwerk Mühlheim, Mühlheim-on-the-Maine, Germany. Eng. Pat. 5857, March 16, 1896.

This is an extension of Eng. Pat. 10,669 of 1895 (this Journal, 1896, 445), which referred to the production of red to violet disazo dyestuffs from ethyl amidonaphthol sulphonic acid, 1′.3.3′.$C_{10}H_5$.OH.NHC_2H_5.SO_3H, by combining it with diazo compounds of the general formula R.N : N.R_1.N : N—, where R is the radicle of an amine generally incapable of giving direct azo cotton dyes, and R_1 is an amine radicle which, when combined with diazo compounds, can be further diazotised. It has now been discovered that the ethyl amidonaphthol sulphonic acid can be replaced by one of the three following acids : 2.4′.2′-amidonaphthol sulphonic acid, 2.4′.2′.1-amidonaphthol disulphonic acid, or 2.4′.2′-phenylamidonaphthol sulphonic acid. The last-mentioned is prepared by heating 2.4′.2′-naphthylamine disulphonic acid (Eng. Pat. 2614 of 1893; this Journal, 1894, 144) with 1 part of aniline and 0·6 part by weight of aniline salt for six hours to the boil, making alkaline with sodium carbonate, removing the aniline by means of steam, evaporating the solution to dryness, and fusing the residue with 1·5 part of caustic potash lye of 50 per cent. for 1½ hours to 210°—220° C. On acidifying, phenylamidonaphthol sulphonic acid separates from the solution. The following example illustrates the method employed. The amido-azo compound from diazotised *p*-toluidine-*m*-sulphonic acid (from *p*-nitrotoluene sulphonic acid) and α-naphthylamine, is dissolved in sodium carbonate solution, mixed with the molecular proportion of sodium nitrite, and poured into cold dilute hydrochloric acid. When the diazotisation is complete, the diazo-azo compound is stirred into a solution containing a molecular quantity of amidonaphthol sulphonic acid kept alkaline with sodium carbonate. The violet dyestuff produced is salted out hot.—T. A. L.

Anthracene Compounds, and of Dyestuffs derived therefrom; The Manufacture or Production of. H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 7538, April 9, 1896.

Liebermann (Annalen, **212**, 14), by acting on quinizarin with hydriodic acid and phosphorus, obtained a product which he termed quinizarin hydride. This substance, according to the present specification, can be much more readily obtained from purpurin by oxidation in an acid solution. Leuco derivatives of quinizarin are formed, which, on oxidation, are converted into quinizarin, and this process can be applied to other hydroxyanthraquinones. Purpurin, when treated with reducing agents, yields two different leuco-quinizarins, designated I. and II. The latter is identical with Liebermann's hydride, and differs from leuco-quinizarin I., which is very easily oxidised, its solution in sulphuric acid being converted even at the ordinary temperature, and when heated to 70°—90° C. it yields quinizarin very readily, whilst II. requires a temperature of about 120°—130° C. The reduction is effected by suspending 50 kilos. of finely-powdered purpurin in 400 kilos. of glacial acetic acid, heating to 70°—80° C., and gradually stirring in 25 kilos. of zinc dust. When the whole of the purpurin is dissolved, the solution is filtered, and the leuco compound is precipitated by adding water. By dissolving purpurin in 20 times its weight of sulphuric acid, and stirring in twice its weight of zinc dust at −10° to −5° C., and pouring the melt, when reduced, into water, yellow flakes, consisting of a mixture of leuco-quinizarins I. and II., separate out. In a similar manner, other hydroxyanthraquinones, containing hydroxy groups in the 1.2.4 or purpurin position, can be converted into derivatives containing a smaller number of hydroxyl groups. Thus, for instance, alizarin hexacyanin yields leuco-alizarin pentacyanin, which is readily oxidised. Similarly, leuco derivatives of quinizarin sulphonic acid can be obtained by reducing purpurin sulphonic acid. In the case of Alizarin Bordeaux, however, no elimination of a hydroxyl group takes place, and it is only the ketonic groups of the anthraquinone nucleus which are reduced. The specification further describes the preparation of dyestuffs by condensing the leucohydroxyanthraquinones with primary aromatic amines in presence of a condensing agent, such as boric acid, glacial acetic acid, hydrochloric acid, benzoic acid, or the like. The condensation products thus obtained are readily converted into dyestuffs by oxidising agents. Thus, a green dyestuff, identical with that obtained by fusing quinizarin with *p*-toluidine in presence of boric acid (Eng. Pat. 23,927 of 1894; this Journal, 1895, 1041), is obtained by heating leucoquinizarin II. with *p*-toluidine in presence of boric acid and allowing the mixture to oxidise, or preferably by employing a mixture of quinizarin, leucoquinizarin II., *p*-toluidine, and boric acid. The leuco dyestuffs obtained can also be sulphonated by means of fuming sulphuric acid (10—20 per cent. SO_3), and may be used as such for dyeing animal fibres, the colour being subsequently developed by treatment with oxidising agents, such as potassium chromate or the like. They may also, of course, be converted directly into sulphonic acids of the dyestuffs themselves. There are 14 claims.—T. A. L.

Polyazo Colouring Matters [Cotton Browns to Blacks], The Manufacture or Production of. W. E. Heys, Manchester. From The Chemische Fabrik vormals Sandoz, Basel, Switzerland. Eng. Pat. 15,294, July 10, 1896.

The azo dyestuff produced in an acid solution from diazotised 2.1′.3.3′-amidonaphtholdisulphonic acid and α-naphthylamine is combined in equimolecular proportions with a tetrazotised *p*-diamine. The intermediate product so obtained may be combined directly with one molecular proportion of a phenol, amine, or their sulphonic or carboxylic acids, giving valuable trisazo colouring matters which dye unmordanted cotton brown to deep blue-black. In place of combining the intermediate product directly, it may be previously diazotised, and the tetrazo compound thus produced combined with two molecular proportions, either the same or different, of a *m*-diamine, a naphthol, dihydroxynaphthalene, or amidonaphthol sulphonic or carboxylic acid. The octazo dyestuffs thus produced are generally bluer or greener in shade than the corresponding trisazo dyestuffs, and dye unmordanted cotton violet, greenish-blue, and blue-black. The following example illustrates one of the methods employed :—34·1 kilos. of the acid sodium salt of 2.1′.3.3′-amidonaphthol disulphonic acid are diazotised, with 6·9 kilos. of sodium nitrite, suspended in 500 litres of water, and combined with 14·3 kilos. of α-naphthylamine dissolved in 12 kilos. of hydrochloric acid (21° B.) and 300 litres of water. The azo dyestuff separates almost completely, and, after standing 24 hours is dissolved by adding 8 kilos. of caustic soda. 24 kilos. of sodium carbonate are then added, and a tetrazodiphenyl solution is run in, prepared from 18·4 kilos. of benzidine. The intermediate product formed, separates as a blackish-violet precipitate, to which is added a solution containing 23·9 kilos. of γ-amidonaphthol sulphonic acid

and 4 kilos. of caustic soda in 300 litres of water. After about 24 hours the combination is complete, and the dyestuff is separated by salting out the boiling solution. The dyestuff gives deep violet-black shades on unmordanted cotton from a bath containing salt and soda.—T. A. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

Silk, the Weighting of, with Metallic Salts: The Theory and Practice of. H. Silbermann. Färber Zeit. **8**, 34—36, 51—53, and 68—71.

(See also pages 297—303.)

The principal substances used for weighting silk are compounds of the heavy metals: bismuth, tin, tungsten, barium, antimony, and lead.

Weighting with Tin Compounds.—Silk possesses a great affinity for stannic salts, but very little for stannous salts. The most useful stannic compounds are stannic chloride and "pink-salt" ($SnCl_4.2NH_4Cl$).

The silk is weighted in the raw condition, *i.e.*, not boiled-off, since sericin acts as a weak acid and exhibits considerable affinity for metallic bases. The silk is steeped for 2—3 hours in the stannic chloride or pink salt solution at 35° B., wrung out, and washed several times with water. During washing, dissociation takes place, and there is deposited upon the fibre a basic stannic chloride together with small amounts of stannic hydrate and metastannic acid. The bath naturally becomes more acid, and for further use, a small quantity of ammonia is added in addition to stannic chloride. When "pink-salt" is used, the strength of the bath in its useful constituent cannot be estimated by the hydrometer, since ammonium chloride is not taken up by the silk in the same proportion as stannic chloride. To bring about further dissociation, the silk is worked in a cold solution of sodium carbonate at 3°—4° B., then soaped in a lukewarm bath with 50 per cent. of soap, or the soap and soda may be used together in one bath. The effect of this treatment is to leave the silk charged with stannic oxide. The operations are repeated as often as desired: a three-fold repetition gives a weighting of 25—30 per cent.; six-fold, 50 per cent.; eight fold, 90—100 per cent.; and ten-fold, 160 per cent.

The silk weighted with stannic chloride before the treatment with soda and soap is extremely sensitive to the action of light, this action causing a tendering of the fibre. The reason of this is not known, but the author considers it probably due to a dehydrating or reducing action whereby the fibroïn molecule is broken down—a decomposition always accompanied by the liberation of hydrochloric acid, which can be detected upon the tendered fibre. In consequence of this action the weighting is conducted as far as possible in a dim light.

Stannic chloride solution is extremely corrosive, and the workmen should be provided with india-rubber gloves.

After weighting with tin a further weighting may be produced by passing through a tannin bath. By three repetitions of tin and tannin (Chinese galls) baths, souple silk, it is said, may be weighted to 200 per cent. Weighting may also be increased by passing tin-weighted silk into ammoniacal solutions of barium, copper, &c., or solutions of organic lead salts, in order to produce heavy insoluble stannates.

A recent improvement is the substitution of sodium phosphate for sodium carbonate. This stannic phosphate weighting leaves the silk perfectly white, and, it is said, does not injuriously affect the lustre or handle of the fibre. With 4—5 repetitions, a weighting of 120 per cent. results, the fibre is very slightly attacked, and the method is equally applicable to raw or boiled-off silk. After the stannic chloride bath, the silk is entered into a sodium phosphate bath at 55°—60° C., worked for 30 minutes, gradually raised to the boiling point, and boiled for 30 minutes. After thorough washing, the silk is again passed into the tin solution, and the process repeated; but in the last fixing bath an addition of H_2SO_4 is made, sufficient to liberate one-third of the phosphoric acid from the sodium phosphate. A soluble stannic phosphate, made by dissolving granulated tin or freshly precipitated stannic oxide in phosphoric and nitric acids, may be used for weighting up to 80 per cent.

Neuhaus proposes to treat the silk already weighted with stannic phosphate in a bath containing a soluble silicate. By thus weighting with stannic phosphate and silicate, after five passages the weight is 100—120 per cent. over "pari."

Since the price of tin is somewhat high, the wash-waters should in all cases be treated for the recovery of tin by precipitation with chalk, lime, or soda, and this treatment may also be applied to the tin-catechu baths from black silk dyeing.

Weighting with Bismuth Compounds.—These form, it is said, excellent weighting agents, owing to their high molecular weight and the readiness with which they are dissociated. Bismuth nitrate is the most suitable, and is followed by a fixing bath of dilute sulphuric acid; three passages weight to 100 per cent. The price at present precludes the use of this substance.

Tungsten Compounds.—Silk can be weighted to 25 per cent. by twice working in a bath of sodium tungstate at 30° B. and rinsing in an acid bath, but the weighting is removed by washing with hot water or soap, since silk has little attraction for this compound. Sodium tungstate is used, however, for rendering light silk fabrics non-inflammable.

Lead Compounds.—Weighting with lead by passing through basic lead acetate solution has been practised, but is strongly to be condemned owing to the soluble and poisonous nature of the weighting. Plumbate of soda has also been proposed, and since, on washing, this deposits insoluble lead hydroxide, it is said to be unobjectionable from the hygienic standpoint.

Antimony Compounds.—These find little use, in spite of the fact that antimony trichloride can be applied like stannic chloride. It is suggested that use might also be made of the insoluble tannate of antimony.

Barium Compounds.—By successive treatment in faintly alkaline solutions of sodium sulphate and barium chloride, a finely divided precipitate of barium sulphate is deposited within the pores of the silk, and gives to the fibre a brilliant white appearance. The weighting is not removed by washing, but comes out as a fine dust on beating the silk.

Silk weighted with vegetable substances should always be treated with an antiseptic solution, such as boric or salicylic acid, otherwise a development of organisms, which will tender or destroy the fabric, will sooner or later take place.

It was formerly the invariable practice to weight silk in the form of yarn, but recently the weighting of piece-goods has been introduced.—R. B. B.

Russian Wools, Adulteration of. U.S. Cons. Reps., March 1897, 353.

The United States Consul at Odessa reports that the Donskoi, Crimean, and other varieties of Russian coarse wools of the clip of 1896, are shorter in the length of the fibre and inferior in quality. He is reliably informed that numerous shipments of wools from Rostoff and Odessa to the United States have, during the present season, been mixed with "sour wools."

In Russia, 90 per cent. of the population wear sheepskin coats, with the woolly side next to the body. In the process of preparing skins for coats, large quantities of chemicals are employed. As the freshly prepared skins on the leather side are perfectly white, the presumption is that arsenic is largely used. When ready to be made up, much of the wool is trimmed, and the skin cut to suit the measure desired. This results in a certain amount of waste material, both in skin pieces and free-wool fibre. The exact quantity of this waste material annually produced must be very large, and the consul calls it "sour wool."

This material is sold largely to a certain class of merchants, who mix it with their Donskoi and Crimean washed wools. When greasy wools are worth at Odessa from 4·50 to 5 roubles per pood (36·112 lb.), these sour wools will cost from 5 to 6 roubles per pood. Should greasy wools be higher or lower in price, the sour wools follow suit. Whenever these sour wools are mixed with other wools sent to the United States, it will be found that the shipment will contain from 10 to 12 per cent. This proportion

has been maintained since wool has been on the free list. During the time there was a duty on wool, the admixture of these sour wools was from 20 to 25 per cent.

The advantage to the shipper in purchasing these sour wools and mixing them with the washed Donskoi and other wools is in this: Donskoi wools in the grease, yield, when washed, from 50 to 52 per cent. of pure wool fibre, while these chemically cleaned sour wools yield 80 per cent. The Donskoi wools when washed are usually worth from 10 to 12 roubles per pood, while the sour wools never cost more than from 5 to 6 roubles per pood. The value of these sour wools to the manufacturer can be very little; the consul thinks not more than 1 rouble per pood (50 cents per 36·112 lb.). It is stated that the chemical treatment to which this wool is subjected, destroys the strength of the wool, leaving it weak and practically worthless.

That the admixture of these sour wools with the other wools shipped from Russia to the United States is done to deceive either the American importer or manufacturer, or perhaps both, admits of no doubt.

PATENT.

Fabrics from Paper Pulp, Impts. in the Manufacture of. C. Brodbeck, Paris. Eng. Pat. 23,598, Dec. 10, 1895.

The fabric formed from sulphite cellulose is perforated by rows of needles, which do not cut the fibres, but push them aside, the holes thus formed not weakening the fabric, but allowing the gases and vapours of the skin to escape. To prevent the cellulose from tearing, it may be formed on an open groundwork fabric of cotton, linen, hemp, &c. In order to give the sheet a soft, smooth, fabric-like finish and a silky lustre, it is subjected to an appropriate hammering or beetling, with a slight addition of appropriate finishing ingredients, and subsequent calendering.—S. P. E.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Bleaching Jute, &c. Grothe. Leipziger Färber- u. Zeugdr. Zeit. **46**, [3], 115.

According to the author, a better white is obtained if the goods to be bleached are treated with sodium silicate than if they are boiled with soda alone. Jute may be bleached satisfactorily with bleaching powder, if it has previously been steeped for 15—20 minutes in a solution of sodium silicate, in the proportion of 6—8 kilos. of silicate for 10 litres of water, at a temperature of 85—100° C.—I. S.

Printing of Cotton and Woollen Fabrics, The Use of Turpentine in the. C. Casanovas. Färber Zeit. **8**, 33—34.

In certain cases where it has been hitherto impossible to print several colours simultaneously, with a machine with several rollers, owing to running of the colours one into another, the author has succeeded in accomplishing this by the addition of turpentine to one or more of the printing colours, since this addition entirely prevents any "bleeding." For example, on a light vat-blue ground there is printed a white or coloured bichromate discharge containing also a resist for aniline black, and an aniline black printing colour is applied in the same machine, an addition being made to this colour of 70—100 grms. of turpentine per litre.

By the addition of turpentine to a sodium citrate discharge, the latter may be printed on mordanted calico immediately after padding with the mordant.

A further effect of turpentine is to prevent the printed colour from penetrating the fabric. This is particularly useful in the printing of woollen cloth, where unevenness frequently results from irregular penetration of the colour during drying, and in order to obviate this defect, in addition to the use of turpentine, drying should be effected by means of a *moving current* of hot air.—R. B. B.

Ammonium Phosphate for the Fixation of Mordants. Leipziger Färber- u. Zeugdr. Zeit. **46**, [3], 107.

Ammonium phosphate is recommended in place of sodium phosphate for fixing mordants in calico printing, it being alleged that this salt, being less powerful in its action than the sodium salt, gives better results than the latter.—I. S.

Albumin at Chinkiang; Manufacture of. U.S. Cons. Reps., March 1897, 386.

An enterprising German firm has undertaken the manufacture of albumin at Chinkiang, it having been found that this place offered the best field for the business on account of the cheapness and plentiful supply of eggs. The factory is now successfully established, with possibilities of conducting it on a much larger scale.

The establishment of this albumin factory has made a good market for the eggs, and the country people bring them in by thousands. The factory uses duck eggs in the manufacture of albumin in preference to chicken eggs, solely on account of their greater cheapness. They are bought, not by the dozen, but by the thousand, the factory paying an average of 8 dols. (Mexican) per thousand.

As soon as the eggs are received at the factory they are tested by a very simple but ingenious process. They are held in pairs in front of a circular opening in a tin cylinder open at the top, containing a lamp giving a powerful light, which is concentrated on the opening. Every speck and flaw in the egg is distinctly seen in this process, and all eggs showing the slightest sign of defect are at once rejected. The percentage of rejected eggs is remarkably small, rarely amounting to 50 per 1,000. The good eggs are sent to the rooms prepared to receive them, where a number of Chinese girls are employed to break them and to separate the yolk from the white, placing each into separate vessels. The white then undergoes one of two processes—it is either carefully cleaned of all stray clots of yolk and then put through a course of stirring in a large vessel by means of a heavy rod or paddle worked by hand, when it is ready for the drying room; or, after being cleaned, it is run into large vats and allowed to ferment, the process of fermentation being accelerated by the admixture of certain chemicals, among which are salt, acetic acid, boracic acid, ammonia, and calcium chloride. When it is found in drawing off that all impurities have either been deposited or thrown off in the form of a thick froth, and the resulting liquid is albumin, as clear as water, the albumin is ready for the final process of drying. This is performed in a series of three rooms, each hotter than the last, the temperature ranging from 40° to 50° C. The albumin is run into shallow tin pans about 1 ft. square, which are placed in ranges of shelves in the drying rooms. Frequent examinations are made to see that the temperature is maintained, and that the process of drying is sufficiently gradual, uniform, and complete, a period of 60 to 70 hours being found necessary for that purpose. When thoroughly dry, the albumin, which now presents the appearance of isinglass, with a yellow tinge, is, after cooling, placed in large square packing boxes, lined with tin, holding about 400 lb. each, and is ready for shipment.

The principal use of albumin is in the preparation of fast dyes for superior kinds of cotton goods, and it is shipped to England, Germany, and France.

The yolk of the eggs, after separation from the white, is passed through two sieves—one coarse and one fine—and is then run off into vessels holding about 20 lb. each, in the form of a yellow, even-running liquid of the consistency of rich cream. It is then poured into vats holding about 100 lb., and a 10 per cent. solution of salt, borax, and calcium chloride is then added for the purpose of preservation. The mixture is thoroughly stirred until it assumes the consistency of thick molasses and is of a dark orange colour, and is then poured, or rather forced, through a funnel into a cask holding about 500 lb. net. It is then ready for shipment. This is used in the preparation and dressing of superior leather goods, and is in much demand in England, Germany, and France. No use has yet been found for the empty egg-shells, and they are thrown away.

Antimonio-Tungstates, and the Separation of Tungsten and Antimony. L. A. Hallopeau. Bull. Soc. Chim. 1897, **17**, 170—175.

See under XXIII., *page* 358.

PATENT.

Gelatinised Paper or other Fabric suitable for the Reproduction, by Printing with Fatty Inks, of Writings, Drawings, and the like: Impts. in the Manufacture of. C. Raymond, Suresnes, France. Eng. Pat. 1556, Jan. 22, 1896.

The patentee claims the application to the lower surface of a paper or other fabric which has been gelatinised, of a suitable solution of one or more metallic salts (sulphate of copper, iron, zinc, lead, &c.), with or without a bichromate or chromate.

It is stated that the strength of the paper is thus increased, and also the fineness, firmness, and penetration of the transfer.—A. S.

VII.—ACIDS, ALKALIS, AND SALTS.

Sodium Nitrate, Newly Discovered Deposits of, in Colombia. Zaracristi. Berg- u. Hüttenmänn. Zeit. 1896, **55**, 391.

Deposits of crude sodium nitrate have been discovered by the author in the San Sebastiano river district, 100 kilom. from San Juan de la Cienaga, and apparently covering an area of about 75 sq. kilom. The beds are found at a depth of $2\frac{1}{2}$—6 m. below the surface of the soil, and resemble in general character the deposits found in Chili and Peru—*i.e.*, consist of a more or less stratified gypsumous and chalky material impregnated with sodium nitrate, chloride, &c. Clean fragments of the nitrate have the sp. gr. 2·01, and consist of: $NaNO_3$, 23·1; NaCl, 34·1; $CaSO_4$, 8·5; $Al_2(SO_4)_3$, 3·4; SiO_2, 24·7; MgO, traces, and H_2O, 5·5 per cent. The average composition of the mass is: $NaNO_3$, 11·4; $CaCO_3$, 32·5; $CaSO_4$, 20·1; SiO_2, 32·4; $Ca_3(PO_4)_2$, 2·5; Fe_2O_3, traces, and organic matter, 1 per cent. It will be seen that in point of richness these deposits cannot compete with Chili nitrate.—H. T. P.

Double Haloid Salts of Lead and Ammonium. H. Fonzes-Diacon. Bull. Soc. Chim. 1897, **17**, [6], 346—356.

The haloid salts of lead are soluble in more or less concentrated solutions of the ammonium haloid salts. On cooling, crystalline double salts separate, generally in the body of the liquid, and the mother-liquors will deposit, on the least fall in temperature, the salts with which they are saturated. Thus the crystalline deposit will be a mixture of several salts. In order to obtain from this the pure double salt, the author washes with a dilute solution of the ammonium compound, which is, however, sufficiently concentrated to prevent the dissociation of the double salt. Microscopic examination shows that the salts so obtained are homogeneous. In the analysis, the salts were converted into sulphates, and ammonium sulphate driven off by the application of a dark red heat. Ammonia was displaced by potash and collected in standard acid. To determine the halogens, the salts were dissolved in potash and excess of acetic acid added; in this solution the total halogens were determined by precipitation with silver nitrate, and also the silver required to precipitate the halogens was found volumetrically, after removal of lead by sulphuretted hydrogen; the two results enabled the amount of each halogen to be calculated.

The following salts were isolated:—

Type MX_3A: $Pb(ClI_2)(NH_4).2H_2O$ and $Pb(BrI_2)(NH_4).2H_2O$; the corresponding acid $PbI_3H.5H_2O$ has been isolated by Ditte.

Type MX_4A_2: $Pb(Br_2Cl_2)(NH_4)_2$, $PbCl_4(NH_4)_2$, $PbBr_4(NH_4)_2$, to which the acid PbI_4H_2 of Lassaigne corresponds.

Type M_2X_5A: $Pb_2(Br_4Cl)(NH_4)$, $Pb_2Cl_5(NH_4)$, $Pb_2Br_5(NH_4)$, $Pb_2(BrI_4)(NH_4)$, $Pb_2(Cl_4Br)(NH_4)$.

Also a fourth group containing the salt $Pb_3I_{10}(NH_4)_4.6H_2O$ alone.

No double salts could be obtained from ammonium fluoride, its action gave rise to the salts PbClF and PbBrF.
—A. C. W.

Sulphocyanides (Thiocyanates) in Gas Liquors, Conversion of, into Alkali Ferrocyanides. A. Dubosc. J. de l'Eclair. au Gaz. **45**, [1], 10—12.

The extended application of sulphocyanides (thiocyanates) in dyeing, calico printing, match making, photography, &c. has directed attention to waste gas-liquors as a source of supply, since considerable quantities of these salts are contained in the mother-liquor from which the ferrocyanide has been recovered, and also in the ammoniacal liquor. In the first case, the ferric thiocyanate solution is treated with potash or soda, to throw down the iron, and the clear liquid is evaporated down. The ammonium sulphocyanide in the ammonia liquor is converted into insoluble copper sulphocyanide by the aid of a cuprous salt, and, the copper being displaced by an alkali, the liquid is concentrated as before.

For the conversion of these sulphocyanides into ferrocyanides, Tcherniac's method—heating to 450° with finely-divided iron, and treating the mass with water—may be employed.—C. S.

Colour Reactions yielded by Tartaric and other Organic Acids. E. Piñerua. Comptes rend. 1897, **124**, [6], 291.

See under XXIII., *page* 361.

Antimonio Tungstates, and the Separation of Tungsten and Antimony. L. A. Hallopeau. Bull. Soc. Chim. 1897, **17**, 170—175.

See under XXIII., *page* 358.

Liquefied Ammonia, Testing Commercial. H. Bunte and P. Eitner. J. für Gasbeleucht, 1897, **40**, 174.

See under XXIII., *page* 358.

Perchlorate, Estimation of, in Saltpetre. F. Winteler. Chem. Zeit. **21**, 1897, 75.

See under XXIII., *page* 358.

PATENTS.

Alkaline Salts, Impts. in Obtaining from Blast-Furnace or other Gases, and Apparatus therefor. G. Craig, Cumnock, N.B. Eng. Pat. 4489, Feb. 28, 1896.

When coal or coke is burned, as in blast furnaces, whether alkalis be added or not for the production of cyanides, the gases blown or drawn off are charged with alkaline salts, chiefly cyanides and carbonates of sodium and potassium. To recover these salts, the gases, after passage through a metal cooling trap, enter a horizontal conveyor tube, in which the salts that deposit are mechanically carried forward, and at the end fall through a short vertical passage into a filter chamber, where they are caught on the uppermost of a series of perforated plates, and spread out evenly by the action of a series of revolving scrapers. The gases, after filtering through the layers of deposited salts and the perforations in the plates, escape into a vertical side passage to the outlet main, whence they may be utilised for heating or other purposes. The excess salts swept off by the scrapers into the corner spaces provided, fall through openings into a conveyor tube for conveyance to a receptable.
—E. S.

Bicarbonate of Soda, Impts. in Apparatus for Filtering, Washing, and Drying, suitable for the Treatment of. W. L. Wise, London. From Solvay et Cie., Brussels, Belgium. Eng. Pat. 4725, March 2, 1896.

The apparatus, though specially designed for the treatment of sodium bicarbonate resulting from the ammonia process, is also applicable to all substances having similar physical properties. The bicarbonate is kept in aqueous suspension in a trough provided with an inlet pipe, and an overflow vertical pipe. A drum revolves in the trough about a horizontal, hollow axle, receiving a tube at each end connected to a pump or other arrangement for maintaining a partial vacuum within. The periphery of the drum is formed of a perforated metal sheet covered with a fine metallic cloth on which a piece of flannel is secured to form the filtering surface. As the drum revolves, the internal

suction draws in liquid to a higher level within than without, and a coating of the bicarbonate forms on the surface. To prevent too great obstruction of the pores of the filtering material, from time to time internal pressure is substituted for the suction, or the pressure of the higher liquid internal level is allowed to act, on the principle of counter currents, so as to reopen the pores.—E. S.

Basic Lead Carbonate, Improved Process of Producing and of Obtaining certain Bye-Products [Caustic Soda and Nitric Acid]. The Chemical and Electrolytic Syndicate, Lim. O. J. Steinart and J. L. F. Vogel, London. Eng. Pat. 6655, March 26, 1896.

See under XIII. A., *page* 340.

Sulphuric Acid Making Apparatus, Improved Joint or Connection for Use with Pipes in. P. Duggan, Rosherville, Kent. Eng. Pat. 5012, March 5, 1896.

The pipe leading the gas from the pyrites kiln to the acid tower is constructed at the end entering the Glover's tower with a hollow jacket, closed at its outer end by a cover plate, made in halves and bolted on, into which plate two small tubes pass, one for the entrance and the other for the exit of a flow of water. Alternatively, the cover plate may be removed, so as to admit a current of air only into the hollow jacket. The lead sheathing of the acid tower is worked out to a flange surrounding the water jacket, and is kept in place by an iron strap.

Instead of the connecting pipe being taken from the brickwork of the stack, it is preferably taken from a metal dome erected on the top of the stack, and the end of the pipe that enters the acid tower is made as a separate piece, and is connected to the dome by means of a removable intermediate piece. This arrangement facilitates the renewal of the pipe, when that is necessary.—E. S.

Bay Salt, An Impt. in the Treatment of. A. MacNab, London. Eng. Pat. 7464, April 8, 1896.

The larger are separated from the smaller crystals of bay salt by sifting or screening in brine.—E. S.

Zinc and Alkalis, Impts. in the Electrolytic Production of. [Two Diagrams.] O. Dieffenbach, Darmstadt, Germany. Eng. Pat. 25,804, Nov. 16, 1896.

See under X., *page* 335.

Ammonia, An Improved Method or Process of Treating Substances containing Nitrogen for the Manufacture of. H. H. Lake, London. From C. Pieper, Berlin, Germany. Eng. Pat. 28,199, Dec. 9, 1896.

The customary processes of treating nitrogenous matters for obtaining ammonia, consisting in successive drying, destructive distillation, and in steaming the coke produced, as distinctly performed operations, are modified by conducting the processes in a series (six being specified) of connected retorts, so as to admit of continuous working. The steam generated in the first retort of the series is conducted through a superheater, and then to other retorts in a specified order, which may be modified for adaptation to a "parallel connection method," or to a "series connection method"; in the first system, the superheated steam enters first that retort which is in the last stage of the steaming process; while in the second method the order is reversed, though later on the first-described order is adopted. The "steaming" process is stated to require a temperature of over 500° C.—E. S.

VIII.—GLASS, POTTERY, ENAMELS.

Kaolins [Pottery], Knowledge of, from a Manufacturing Point of View. Seger. Monit. Céramique, **28**, 53 and 64.

Analyses of kaolins, which state merely the composition of the clayey matter and neglect the subsidiary mineral substances, are insufficient to afford an explanation of the different behaviour of the various classes in practice; nor does the ultimate analysis of the whole mass of the substance assist the manufacturer, who desires rather to know the proximate constituents of his raw material. The subjoined table shows the variable nature of different kaolins used for pottery, and demonstrates the approximately regular composition of the actual clay in all, revealing at the same time a wide range in the proportion of the three proximate constituents, sufficient to explain the considerable differences of the kaolins when subjected to manufacturing processes.—C. S.

	Kaolin from							Kaolin Used at Royal Pottery, Charlottenburg.
	Lolez.	Kottiken.	Tremosna.	Zettlitz.	Lettin.	Kaschkau.	Sennewitz.	
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Elementary composition—								
Silica	49·19	49·91	49·48	46·82	57·08	56·72	64·87	63·07
Alumina	36·73	35·99	36·64	39·49	29·94	31·07	23·83	24·67
Iron oxide	0·81	0·63	0·66	1·09	0·65	0·59	0·83	0·59
Magnesia	0·18	0·30	0·41	Traces	0·49	0·22	0·50	0·40
Potash	1·18	0·75	1·60	1·40	2·26	0·51	1·39	4·25
Water	12·41	12·34	11·99	12·86	9·87	11·18	8·36	7·00
Total undecomposed mineral matter.	*11·74*	*12·59*	*9·71*	*3·45*	*25·91*	*21·49*	*36·22*	*45·08*
Containing—								
Silica	9·05	10·65	7·91	3·04	23·28	21·29	35·98	38·09
Alumina	1·70	1·21	1·09	0·21	1·73	0·11	0·14	4·15
Alkalis (by difference)	0·90	0·73	0·71	0·20	0·90	0·09	0·10	2·84
Proximate constituents—								
Clay	88·22	87·41	90·29	96·55	74·09	78·51	63·77	54·92
Quartz	3·08	6·40	4·68	2·30	17·21	20·99	35·50	23·52
Felspar	8·66	6·19	5·63	1·15	8·70	0·59	0·73	21·56
Composition of the clay—								
Silica	45·36	44·76	45·98	45·36	45·63	45·00	45·30	45·46
Alumina	39·58	39·65	39·36	39·71	38·08	39·32	37·15	37·35
Iron oxide	0·92	0·72	0·73	1·13	0·88	0·75	1·29	1·07
Magnesia	0·20	0·34	0·45	..	0·66	0·28	0·78	0·73
Potash	0·21	0·02	0·39	1·24	1·84	0·53	2·02	2·57
Water	14·02	14·07	13·28	13·32	13·32	14·20	13·11	12·74

Glass Pots and Trough Furnaces, Clay for. H. Seger and E. Cramer. Thonind. Zeit. **21**, [6], 47–49.

The chief qualities requisite in clay for making glass pots are:—Capacity to withstand the highest temperatures necessary for fusing the most refractory glass; sufficient plasticity to permit of the admixture of some 50 to 60 per

cent. of granular burnt fireclay; a comparatively low sintering point; and power of resisting the solvent action of the fused charge. This latter property is possessed in the highest degree by the clays approximating in composition to the formula $Al_2O_3 2SiO_2, 2H_2O$.

Concerning the fineness of the burnt fireclay, it is found advisable to use up the fine powder formed during the grinding, as well as the coarser particles, in order to ensure a more perfect cohesion of the materials by reducing the shrinkage of the clay. The addition of burnt fireclay raises the sintering point of the clay considerably. For example, the sintering point of a clay, initially the same as the No. 1 Seger cone, was raised to the equivalent of the No. 5 cone, by mixing with 120 per cent. of burnt fireclay, and another from No. 6 cone temperature to that of No. 15 cone, by addition of 50 per cent. of burnt fireclay.

The best clays for this purpose are found at Vallendar, Mehlem, Gross-Almerode, Montabaux, Grünstadt, Passau, Deggendorf, Klingenberg, Saarau, &c., in Germany: Pilsen, Melnik, Goltivech, and Leoben, in Austria; Gluchow in Russia; Höganäs in Sweden; Stourbridge and Garnkirk in Great Britain; Forges les Eaux in France; and Huy in Belgium.

A comparative examination of several samples of Belgian and Girode (German) clay showed that in the burnt condition the former contained between 52·72 and 78·1 per cent. of silica, 19·5 and 42·39 per cent. of alumina, and 0·9 to 1·4 per cent. of iron oxide; whilst the Girode clay contained per cent., 52·72 to 55·89 of silica, 40·44 to 44·85 of alumina, and 1·17 to 2·07 of iron oxide. The fusing points coincided in the former case with Nos. 28 to 39 Seger cones, and in the latter with Nos. 34 and 35. In respect of plasticity, shrinkage, and sintering temperature the Girode clay was approached only by the best of the Belgian samples, the remainder containing too much sand and requiring too high a firing temperature to be suitable for glass pots.—C. S.

Sheet-Glass Making, German and Belgian Methods of. W. M. Sprechsaal, **30**, [1], 3.

The chief difference between these methods is in the form of the cylinder from which the sheet is prepared, the practice in the one case (German) being to make a short wide tube, the length of which forms the shorter dimension of the sheet, whilst in the Belgian system the tube is long and narrow, the length of the cylinder being that of the sheet. The advantages of the latter method are:—The facility with which long sheets can be made—it being very difficult with the German system to make them over 1·5 m. long, whereas 2-metre lengths can be easily made by the other process—and the diminished proportion of waste in the shoulder, which, in the case of wide cylinders, amounts to ¼ per cent. of the mass. The Belgian method has, it is said, almost entirely supplanted the other, even in Germany. —C. S.

PATENTS.

Oven, An Improved, for Burning or Fusing Lettering, Designs, Colours, and the like into or upon Articles of Glass, Earthenware, and other Glazed or Enamelled Goods. F. Albrecht, Liverpool. Eng. Pat. 4189, Feb. 25, 1896.

This oven is intended more particularly for use in carrying into effect Mr. Knapp's process for the production of designs upon glass (Eng. Pat. 13,838 of 1893; this Journal, 1893, 927). The oven is a square box of sheet-metal plates, strengthened by angle-iron bars. It is provided with a pair of doors, is mounted upon a firebrick setting, and is cased in brick, a space being left all round to allow of the circulation of the products of combustion.—V. C.

Glass, Impts. in Furnaces [Utilisation of Heat] suitable for Use in Manufacture of. F. Radcliffe, Plumstead, Kent. Eng. Pat. 4945, March 4, 1896.

A chamber is added between the melting and the working chamber, which serves as an exit flue. This added chamber is nearly surrounded by the working chamber, whereby the hot gases from the melting chamber "travel practically over the whole surface of the molten metal in the working chamber, before escaping therefrom."—V. C.

Ceramic Stone Objects, Manufacture of, by Devitrification of Glass. L. A. Garchey, Lyons. Eng. Pat. 5772, March 14, 1896.

The objects are produced by *first*, partly devitrifying the glass powder in a furnace; *secondly*, completing the devitrification and fusing at a high temperature; *thirdly*, moulding under pressure in a frame with a movable bottom plate. By stirring in fused coloured glass, veined marble may be imitated.—V. C.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Mortar, Influence of Sea-Water on. Thonind. Zeit. **21**, 128—130 and 142—144.

The results of the experiments carried out at La Rochelle since 1856 with cubical blocks (0·6 m. linear) of masonry, cements, and mortars of different composition, are summed up by Viennot in the following conclusions: (1) Cement employed without any admixture crumbles sooner than certain mortars; (2) Mortars containing either 1 or 2 volumes of sand per volume of cement—650 or 1,300 kilos. of cement per cb. m. of sand (volume being taken by reason of the variable amount of moisture in sand)—offer the greatest resistance to sea-water; some of the blocks lasting for 38 years. The same authority also concluded from a second series of experiments with cement, concrete, and slag cement, between 1880 and 1895, that: (1) Hydraulic lime mortars begin to decay in sea-water after one or two years, and are completely disintegrated after a maximum period of 15 years; (2) Concrete, on account of the compression effected, resists better than masonry; (3) Quick-setting cements begin to decay after five to eight years' exposure; (4) Slow-setting cements begin to crumble after six to eight years, but resist disintegration for over 38 years; (5) The best proportions for cement and sand are those given above.

One most important fact revealed by these experiments is that not one of the hydraulic cementing materials known is capable of resisting the action of sea-water when impregnated by the latter; and another is the necessity for the absence of free lime capable of slaking after the cement is mixed, whether due to an insufficient amount of silica and alumina to saturate the base, or to the irregular admixture of separately calcined materials. The danger arising from the presence of such free lime increases in proportion with the calcining temperature employed, especially in the case of the natural Portland cements, the expansion occurring on the absorption of sea-water resulting in disintegration of the mass. For minimising the permeability of the cement, the best results are obtained by using a coarse sand, the mass being less porous than when fine sand is employed; and as far as the cement itself is concerned, Portland cement is superior to Roman cement, Teil lime, &c., though theoretically these latter should be the best. The reason for this is to be found in the ratio between the amount of water required for mixing and that actually taken into combination with the cement; Portland cement, for example, combining with 18 to 20 per cent. out of the 25 per cent. necessary for mixing, whilst hydraulic lime and Roman cement retain only 8 to 10 per cent. out of 40 to 60 per cent., and slag cement only 5 to 6 per cent.; and one of the advantages possessed by coarse sand over fine, consists in the maintenance of this ratio, the amount of water required by the former being comparatively small; whilst the porosity of the mass is slight, amounting in the case of a mixture of 650 kilos. of cement and 1 cb. m. of sand, to only 9 per cent., whereas with lime in the same proportion the porosity is 24 per cent. To the same circumstance the superiority of concrete is due. The brittleness of unmixed cement is due to excessive crystallisation and the expansion of the free lime during hydration, the advantage of an admixture of sand being shown by tests wherein pure cement, after four years' exposure in sea-water, resisted a pressure of 6·4 kilos. per sq. cm., whilst that mixed with an equal weight of lime withstood up to 54·7 kilos.—C. S.

Bricks and Brickwork, Incrustations on, or Efflorescence, through Weathering. B. Deutsche Bau Zeit.; Thonwaaren Industrie, **11**, 21 and 35.

This subject was treated by H. Günther in an inaugural address at the University of Rostock. Vegetable growths appear on light-coloured stone surfaces exclusively, and require for their development moisture and shade. Mineral incrustations are mostly white or grayish, more rarely yellow or green, these latter being due to vanadium. In appearance these incrustations vary, according to the solubility of the component salts, from floury or woolly powders to stalactitic masses, and may result from various causes present either in the raw clay, or introduced in the water employed in brickmaking, or from pyrites in the clay or fuel, or from the ash of the latter material. Again, the infiltration of soluble substances from the mortar, or combination of the alkalis in the latter with the gypsum in the bricks; or finally the absorption of nitrates from the soil and ammonia or sodium chloride (near the sea) from the air may give rise to incrustation.

The sulphates and other soluble salts in crude clay are brought to the surface of the bricks by the evaporation of water in the drying process, and if this is conducted irregularly so that the water evaporated in one part of the kiln is condensed in another and re-evaporates as the heat diffuses, the bricks practically undergo a process of lixiviation, which results in the formation of injurious cracks. In burning, the sulphur of any pyrites present in the clay or coal becomes finally oxidised to sulphuric acid, which combines with the bases, and especially with lime, this being particularly the case in the continuous system of burning, by reason of the excess of air admitted. This reaction is outwardly manifested by an intense red coloration on the affected surfaces.

From the mortar, a portion of the caustic lime may be extracted and on exposure to the air may form an incrustation of carbonate: or the other caustic alkalis may by reaction on the gypsum of the bricks give rise to an efflorescence of sulphates, especially of sodium. Nitrates are formed by bacterial action on ammoniacal compounds absorbed from the air, or may be taken up from the ground with which the bricks are in contact.

Whilst incrustations of calcium carbonate do no harm beyond spoiling the appearance of the bricks, the soluble alkali salts, by the repeated solution and recrystallisation in the interior, resulting from atmospheric changes, cause disintegration on exposure to the weather. The tendency to rot from this cause increases with the percentage of lime or magnesia in the clay.

To prevent these incrustations, pyrites and sulphates can either be removed by the slow process of seasoning the clay by prolonged exposure to the weather before making up into bricks, or by adding barium salts to the clay before burning, so as to produce the insoluble barium sulphate. Another means of assistance is the prevention (by restriction of the air supply) of the oxidation of the sulphur beyond the SO_2 stage: and finally by burning the bricks very thoroughly, since in that state they are less disposed to absorb the moisture necessary for the extraction of the soluble salts.—C. S.

PATENTS.

Artificial Stone [from Sand and Lime], Impts. in the Manufacture of. S. Neffgen, Mülheim-on-the-Rhine, Germany. Eng. Pat. 3972, Feb. 21, 1896.

Nearly dry slaked lime is mixed with quartz sand, moulded in the required shape, and placed in a heating chamber. The temperature is then raised so as to attain 50° C. in 5 or 6 hours, and 80° C. after a further 12 hours. This temperature is maintained constant for 2 or 3 hours, and then the heat is rapidly increased to 95° C., and there maintained for from 4 to 6 days. The last operation produces a smooth, compact surface of calcium silicate. The pieces have a uniform colour throughout, and the smooth surface does not require any finishing operation.—V. C.

Compressing Wood and Utilising Certain Waste Products therefrom, Impts. in. A. de Pont, Lancaster. Eng. Pat. 5973, March 17, 1896.

The inventor claims (1) "the process of compressing wood to increase its density, which consists in subjecting it to an equal pressure from both sides whilst it is maintained at a high temperature, sufficient to soften or liquefy the gums therein, and retaining such pressure until it is cool and the gums have set"; and (2) "the process of constructing or manufacturing a product resembling wood, which consists in mixing with or adding to sawdust or wood refuse a quantity of gums, resins, or other suitable agglutinants, either in a dry state or dissolved, and subjecting the compound to a great pressure at a temperature sufficiently high to soften or melt the gums or resins."
—V. C.

Readily Fusible Composition or Cement, Impts. in or connected with. T. Smith, Richmond, Surrey. Eng. Pat. 8595, April 23, 1896.

"A fusible composition or cement [of the class known as 'Spence's metal'], composed of sulphides, sulphur (or bitumen, resin, refuse wax, or shale grease), slate dust or silica, alumina, bitumen, and wax." No coal or coke is mixed with the materials, which, in this improved process, are heated in a closed vessel.—V. C.

Artificial Stone, Impts. in, for Street Pavements, Pathways, and like Purposes, and in the Method of Producing the same. A. J. Boult, London. From J. Jungbluth, Cologne. Eng. Pat. 18,714, Aug. 24, 1896.

"An artificial stone consisting of a top layer of pulverised asphalt, a binding material of ground Portland cement, powdered unslaked lime, and ground trass, and a ground-material of hard porous stone in the form of powder and small pieces of about 5 mm. and 10—12 mm. in diameter." The binding materials are introduced in a very slightly wetted condition, the top material being introduced very hot. Combination is brought about by a pressure of about 300 atmospheres, and the setting, after removal from the mould, takes place under the action of a water spray.—V. C.

Drying or Sterilising Wood, A New or Improved Process and Means for. A. L. C. Nodon and L. A. Bretonneau, both of Paris. Eng. Pat. 19,047, Aug. 28, 1896.

The wood is steeped in a concentrated solution of soda soap, and afterwards in a solution containing a soluble salt of an alkaline-earthy base, and is then dried in air. It is stated that "the density of the wood has increased; the colour has not changed; it has a silky appearance; it does not crack, nor does it split; wet does not affect it, nor sun; and it is unattackable by insects."—V. C.

Slating Compounds [Writing Slates], Impts in. W. P. Thompson. From W. H. Brawley, J. B. Coles, and C. N. Coles, New York, U.S.A. Eng. Pat. 22,157, Oct. 6, 1896.

The inventor proceeds as follows:—To about 1 lb. of shellac, ½ lb. of borax and about 1 gal. of water are added, and the solids allowed to dissolve. The solution is then diluted with about ½ gall. of water, and ½ lb. of alumina or cryolite is added. This liquid is applied as a first coating to the cardboard or other sheet, which is then allowed to dry. A second coat of material is then put on, which makes the paper thoroughly waterproof, the surface being smooth and even, and the texture of the paper not being destroyed. The surface of such material is also capable of taking the marks of a lead pencil, which may be readily erased or rubbed out again.—S. P. E.

Paving and like Purposes; Impts. in the Manufacture of Compositions for. [*Tar Paving.*] J. A. Just, Syracuse, New York, U.S.A. Eng. Pat. 23,105, Oct. 17, 1896.

A composition prepared by treating tar with sulphur at from "138° C. to 204·5° C.," adding rosin, also a sulphurised heavy hydrocarbon, and agitating the mixture. The asphalt-like material, mixed in the proportion of about 15 parts to 85 with fine sand, forms a paving material.
—V. C.

Burning Cement, Lime, and the like, and for Smelting Ores; Impts. in Furnaces for. A. Stein, Wetzlar, Germany. Eng. Pat. 27,219, Nov. 30, 1896.

Instead of using a ining of fire-proof material, the inventor seeks to protect the furnace shell from overheating by means of open passages *outside* the shell, and "by means of the draught or current of air rising up in the same, or it may be by radiation of the heat, there is produced an effective cooling action."—V. C.

X.—METALLURGY.

Gold in Formosa. Eng. and Mining J. 1897, 189.

Although the present alluvial gold-fields have only been discovered about 10 years, there are now engaged more than 3,000 workmen collecting gold on the upper reaches of the Tamsui river. According to the Chinese Customs returns, over 700,000 taels of gold-dust have been exported to Hong Kong and Shanghai. In addition to this, large quantities have been smuggled from the island, and it is anticipated that, with the use of suitable machinery and with proper supervision, the output of gold will greatly increase.—A. W.

Gold, Cyanide of: Solution and Precipitation of. S. B. Christy. Trans. Amer. Inst. Mining. Eng. 1896, 1—38.

The paper is divided into three main sections, with sub-headings:—

I. The Solution of Gold in Cyanide Solutions.

1. *Solution of Metallic Gold in Potassium Cyanide.*—The author confirms Ellsner's well-known reaction ($4KCy + 2Au + O + H_2O = 2AuKCy_2 + 2KHO$), and finds that pure potassium cyanide dissolved in pure water is entirely without action on metallic gold; but when oxygen is admitted to the solution, the metal dissolves at a rate proportional to the oxygen added, but without immediately oxidising the cyanide to cyanate, as MacArthur had assumed.

2. *Effect of Oxidising Agents.*—Oxidising agents, like oxygen itself, when added to the cyanide solution, increase the solubility of the gold. Among the most convenient and efficient of these were found to be the peroxides of sodium and manganese, and potassium ferricyanide: but the chlorate, nitrate, permanganate, and bichromate of potash, and the peroxides of barium and lead, were also effective. It thus being apparent that the essentials of the cyanide process were free cyanogen to form gold cyanide, and free potassium cyanide to form the double cyanide, cyanogen bromide was added to potassium cyanide liquors in order that nascent cyanogen might be formed (this Journal, 1895, **14**, 870); this was so successful (in 1894) that experiments were extended, and bromine water was used in place of the bromide. The chemical effect of the two reagents is the same, and the author prefers the bromine water, in opposition to the opinion of Teed. The bromine solution must be added little by little as required, otherwise cyanogen is generated faster than it can be used, great loss of this substance takes place, and para-cyanogen and dark-coloured oxidised compounds are formed. Both bromine and cyanide liquors must be very dilute. The reactions are as follows: (*a*) $2KCy + Br = KBr + Cy + KCy$; and (*b*) $Au + Cy + KCy = AuKCy_2$, rather than as given by Mulholland (this Journal, 1895, **14**, 662), in which the influence of oxygen is postulated. The relative gold-dissolving values of bromine, chlorine, and oxygen are shown by the fact that 1 oz. of each element, respectively, will lead to the solution of 2·45, 5·52, and 24·5 oz. of gold. Oxygen is often to be preferred, and is usually present in the cyanide solution in sufficient quantity for the treatment of low-grade ore; in treating higher grade ore the greater solubility of the halogens gives them the advantage; but the solution must not be too strong, for the reason given above, and because secondary actions may result from the presence of sulphides, &c., with the generation of cyanicides. Even with rich ores, unless used with the nicest discrimination, the addition of oxidising agents may do more harm than good.

3. *Solubility of Gold Sulphide in Potassium Cyanide.*—The occurrence of gold as sulphide is sometimes disputed, although it is known to be found as telluride. The combination of sulphur and gold in the dry way could not be effected, and therefore experiments were made with Au_2S_2 precipitated from solution by hydrogen sulphide. This compound was found to dissolve in a 5 per cent. solution of KCy in 4 minutes, in a 1 per cent. solution in 10 minutes, and in a $\frac{1}{5}$ per cent. solution in an hour. No oxygen is necessary to the action, which is somewhat complex; three reactions having been observed, depending upon the strength of the cyanide solution used:—(*a*) $Au_2S_2 + 4KCy = 2AuKCy_2 + K_2S + S$: (*b*) $KCy + S = KCyS$; (*c*) $Au_2S_2 + K_2S = Au_2K_2S_2 + S$. The presence of suspended sulphur was most apparent in dilute cyanide solutions.

4. *Solubility of Gold Telluride in Potassium Cyanide.*—Tellurides of gold and silver, prepared by direct combination of the elements, or obtained from natural sources, are only dissolved by the cyanide liquor with the greatest difficulty. This is probably because the affinity of tellurium for potassium is less than that of sulphur, and because tellurous acid is formed by oxidation, and retards the oxidation of the potassium and the solution of the gold.

5. *Solubility of Gold Antimonide in Potassium Cyanide.*—This compound behaved like the telluride, a copious precipitate of antimony oxide forming in the solution, and retarding the action.

II. The Precipitation of Gold from Cyanide Solutions.

Potassium aurocyanide solutions contain the gold in a most stable form, and text books are wrong in stating that the yellow gold cyanide is precipitated on acidification with sulphuric acid from (say) a 1 per cent. cyanide solution. Only hot concentrated sulphuric acid causes such a separation; and the cyanide of gold (AuCy) is more stable than the telluride, requiring the strongest hot sulphuric acid to decompose it, with the deposition of metallic gold. All indications tend to show that the gold exists in the solution as aurocyanhydric acid ($HAuCy_2$) rather that as the double cyanide (AuCy.KCy).

1. *Regeneration of Potassium Cyanide from Dilute Solutions by Acidification.*—A 1 per cent. solution of KCy was not found to give any apparent trace of HCy when acidified with sulphuric acid; yet, if such a solution be allowed to stand for several weeks near an open vessel containing caustic potash, nearly all the KCy may be absorbed by the alkali. By passing air and steam first through the acidified solution and then through the solution of caustic (using the same air over and over again to eliminate the action of carbon dioxide), the free hydrogen cyanide in the stock solution could be reduced from 1 per cent. to 0·07 per cent. in 3 hours; without steam the same result was obtained in from 8 to 10 hours. On a large scale, absorption towers could be used. In this way a concentrated solution of any strength of KCy or $CaCy_2$ may be regenerated from a 1 per cent. stock solution with a loss of only 10 to 20 per cent. of cyanide. Alkaline sulphides must, however, be removed before treatment, by the addition of lead carbonate or sulphate.

2. *Precipitation of Gold from Aurocyanhydric Acid.*—Although free HCy could be removed by the last process down to 0·01 per cent, all the gold, even up to $603 per ton (0·1 per cent.), would remain dissolved in the clear, colourless solution. Oxalic or sulphurous acids or hydrogen sulphide fail to precipitate the gold, which can, however, be partially thrown down by nitrate of lead or mercury, and completely so by an excess of silver nitrate. The use of the silver salt would be practicable on the large scale if it were not for the cost of the reagent. Oxidising agents, such as potassium permanganate or the peroxides of lead and manganese, facilitate precipitation in acid solutions. Filtration through massicot was patented by de Wilde in 1894.

3. *Precipitation of Gold from Potassium Aurocyanide by means of Charcoal.*—An American patent was granted to W. D. Johnson in 1894 for the recovery of gold from cyanide liquors by passing them through a series of charcoal filters, the material from which, after calcination, was to be

smelted. It was claimed that 25 per cent. of the gold was removed by a single filtration, and that by repeated filtration 95 per cent. could be recovered. It is now found that the charcoal is capable of precipitating a certain percentage of its own weight of gold rather than a definite proportion of the metal present. Thus, with a 0·1 per cent. solution of gold ($603 per ton of solution), only 15 per cent. of the gold present was extracted after four filtrations when the liquid was acid, and only 6·5 per cent. when it was alkaline, the charcoal after filtration assaying 0·21 per cent. and 0·091 per cent. of gold respectively. But by employing 2,000 c.c. of a 0·001 per cent. gold solution (aurocyanide), in one case without, and in another case with 0·2 per cent. of free KCy, and filtering the liquid through glass tubes (10 cm. long and 2 cm. in diameter) containing 10 grms. of charcoal dust, it was found that the recovery of gold from the former solution amounted to 99·95 per cent., and that from the liquor containing free cyanide was 93·03 per cent., whilst the free cyanide in the latter instance was reduced to 0·118 per cent. The charcoal contained 0·2 per cent. and 0·187 per cent. of gold in the two experiments respectively. Filtration was effected under constant head. The first 800 c.c. took two days to filter, and then contained 0 and 0·01 mgrm. of gold in the two cases respectively; the remaining 1,200 c.c. required five days, and afterwards contained 0·01 and 1·4 mgrm. respectively. It would thus seem that free acid slightly raised the absorbing power of the gold, whilst free cyanide diminished it. The limit of saturation was probably reached in these experiments at about 0·2 per cent., which compares unfavourably with the concentrated material obtained by zinc, cuprous, or electrolytic precipitation.

4. *Precipitation of Potassium Aurocyanides by Mercuric Chloride.*—Fremy gives the following equation:—

$$2KAuCy_2 + HgCl_2 = 2AuCy_2 + 2KCl.HgCy_2.$$

But the reaction takes place very slowly in the cold. It is found to be hastened by warming; and on boiling for a few minutes, almost the whole of the gold may be precipitated as the easily filtered yellow cyanide. For practical work potassium cyanide could be regenerated in the filtrate by precipitating with just sufficient potassium sulphide to throw out all the mercury as sulphide; in this case the small quantity of gold left in the solution would not be lost. The yellow cyanide of gold may be ignited to pure gold at a low red heat. Free cyanide, however, prevents the precipitation, and the heating of the solution introduces complications industrially.

5. *Precipitation of Potassium Aurocyanides by Copper and its Compounds.*—Copper immersed in the aurocyanide solution causes the deposit of a yellow sediment, not upon the metal, but in the solution; in one experiment 75 per cent. of the gold was thus precipitated in three days, and then the copper was removed, but the deposit continued to increase for over a week, until only 11 mgrm. of gold remained dissolved. The successful use of cupric sulphide as a precipitant in the chlorination process led the author to experiment with it for cyanide work. Using freshly precipitated CuS and an aurocyanide solution containing 0·25 grm. of gold and 0·02 per cent. of KCy, the whole of the gold was precipitated from the solution when acidified with sulphuric acid, and two-thirds from neutral or alkaline solutions, after agitation with the CuS for four hours. The solution must contain free mineral acid, and not more than 0·1 per cent. of free HCy. The CuS.Aq produced by the action of H_2S on a solution of $CuSO_4$ is added to the gold solution, the mixture is stirred at intervals for several hours, and the gold will then be found in the residue. In one case 5 grms. of CuS completely precipitated 1 grm. of gold from 1,000 c.c. of acidified solution. The gold was readily recovered from the residue by digestion with sodium sulphide, in which the precious metal dissolved, leaving the CuS ready for further use. Electrolysis precipitates the gold from the sulphide solution, the more readily from concentrated solutions. Filtration through CuS may suffice for the precipitation of the gold in the first case; but agitation is preferred, and in the absence of free HCy, the gold is precipitated by the copper, atom for atom. It is noteworthy that not more than a trace of copper passes into solution when the gold is deposited, and it appears that the gold-containing precipitate is mainly a cuprous aurocyanide, or the corresponding sulpho-salt. For this reason cuprous chloride or cyanide (or other cuprous salt) is equally effective with the cupric sulphide. Cuprous thiosulphate (hyposulphite) could also be used, but the resulting precipitate is soluble in excess of either potassium cyanide or thiosulphate, so that complete precipitation is almost impossible, except after acidification. The author uses and prefers cuprous chloride as a precipitant, and adds it to the acidified solution (from which free HCy has been removed) until a drop of the filtrate from the resulting yellowish precipitate gives a red precipitate with potassium ferrocyanide. De Wilde (see this Journal, 1895, **14**, 163) also uses cuprous chloride, but forms it in the solution, and acidifies at the same time by means of sulphurous instead of sulphuric acid. The recovery of the excess of cyanide recommended by de Wilde is possible, but is said to be thoroughly unpractical. When de Wilde's gold precipitation method is used, the gold comes down very slowly, and the solution must be allowed to stand longer before and after filtering, it is said (the latter to ensure that precipitation is complete), than when the author's process is used.

6. *Use of Electricity for Precipitating Gold from Cyanide Solutions.*—The difficulties to be encountered here are the high resistance and low concentration of the solution, and the gradual change in its composition. Large electrodes must be used to reduce the resistance; but even then gold is sometimes deposited on the anode as well as on the cathode, and is partly contained in a deposit which forms in the solution. The cause of this is that the salt, $KAuCy_2$, breaks up under electrolysis into K, which is deposited at the cathode, and the ion $AuCy_2$ which tends to separate at the anode, the deposition of gold on the cathode being due to the secondary chemical action of the potassium upon the solution. High current-density and long treatment are therefore required to separate all the gold. By acidifying the bath with sulphuric acid, the recovery of the gold is practically complete (on a small scale) in 12—24 hours, with either soluble or insoluble anodes; but the cyanide is lost unless special means be used to recover it. In the absence of such technical skill as was shown by Siemens and Halske, failure, it is believed, is almost certain to result from an attempt to introduce a new electrolytic recovery process.

7. *Precipitation of Gold from Cyanide Solutions by Means of Zinc.*—The difficulties in this precipitation are well known; but sufficient attention has not been paid to the incompleteness of the precipitation of gold by zinc. The usually accepted equation—

$$2AuKCy_2 + Zn = K_2ZnCy_4 + 2Au$$

is apparently incorrect. It is found that burnished zinc gives no gold deposit, whilst the same zinc, as rough turnings from the lathe, works satisfactorily; and that the reaction proceeds best in the presence of a little free cyanide. But, at best, precipitation is irregular and incomplete. The author considers it probable that the double cyanide tends to dissociate in part into the ions K and $AuCy_2$, and that these then react with zinc and water as follows:—

$$2KAuCy_2 + 2Zn + 2H_2O = 2ZnCy_2 + 2KHO + 2H + 2Au.$$

The rough surface of the zinc turnings is here supposed to facilitate the escape of the hydrogen, and the presence of free cyanide to cause the solution of an insoluble film of zinc cyanide, which, otherwise, would prevent further action. When once started, a further reaction between the zinc cyanide and caustic potash would set in, so that the complete reaction would be thus represented:—

$$4KAuCy_2 + 4Zn + 2H_2O = 2ZnCy_2 + ZnCy_2.2KCy + ZnK_2O_2 + 4H + 4Au.$$

Hydrogen is undoubtedly produced, but it may partly result from the action of the gold-zinc couple upon the double cyanide of potassium and zinc. It is probable also that KCy is dissociated, forming KOH and HCy, and that methylamine is produced by the action of nascent hydrogen upon the latter. It may be noted that phenolphthalein gives no coloration with either pure potassium aurocyanide or

with water containing zinc turnings; but on mixing the two liquids, the usual deep rose colour is seen in the neighbourhood of the zinc. This may be due to caustic potash, methylamine, or possibly to zinc-potassium cyanide. The enormous consumption of zinc in the process is caused by the action of potash, potassium cyanide, and oxygenated water upon this metal. In practice, it is impossible to remove all gold from a cyanide solution within (say) 24 hours, unless there be 0·1 to 0·2 per cent. of free cyanide present.

8. *Recovery of Potassium Cyanide from Strong Solutions by Zinc Sulphate.*—When strong solutions of cyanide are used (1 per cent.), the cyanide should be recovered before precipitating the gold by the cuprous method. This may be done by Hunt's process (patented in 1895). Alkaline sulphides are first removed from the cyanide liquor by means of lead sulphate; excess of zinc sulphate is then added, and the resulting precipitate of zinc cyanide is separated by decantation or filtration, which is easily effected if the zinc sulphate be in excess, but not otherwise. The zinc cyanide is then decomposed by sulphuric acid, and the hydrocyanic acid is recovered in caustic potash as already described. Even when 0·1 per cent. of gold is present in the liquor, only a little separates with the zinc cyanide, and this passes into the zinc sulphate solution (as $HAuCy_2$), from which it is recovered by precipitation with sulphurous acid and sulphate of copper, or with cuprous chloride direct. This process is altogether superior to that of de Wilde, but gives trouble when the liquors contain potassium ferrocyanide, which yields zinc ferrocyanide, that settles and filters only with great difficulty, having the consistency of lard. When this substance is present, simple acidification of the original solution, with direct absorption of the HCy, would give a better result than the preliminary zinc sulphate precipitation.

9. *Precipitation of Aurous Cyanide by means of Zinc Chloride.*—This is the basis of Rinder's patent (1895). The author found that zinc chloride formed by solution of zinc in hydrochloric acid produced only a trace of aurous cyanide precipitate in aurocyanide liquors; but that when prepared by treating an excess of zinc oxide with hydrochloric acid, so that an oxychloride was formed, 99·4 per cent. of the gold present was precipitated. The process is, however, inferior to the cuprous method, because the gold cyanide precipitate is soluble in excess of either acid or alkali. Before adding the zinc chloride to the liquor, the silver in the latter must be removed as sulphide by the addition of iron sulphide.

III.—The Treatment of Ores.

An extraction efficiency of 60 to 70 per cent. may be sufficiently good for the treatment of low-grade tailings in South Africa, but would not be satisfactory in the case of ores assaying 100 dols. per ton, such as those to which the author has directed his attention. By studying each ore carefully the author has been able to bring up the efficiency of the cyanide process as follows:—From 70 per cent. to 99 per cent. in the case of Idaho-mine concentrated sulphurets assaying 120 dols. per ton; and from 75 per cent. to practically 100 per cent. with Alaska Treadwell concentrates assaying 50 dols.; whilst Bald Eagle (Alaska) concentrates assaying 280 dols. yielded 97 per cent. On the other hand, Bay State concentrates (California) assaying 160 dols. have at present given only a 90 per cent. extraction efficiency. There is every probability that in time equally good results may be obtained on a large scale. The cyanide process may not displace, but it should supplement, existing methods, and has a promising field in the treatment of low-grade ores.—W. G. M.

Alloys containing Zinc, On the Freezing-Point Curves of. C. T. Heycock and F. H. Neville. Proc. Chem. Soc. 1897, [176], 60—62.

The paper is divided into two sections, the first dealing with cases where the freezing point of zinc is depressed by the addition of another metal, the second with the cases where it is raised by such an addition.

In section I., complete binary alloys of the following pairs of metals, zinc-cadmium, zinc-aluminium, zinc-tin, and zinc-bismuth, are given. In the first three cases the metals appear to be miscible with each other in all proportions, but for zinc-bismuth the freezing point curve shows the horizontal line of identical freezing points characteristic of the state when the alloy has separated into two conjugate liquids.

Taking the freezing point of zinc as 419°, the authors find for the temperatures of freezing and the composition of the eutectic mixtures the following:—

Alloy.	F.P.	Atomic Percentage of Zinc.
Zn—Cd	264·5	74·0
Zn—Al	381·0	11·0
Zn—Sn	198·0	84·0
Zn—Bi	254·5	91·8

They do not consider that there is any indication of these metals combining chemically to form definite compounds when they are melted together.

Dilute solutions of the following metals in zinc were also examined:—lead, thallium, antimony, magnesium.

The authors find that, with the exception of aluminium and cadmium, all the above-mentioned metals, when added in small quantities to molten zinc, cause the same atomic fall; that is, an alloy containing 1 atomic weight in solution in 99 atomic weights of zinc has a freezing point lower by 5·1° than that of pure zinc. Cadmium causes a somewhat smaller and aluminium a decidedly smaller depression. The atomic depression of 5·1°, when used with Van 't Hoff's equation for the latent heat, gives 28·3 calories for the latent heat of fusion of zinc, instead of Person's value of 28·13.

In section II., dilute solutions of copper and of gold in zinc are considered, and also a complete freezing point curve for all alloys of zinc and silver.

When small quantities of any one of these three metals are added to molten zinc the effect is the same: the freezing point is raised, and the whole mass of metal appears to solidify at a temperature above the freezing point of pure zinc. The rise in the freezing point, moreover, is proportional to the amount of the second metal present. But when as much as 2 atomic per cents. of silver or of copper or 3·3 of gold have been added, the phenomenon alters, apparently abruptly. There are now two freezing points, a higher one which is very fugitive, and which is followed by the precipitation of much solid, and a lower one which is very constant, and which is independent of the amount of the added metal so long as the above-mentioned minimum is present. In the zinc-silver curve singularities are also found near 70 and 60 atomic per cents. of zinc, indicating the existence of more or less stable compounds, but the formula of these compounds is uncertain. Near 37·5 atomic per cents. of zinc there is another well-marked angle and a series of eutectic second freezing points, the phenomenon being possibly due to the separations of the alloy into conjugate liquids.

The authors also describe briefly some of the physical properties of the zinc-silver alloys, which appear to change in character at the angles of the curve.

The composition of the silver-zinc alloys at each freezing point was determined by extracting a portion in the liquid state, and estimating the percentage of silver by a volumetric analysis.

Gold or Auriferous Mineral, Method for Estimation or Extraction of. E. Serrant. Comptes Rend. 1897, **124**, [9], 480.

The process consists in adding to the crushed mineral a mixture of sodium chloride, sodium nitrate, and sulphuric acid, in proportions approximately calculated according to the richness of the mineral. When the reaction is complete, water is added to dissolve the chloride of gold, and the gold is precipitated from this solution by ferrous sulphate, oxalic acid, or sulphuretted hydrogen.—C. A. M.

Melted Copper and Copper Alloys, Removal of Oxide from, by Phosphorus. S. Kern. Chem. News, 1897, **75**, 97.

The phosphorus used for removing oxide from melted copper and copper alloys is first submitted to the following treatment:—The phosphorus is cut into pieces weighing about 3 oz. each, and placed in a concentrated solution of copper sulphate, where they are left until they are covered with precipitated copper. The phosphorus is thus rendered easy and safe to handle, and also, when thrown into the pots, does not take fire at once, but safely reaches the molten metal.

About two pieces are usually sufficient for 1 cwt. of metal in the pot, the phosphorus playing the part ferromanganese does in steel.—A. S.

Aluminium and its Alloys, Analysis of. F. Jean. Rev. Chem. Ind. **8**, 5—8.

See under XXIII., *page* 359.

Chromium, Determination of, in Ferrochrome and Chromium Steel. Spüller and Brenner. Chem. Zeit. 1897, **21**, 3.

See under XXIII., *page* 360.

Copper; Analysis, Quantitative, of Commercial. E. Murman. Monatsh. f. Chem. 1896, **17**, 697.

See under XXIII., *page* 359.

PATENTS.

Gold, Silver, and other Metals, the Extraction of, from Ores and the like; Impts. in. J. B. Torres, London. Eng. Pat. 3086, Feb. 11, 1896.

The ore, crushed to pass a 20-hole per linear inch sieve, is placed in a tank with perforated walls covered with a filtering medium, and set within a larger tank. It is here subjected to the action of an electrolysed solution of sodium fluoride and sodium chloride, and, subsequently, to further action of the hydrofluoric acid solution, to which a mixture of potassium cyanide and potassium bromide has been added. Carbon or other anodes stand in the ore compartment, and lead or other cathode plates receive the electrolytic deposit in the outer cell.—W. G. M.

Gold Ore, Amalgamating and Extracting Gold or other suitable Metals from, when Dry-crushed; New or Improved Process for. E. L. Oppermann, London. Eng. Pat. 4165, Feb. 25, 1896.

The crushed ore falls from a hopper into a vertical steam-jacketed chamber, in which is a perforated spiral pipe. A mixture of mercury vapour and steam is projected through the perforations, and the ore and condensed vapours fall over a series of sloping zig-zag plates through a vertical amalgamating chamber. The lowest sloping shelf is provided with a gutter to retain mercury, and the tailings passing from this plate come in contact with a blast of cold air, which serves to condense any mercury vapour still remaining. Steam and mercury vapour may also be introduced through subsidiary pipes in the amalgamating chamber if necessary.—W. G. M.

Aluminium, or Metals alloyed with Aluminium, Soldering of. F. C. Barnes and F. Campbell, Greenwich. Eng. Pat. 4667, March 2, 1896.

The basis of an aluminium solder must be zinc. The best results are obtained with an alloy of 95 per cent. of Zn and 5 per cent. of Al, or, for a solder with a better colour, 80 per cent. of Zn, 5 per cent. of Al, and 15 per cent. of Ag. Tin must be absent, or the joints will be unsound and will lack permanence. The surfaces to be joined are filed or scraped, and are then heated "to the right temperature," when the solder is applied without flux.—W. G. M.

Alloys, Impts. in. [*Copper*] J. C. Bull, Erith. Eng. Pat. 4899, March 4, 1896.

A tin amalgam, containing from 2 to 4 per cent. of mercury, is added to molten electrotype copper, to the extent of 2 to $1\frac{1}{2}$ per cent. Less pure copper may be used, but then phosphide of copper must be added before the amalgam, taking care that no appreciable amount of phosphorus be left in the metal. Such an alloy is said to roll and work well at a red heat, and to be sufficiently hard for ordinary purposes when cold.—W. G. M.

Sewage and other Waste Liquids, The Utilisation of By- and Waste Products [Flue Dusts] from Iron and Steel Industries for the Manufacture of Improved Precipitants for the Purification of. W. E. Adeney, and W. K Parry, both of Dublin. Eng. Pat. 5955, March 17, 1896

See under XVIII. B., *page* 349.

Aluminium Alloys, Manufacture of; Impts. in. W. Pearson, Birmingham, and J. A. Bott, Colwyn Bay. Eng. Pat. 6033, March 18, 1896.

To alloy aluminium with 0·1 to 5 per cent. of cobalt, the former metal is melted in a closed nickel crucible, and is then poured into a crucible containing the required quantity of cobalt in a state of fusion.—W. G. M.

Metals, Volatile; Apparatus for Separating from their Alloys. J. Imray, London. From M. A. J. Roux, Paris. Eng. Pat. 6572, March 25, 1896.

A horizontal distillation-chamber is supplied with charcoal and heated to the temperature necessary to volatilise the metal required; at one end is a smaller and separate melting chamber connected with it by a U-shaped channel that serves both as a liquid seal and as a means of conveying the melted alloy or amalgam into the larger chamber. At the opposite end is a second U-shaped seal for the withdrawal of the residual alloy, and a flue leading to a condensing chamber for the reception of the volatilised metal.

—W. G. M.

Chemically Pure Gold from Fine Gold and Alloys rich in Gold, An Improved Process for Obtaining. [*Hot Gold Chloride Bath.*] W. P. Thompson, of the Agency for Foreign Patent Solicitors, London. From Dr. E. Wohlwill, Hamburg, Germany. Eng. Pat. 7783, April 13, 1896.

—G. H. R.

Ores, Refractory; Improved Process for Treating. E. Govett, Melbourne. Eng. Pat. 12,106, June 3, 1896.

The crushed ore, dried if necessary, is mixed with from 1 to 4 per cent. of charcoal, and is then passed into a bath of molten lead. From 45 to 70 per cent. of the silver present is retained by the lead, which is then withdrawn and treated accordingly. The residue, which contains 1 to 10 per cent. of the lead used, is concentrated to remove zinc and gangue (if present), and the concentrates are then smelted.—W. G. M.

Zinc and Alkalis, Impts. in the Electrolytic Production of. O. Dieffenbach, Darmstadt, Germany. Eng. Pat. 25,804, Nov. 16, 1896.

The electrolytic vessel is divided into three parts by two diaphragms, the anode and cathode compartments containing respectively sodium sulphate and an alkaline solution of zinc, while the centre compartment contains sodium sulphate, or a solution of alkaline chlorides or dilute alkaline lye. In the first case, on the passage of the current, zinc is deposited in the cathode compartment, and the strength of the solution in zinc may be maintained by heating it together with zinc oxide, zinc hydroxide, or even metallic zinc. Simultaneously with the separation of zinc, caustic soda is formed, which passes into the centre compartment instead of being neutralised by the free acid formed in the anode compartment, as is the case when only one diaphragm is employed. The anode solution is kept constantly circulating and the acid is neutralised with lime, &c., as soon as it is formed. The solution in the centre compartment is regularly drawn off, and the caustic soda there formed separated by evaporation from the sulphate of soda, which crystallises out.—G. H. R.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(A.)—ELECTRO-CHEMISTRY.

Manganese Compounds, Influence of, on Lead Accumulators. G. v. Knorre. Zeits. f. Elektrochemie, **3**, 362—364.

In the literature of the subject, especially in patent specifications, it has frequently been proposed to replace part of the lead peroxide in the positive plates by manganese peroxide. Thus, Heinze proposes to form the electrodes by the electrolysis of organic lead compounds in presence of manganese salts, stating that this increases the E.M.F. and prevents buckling. Fournier and Epstein specify an addition of manganese peroxide in forming the plates. The author shows by the following experiments that when manganese compounds are present in a lead accumulator (either as peroxide on the positive plates or dissolved in the electrolyte) they act as oxygen carriers from the positive to the negative plate; thus they tend to discharge the accumulator and diminish its capacity. (1.) A freshly-charged positive plate is placed in dilute sulphuric acid containing a little dissolved manganese sulphate. The characteristic colour of permanganic acid is exhibited in a few minutes, and the liquid shows plainly the absorption-spectrum of this substance. The same reaction takes place slowly (12 to 24 hours) when the acid solution of manganese sulphate is simply shaken up with lead peroxide; it is much more rapid when the latter is in the form of a freshly-charged plate. (2.) A freshly-charged positive plate containing some manganese peroxide is placed in dilute sulphuric acid: permanganic acid is again formed, and can be recognised by its colour and absorption-spectrum. (3.) Potassium permanganate is added to dilute sulphuric acid until the latter is distinctly coloured red. When a freshly-formed negative plate of spongy lead is hung in the solution the colour gradually disappears. The spongy lead reduces the permanganic acid. A similar experiment can be performed with finely-divided crystalline lead, prepared by electrolysis from lead acetate. As the permanganic acid is reduced, manganese sulphate is formed, and at the same time an equivalent quantity of lead oxide or sulphate. (4.) From the above experiments it follows that when manganese peroxide is present on a positive plate (or manganese sulphate in solution) it is converted by the lead peroxide into permanganic acid; the latter is then reduced by the spongy lead plate, on which it sets free oxygen. Thus manganese compounds play the part of oxygen carriers in lead accumulators and diminish their capacity. If a charged accumulator containing such compounds be left to itself, it loses its charge with comparative rapidity; whereas a similar cell containing no manganese in the electrodes or electrolyte will, under similar circumstances, retain a considerable proportion of its charge.

—D. E. J.

Secondary Battery, A New. D. Tommasi. Bull. Soc. Chim. 1897, **17**, 211—213.

The author describes a form of secondary battery which differs from those ordinarily in use, inasmuch as the electrodes consist of lead grids, embedded in spongy lead or peroxide, the whole being enclosed in envelopes or sheaths of pure celluloid perforated with a large number of small holes. The cement used in the manufacture of the sheaths is obtained by macerating scraps of celluloid for 48 hours at 40° in acetone or glacial acetic acid mixed with alcohol. Cells in which these electrodes are employed, contain, for a given weight, a very large proportion of active material, *viz.*, 70—75 per cent., and consequently have a very large capacity. The current used in charging the cells may amount to 3 or 4 ampères per kilo. of electrodes, whilst on discharging, 8 or even 15 ampères per kilo. may safely be taken. The following constants are given:—

Initial electro-motive force	2·4 volts.
Mean " "	2·0 "
Capacity per kilo. of electrodes	22—25 ampère-hours.
Output in ampère-hours	95 per cent.
" " watt-hours	80 " "

The author recommends a discharge varying from 1 to 3 ampères for cells having a capacity of 22 ampère-hours.

—J. S.

Calcium Carbide, The Cost of Production of. D. Korda. Rev. de Chim. Ind. 1896, **7**, [83], 341.

See under II., *page* 318.

PATENTS.

Electrolytic Processes and Apparatus [Spiral Electrodes], Improved. A. E. Peyrusson, Limoges, France. Eng. Pat. 5616, March 12, 1896.

Helical electrodes are claimed, of which several examples are shown, whereby a prolonged circuit is imparted to the electrolyte in circulation through the coils of the helix whilst undergoing electrolysis. "The helical form presents the advantage that the electrolytes must follow the spiral course, which, according to the pitch of the helix, may be very many times greater than the height of the cell."

—J. C. R.

Electrolytic Apparatus ["Venetian-Blind" Electrodes], Impts. in. A. Le Royer, A. E. Bonna, and P. van Berchem, Geneva, Switzerland. Eng. Pat. 13,690, June 20, 1896.

To dispense with diaphragms for the separation of gases, vertical electrodes are constructed like Venetian blinds, the superposed inclined slats ("blades") of which, have the upper surface of their lower edge covered with a coating of suitable insulating material, in order to prevent the gas bubbles, formed during the electrolytic action, from escaping directly vertically from the said lower edges in the direction of the opposite electrode. The blades of one of the electrodes are preferably inclined in an opposite sense to those of the other electrode, so that the gas-bubbles formed on the surface of the several blades of one electrode are guided by the blades in one and the same direction.—J. C. R.

Secondary Batteries, Impts. in. H. H. Lake, London. From J. Julien, Brussels, Belgium. Eng. Pat. 7255, April 2, 1896.

The claims are: 1st, for "a secondary battery characterised by the arrangement of its electrodes, of the two names, whether of the same nature or different, so that those of the same name dip into a different liquid from those of the opposite name, thus forming a secondary battery with two liquids, for the purpose of augmenting the work at the negative pole, and hence the electro-motive force of the element."

2nd. "For the secondary battery of the first claim, a constructional form, *e.g.*, characterised by the constitution of the negative pole, as a strip of lead, surrounded by peroxide of lead, dipping into dilute sulphuric acid, and of the negative pole as a strip of zinc dipping into an aqueous solution of caustic soda, the two liquids being separated by a porous diaphragm or vessel.—J. C. R.

Secondary Batteries, Impts. in, and Process of Forming same. G. W. Harris and R. J. Holland, New York, U.S.A. Eng. Pat. 251, Jan. 5, 1897.

Plates of lead are grooved on both sides to receive the active material. They "may be made either by casting, rolling, or sawing out, to form the grooves between the ribs." The plate is borderless except at its top, where it is provided with a lug. In "forming" the plates, they may be first immersed in a bath "composed of dilute sulphuric acid (which is practically a non-solvent of lead) and an acid (preferably acetic or nitric) which is a solvent of lead." They are subsequently treated electrolytically in a bath of sulphuric acid and a metallic sulphate, magnesium sulphate, and "any one of a series of suitable acids that are ready solvents of lead—for example, acetic acid—with, lastly, a corresponding salt for the lead solvent, *viz.*, magnesium acetate." Where aluminium sulphate is employed, "the acid solvent of lead becomes aluminium acetate." To nitric acid, as the acid solvent of lead, is added a nitrate, and so on. "Selenic acid might be used in place of

sulphuric; the selenates would then be used instead of the sulphates." The formation is completed in a "bath composed of dilute sulphuric acid and a proportionately small quantity of an acid sulphate, as that of sodium or potassium." "Such of the positives as are intended to become negatives are reduced in this same electrolyte or bath, by being connected with the negative terminal of the charging current."—J. C. R.

Electric Batteries ["*Dry*"], *Impts. in.* P. Schmidt, Berlin, Germany. Eng. Pat. 10,095, May 12, 1896.

The improvements relate to the construction of a "dry" cell. A carbon cylinder closed at top is placed in the annular space between an outer zinc cylinder and (capping) an inner zinc cylinder (previously filled with a solution of sal ammoniac). The space between the carbon and outer cylinder is filled with an exciting paste, consisting, "in addition to finely-divided quebracho wood, of extract of tannin infused in boiling water, and purified by filtering together, with the addition of a small quantity of a solution ot chloride of zinc, the wood particles acting to prevent polarisation." By turning over the cell and placing it upright, the solution of sal ammoniac is transferred from the inner zinc cylinder to the annular space between it and the carbon cylinder, "thus reviving the cell by the gradual sinking of the liquid in the said annular space, because it passes through the porous carbon, and keeps the paste moist." The zinc cylinders are in electrical connection at the bottom. The carbon cylinder rests on a layer of paraffin run into the annular space between them.—J. C. R.

Oxygen and Hydrogen by Electrolysis [*Perforated Metallic Diaphragm*]. *New and Useful Impts. in the Apparatus for the Production of.* P. Garuti and R. Pompili, Naples, Italy. Eng. Pat. 23,663, Oct. 24, 1896.

These improvements on Eng. Pat. 16,588 of 1892 (this Journal, 1893, 768), relate to means for separating oxygen and hydrogen in electrolysis by metallic diaphragms placed between the electrodes, the said diaphragms being perforated in their lower part, up to a little height, by small holes as near as possible to each other.—J. C. R.

Electric Insulating Compound. L. Honig, St. Louis, Missouri, U.S.A. Eng. Pat. 23,868, Oct. 27, 1896.

100 parts contain—alcohol, 30; gum shellac, 25; wheat flour, 20; powdered asbestos, 20; glue, 2.; varnish, 2; glycerin, 1.

The shellac is dissolved in the alcohol, then the asbestos is added, then the glycerin and glue, and lastly the varnish and wheat flour. The compound is applied with a brush. The flour is added merely to give body, and the proportion is varied as required. The varnish may be omitted, or varied in quantity, as it is only used as a finish. Glycerin makes the mixture flow smoothly, and the glue tends to make the material tough.—L. A.

Storage Batteries [*Preparation of Active Material with Sulphates*], *Impts. in the Manufacture and Production of Elements or Plates for Electrical Storage Batteries.* C. Brault, Paris, France. Eng. Pat. 29,907, Dec. 29, 1896.

The claims are for manufacturing electrodes "by compressing between the parts of conductive grids, active material or material to be rendered active, prepared from a mixture of oxide of lead or litharge and sulphate of an alkali or of an alkaline earth, treated with a solution of ammonia, or of ammoniacal salt (with or without bisulphate of mercury according to the denomination of the plate . . .)." For the negative plates 1 to 2 per cent. of bisulphate of mercury is added. Before forming the compressed material, it is steeped for a few hours in a weak solution of sulphuric acid to which is added 2 to 3 per cent. of the salt employed in the preparation of the plates. To provide for expansion in the course of "formation," the tablets of active material, or material to be rendered active, are perforated.—J. C. R.

Ozone [*Electrolytic-Cooling Gas Arrangements*], *Impts in and Apparatus for Producing.* J. Y. Johnson, London. From the Electric Rectifying and Refining Co., Camden, New Jersey, U.S.A. Eng. Pat. 29,966, Dec. 29, 1896.

The process consists essentially in subjecting oxygen gas to an electric current in the form of a brush discharge, the oxygen gas being, by preference, at a low temperature while it is being subjected to the action of the current. Apparatus for carrying out the process is also fully described and shown. The oxygen gas may be cooled by passing through a cooling medium, or compressed, then cooled and permitted to expand either directly into the ozone generator or into a supply chamber therefor, thereby cooling it to the desired degree. "As a rule, the lower the temperature, the better the result." It is preferred "to use oxygen gas of as pure a character as can be manufactured on a commercial scale . . . ." The process and apparatus, it is said, are particularly adapted for use in purifying and decolorising and deodorising alcoholic and other liquids, and for general application where ozone gas is required.—J. C. R.

(*B.*)—ELECTRO-METALLURGY.

Electrical Resistivity of Electrolytic Bismuth at Low Temperatures, and in Magnetic Fields. J. Dewar and J. A. Fleming. Proc. Roy. Soc. **60**, (1897), 425—432.

The authors have measured the electrical resistance of a wire of pure bismuth placed transversely to the field of an electro-magnet and at the same time subjected to low temperatures. The electro-magnet was a powerful one provided with adjustable pole-pieces: it was excited by a constant current and the intensity of the field was varied by varying the interpolar distance. The bismuth (electrolytically pure) was in the form of a wire about 5 mm. in diameter and 3—4 cm. long, looped in a single turn. By wrapping it in cotton-wool and pouring on this liquid air, the temperature was reduced to 185° C.: by enclosing it in a small glass vacuum vessel a further reduction of temperature to 203° C. was attained. The following results were obtained:—

If the transverse field be zero, then cooling the bismuth always reduces its resistance. If the bismuth be then transversely magnetised, the resistance is increased, and for every temperature below the normal one (about 20° C.) there is some particular strength of transverse field which just annuls the effect of the cooling, and brings the resistance of the bismuth back again to the same value it had when not cooled, and not in any magnetic field. Hence the curves showing the resistance at any temperature lower than the normal one (20° C.) as a function of the transverse field, cross the curve showing the resistance as a function of the field when taken at a normal temperature. These crossing points are, however, not identical for different resistance-temperature fields. The lower the temperature the less is the strength of field which will bring the bismuth back to its original resistance when not cooled and not in the field.

In a transverse magnetising field having a strength of 22,000 C.G.S. units, the resistance of bismuth taken in liquid air is 150 times greater than its resistance in a zero field at the same temperature. The lower the temperature the greater is the multiplying power. In this respect bismuth is a remarkable exception to other metals. Thus in the case of zinc, iron, and nickel, the resistance is affected by magnetisation to a small degree; but the authors do not find any effect which is sensibly greater at low than at ordinary temperatures.—D. E. J.

Aluminium, Recent Determinations of Electrical Conductivity of. J. W. Richards and J. A. Thomson. J. Franklin Inst. 1897, **143**, 195—199.

The authors attribute the variable figures which have been given for the electrical conductivity of aluminium to:—1st. The use of impure metal, as only in recent years has 99 per cent. aluminium been obtainable. 2nd. The reference

of the conductivity to copper or silver standards, without the indications of chemical or physical condition. 3rd. The lack of an accurate standard of absolute resistance. 4th. Imperfect methods of measurement.

They have therefore carried out a series of experiments with the object of more accurately determining the conductivity by the help of more efficient and better appliances as indicated above.

The results are expressed in absolute measurement. A certified standard resistance coil of 1 "international" ohm, with a possible error of 0·02 per cent., was used.

The Carey Foster method of comparison was adopted. The metal in the form of wire (50 ft. lengths, the diameter measured by micrometer, checked by weighing and specific gravity), was wound on wooden bobbins, immersed in an oil bath, temperature of which was carefully noted. The standard coil was immersed in water, and temperature of room kept constant. The bridge wire was carefully calibrated, and a reflecting galvanometer employed.

Two separate wires were used of the first specimen, and the results only differed by one hundredth per cent. from each other.

Analysis.						Resistance at 0° C. of a Wire 1 metre long by 1 mm. Diameter in Ohms.	Specific Resistance at 0° C., *i.e.*, Resistance of 1 c.c. in Absolute (C.G.S.) Units of Resistance.	
Aluminium.	Iron.	Copper.	Silicon.	Sodium.	Zinc.	Hard.	Hard.	Annealed.
99·66	0·10	0·00	0·16	0·008	..	0·031245	2453·7	2432·2
99·58	0·25	0·00	0·16	0·052	..	0·03290	2584·0	2535·0
98·77	0·20	0·57	0·45	0·012	..	0·03627	2848·0	..
97·16	0·25	2·26	0·30	0·032	..	0·03590	2819·6	..
94·39	0·25	3·07	0·24	0 052	1·50	0·03583	3011·4	2984·7

The authors show, after comparing their figures with those of previous experimenters, that when the impurities are taken into account, the results are fairly in agreement.

McGee determined in 1890 the conductivity of aluminium of 98·52 per cent. purity to be 54·8 per cent. that of copper, when unannealed.

The authors were led to the conclusion that the conductivity of hard-drawn commercial aluminium was strongly affected by impurities, being approximately:—

	(Copper = 100)
98·5 per cent. of aluminium..........	55·0
99·0 „ „	59·0
99·5 „ „	61·0
99·75 „ „	63·0 to 64·0
100·00 „ „ .probably	66·0 to 67·0

Annealed wire was found to have a conductivity very nearly 1 per cent. greater than unannealed.—O. H.

Platinum in Russia, Production of. Handelsmuseum, through Imp. Inst. J., April 1897, 132.

The Russian Ministry of Finance has recently issued a report on the production of platinum. Russia is the most important country in the world as regards platinum, producing forty times as much as all the other countries together. In 1880 the amount was 2,946 kilos., and in 1895, 4,413 kilos., whilst in 1894 the production was 5,208 kilos. The rare metal is found entirely in Southern Ural. Its manufacture is not carried out in Russia, but in Germany, where it is imported in the ore; and what Russia requires in the shape of prepared platinum has to be purchased in Germany. The price of platinum has been very high during recent years; at present, raw platinum costs 900 marks per kilo. The still rarer metal, iridium, is also found with platinum, but in very small quantities. In 1895 the amount was 4·1 kilos., and in 1894 but very little more.

PATENTS.

Reducing Metals from their Ores, and for Obtaining Carbides, Acetylene, Cyanogen, Cyanides, Nitrides, and for Fixing Nitrogen; Impts. in Processes and Apparatus for. [*Electrical Blast Furnace.*] H. Maxim, New York, U.S.A. Eng. Pat. 1905, Jan. 27, 1896.

The apparatus consists of a primary heater, an electrical superheater, and a blast furnace, all arranged under one gastight casing. The primary heater consists of a furnace in which either solid or gaseous fuel may be burnt, and which heats a long series of pipes arranged transversely across it, through which the gaseous fluid on its way to the blast furnace is passed. The furnace is adapted to work with a blast under pressure, and fuel is supplied to it by means of a screw feed provided with valves. The primary heating pipe communicates with two electric furnaces placed side by side, and connected by a pipe at their upper end, and the gas, after passing through them, leaves the second at the bottom, and enters the blast furnace, and thence passes through scrubbers into a gasometer, from whence it is passed back under pressure through the heating apparatus, or otherwise treated as desired. The linings of the gas passage and the electric furnace are composed of some material which will not be acted on by the gaseous fuel; for instance, it is carbon if carbonic acid is employed. In the first electric furnace which is filled with carbon blocks or other refractory material, the carbon electrodes are placed preferably at the top and bottom. In the second they are arranged spirally, and the furnace is filled with fireclay. When calcium carbide is to be manufactured, the blast furnace is fed with lime and coke by means of a screw feed, and the passage through it of the highly-heated carbon monoxide forms calcium carbide, which is drawn off at the bottom, or can be blown off by an atomiser in a flocculent form. The electric superheater may also be arranged horizontally and be rotary. Several modifications of the apparatus are given, and the applications of it to the manufacture of carbides, acetylene, fixation of nitrogen, production of cyanogen, cyanides, and illuminating gas are described and claimed.—G. H. R.

Electro-plating or Metallisation of Wood, An Improved Process for the. H. A. V. Wirth, Paris. Eng. Pat. 5263, March 9, 1896.

The wood is immersed in a bath of boiling wax which fills the pores. On cooling it is brushed over with plumbago, washed with alcohol, and plunged in the electrolytic bath.
—V. C.

Refining Metals [Copper, &c.] by Electrolysis, Impts. in Apparatus for. A. S. Elmore, Leeds. From J. O. S. Elmore, Kapurthala, India. Eng. Pat. 5673, March 13, 1896.

In this process the electrodes are arranged as follows:—On a rectangular frame of wood is laid a sheet of lead, or iron covered with lead if the sheet be large. On this is placed a plate of the crude copper or other metal to be refined, which is shorter than the lead plate, and is surrounded by a wooden frame thicker than itself. Several pads of rubber or other elastic insulating material are laid on the copper, and then another lead plate carrying a copper plate in a wooden frame as before, apertures being provided in the lead plate at the opposite end to that in which they were provided in the previous plate. These frames and sheets are piled over one another and clamped firmly together, the lowest and uppermost sheets being connected respectively to the positive and negative terminals of a

dynamo. As the apertures for the passage of the electrolyte are at opposite ends of adjacent plates, it is forced to flow in a zigzag course through the frame, and the metal dissolved on the crude anode is deposited on the lead cathode above it. The solution enters through a pipe at the top, and is discharged at the bottom into a rocking trough which breaks the electric circuit through the electrolyte, and discharges its contents into a tank, whence the liquid is pumped back with or without filtration, into the inlet pipe. The arrangement of the electrodes may be vertical instead of horizontal, and they may be bipolar, in which case the intermediate lead sheets are dispensed with.—G. H. R.

Metals, Impts. in the Electro-deposition of, particularly applicable to the Deposition on Wires. [*Rapid Circulation.*] J. C. Graham, London. Eng. Pat. 8410, April 21, 1896.

The current strength employed in electro-deposition on wires, strips, sheets, tubes, or cylinders may be greatly increased, and the rate of deposition accelerated by causing the electrolyte to circulate rapidly through tubes or troughs over the whole surface of the anode and cathode. The tube itself may form the anode, or it may contain pieces of metal which act as anodes, and the wire or matrix forming the cathode is led through it resting on suitable supports. One end of the tube is connected to a reservoir placed about 2 feet above it, and the other with a centrifugal pump which returns the electrolyte to the reservoir. Baffles placed in the tube maintain a uniform distribution of the electrolyte, and the current strength which can be used depends on its density and rate of flow.—G. H. R.

Gold and Silver from their Cyanide Solutions, Impts. in the Electric Precipitation of. [*Lead Anodes.*] A. James, Glasgow. Eng. Pat. 1535, Jan. 20, 1897.

Lead anodes are employed, as it has been found that the coating formed on them during electrolysis, protects them. The cathode may be lead, as in the Siemens and Halske process, and the gold and silver may be recovered in the same way. Or aluminium cathodes may be used, when the voltage required is about ½ volt higher than in the former case, and the precious metals are recovered by scraping.—G. H. R.

XII.—FATS, OILS, AND SOAP.

Oleic Acid, Industrial Transformation of, into Stearolactone and Monohydroxystearic Acid. David. Comptes Rend. 1897, **124**, [9], 466—468.

When sulphuric acid is mixed in molecular proportion with oleic acid cooled to zero, and water is added to the sulpho acids produced, with precautions to prevent much rise of temperature, two layers are formed, the lower of which contains 50 per cent. of the sulphuric acid taken, whilst the upper is an oily liquid consisting of 50 per cent. of a sulpho acid not decomposed by water and 50 per cent. of one or more of the three isomerides of hydroxystearic acid.

The author considers that the α- and γ-isomerides are present, and his experiments have led him to the conclusion that the [OH] group is transposable according to the temperature, so that the α-acid may be transformed into the γ-acid (or more strictly speaking its inner anhydride). To a mixture of oleic acid and sulphuric acid an equal volume of cold water was added and the whole left for an hour. The lower layer of acidulated water was removed and an equal volume of pure water added. This dissolved the oily layer, and the clear solution, when left for 12 hours at -4 to $-5°$ C., yielded a quantity of small white crystals of stearolactone—the inner anhydride of γ-hydroxystearic acid, the amount being about 18 to 20 per cent. of the weight of oleic acid taken. When the stearolactone was removed the clear liquid still contained hydroxystearic acid (30 per cent.). On boiling this liquid for about an hour and then leaving it over night, granular crystals were produced, which melted at 80° C. and solidified at 68° C. (Fremy's hydroxystearic acid). If, on the other hand, the clear liquid, instead of being boiled, was kept overnight at a temperature of -4 to -5 C., a fresh crop of crystals of stearolactone was formed, and the filtrate from these on being exposed to the cold for 24 hours yielded still more, and so on as long as any hydroxystearic acid remained in the liquid.

On an industrial scale the author has thus obtained 50 per cent. of solid products from a given weight of oleic acid, and he considers that by using sulphuric acid of 66° B., instead of a little over 65° B. (which was the strongest he could procure), the yield would be increased to 70 or even 80 per cent.—C. A. M.

Vegetable Tallow, Chinese. G. de Negri and G. Sburlati. Chem. Zeit. 1897, **21**, [1], 5—6.

Vegetable tallow is obtained from the fruit or seed of a variety of plants in many parts of the world: a more or less complete enumeration of which is given in the paper. By far the most important is the Chinese, from the so-called yaricou tree (Croton sebiferum, Linn.). The commercial article often contains an admixture of linseed oil. The seeds are husked and roughly broken, steamed till soft, beaten with wooden hammers, and the paste put into warm sieves, through which the melted tallow drops into pots. Or the seeds are bruised and boiled with water; the oil collects at the top and solidifies to a cake on cooling. The yield is from 20 to 30 per cent. In some places 30 or 40 per cent. is got by hot-pressing. Yet another method consists in steaming the seeds in a perforated inner vessel surrounded by an outer one. The "tallow" which envelops the seeds drops through the perforations into the outer vessel. From the remaining substance, by pressure, an oil called "ting-you" is got; this is used for lighting, and also in making varnishes.

Samples of commercial vegetable tallow are white or greenish, and all are destitute of smell and taste. They appear usually as egg-shaped masses of from 55 to 60 kilos., which are covered with rice-straw and wrapped in rush mats. Hannau, De Negri and Fabris, and more recently Hobein, have investigated the properties of vegetable tallow. Some of their figures are given in the following table, with a summary of the results obtained by the present authors from 10 samples. These 10 samples left a faint greasy mark on paper, and showed a slightly acid reaction. They all dissolved freely in warm absolute alcohol, but almost completely separated out again on cooling. They were easily soluble in ether, benzene, chloroform, and carbon bisulphide. Their specific gravity at 15° was from 0·9182 to 0·9217, and 10 grms. yielded from 8·2 grms. to 8·7 grms. of fatty acids, which were white or yellowish.

——	Lowest of 10 Samples.	Highest of 10 Samples.	Average of 10 Samples.	Hobein Commercial Product.	Hobein. Direct from Seeds.	Negri and Fabris. From Seeds by Ether
Melting point	36°·5	44°·1	40°·7	43°—45°	39°—42°	36°—39°
Freezing point	27°·2	31°·1	28°·8	35°	32°	26°—28°
Melting point of fatty acids	53°·0	56°·9	55°·3	51°	49°	40°—42°
Freezing " "	45°·2	47°·9	46°·7	41°	40°	36°—38°
Saponification number	198·5	202·2	200·2	203·6	203·3	..
" " of fatty acids	202·0	209·5	207·3	207·9	206·4	..
Iodine number	28·50	37·74	32·28	35·5	28·5	52·2—53
" " of fatty acids	30·31	39·50	35·10	38·1	29·6	..
Acid number	..	..	..	..	..	2·2

The difference between Hobein's figures and those of Negri and Fabris arises from the fact that the ether extracts not only the tallow but also some of the oil contained in the endosperm of the seeds. Hobein prepared some of this latter oil as a brownish-yellow fluid of sp. gr. 0·9498, saponification No. 203·8, iodine No. 145·6.

The differences among commercial vegetable tallows arise partly from their being mixtures of tallows from different plants, partly from the different processes of extraction, and partly from changes which they undergo with the lapse of time.—J. T. D.

Hyoscyamus Niger, Oil extracted from the Seeds of; Properties of. A. Mjoen. Arch. de Pharm. **234**, 286.

This fatty oil has a density of 0·939 at 15° C. It is very easily soluble in ether and chloroform. 1 part requires for its solution 56 parts of absolute alcohol, or 200 parts of 90 per cent. alcohol. When saponified, the oil yields glycerin, and a mixture of oleic and palmitic acids, together with a third acid, the exact nature of which is, as yet, undetermined. —H. T. P.

Melting Points, Note on a Method for Determining. E. H. Cook. Proc. Chem. Soc. 1897, [177], 74—76.

See under XXIII., *page* 357.

Fatty Acids [Stearic Acid, &c.], Determination of Unsaponified Fat in. F. and J. Jean. Rev. de Chim. Ind. 1896, **7**, [83], 338.

See under XXIII., *page* 363.

Colza Oil, New Colour Reaction of. Palas. Ann. de Chim. Analyt. **1**, 434; Bull. Assoc. Belge. des Chim. **10**, [9], 358.

See under XXIII., *page* 361.

Lards, Determination of Solid Fats in Compound. G. F. Tennille. J. Amer. Chem. Soc. 1897, **19**, 51.

See under XXIII., *page* 363.

PATENT.

Washing Material, an Improved, and Process for Making the Same. G. Rezek, Vienna, Austria. Eng. Pat. 123, Jan. 2, 1897.

125 kilos. of "ammonia soda" are mixed with 60 kilos. of ground resin and the powder is kneaded with 120 kilos. of water to the consistency of soft-soap. Glycerin, "benzine," fats, oils, perfumes, and colours may be added. This represents substantially the claim.—L. A.

XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

(*A*.)—PIGMENTS, PAINTS.

PATENT.

Basic Lead Carbonate, Improved Process of Producing and of Obtaining Certain Bye-Products. The Chemical and Electrolytic Syndicate, Ltd. O. J. Steinart and J. L. F. Vogel, London. Eng. Pat. 6655, March 26, 1896.

Pig lead is oxidised, and the oxide is dissolved in nitric acid. The solution of lead nitrate, about saturated in the cold, is treated with finely divided lead to remove silver and other foreign metals, is heated nearly to boiling and run into a similarly heated solution of sodium carbonate, containing sufficient excess of alkali to produce a basic lead carbonate precipitate free from nitrate, and the liquid containing the precipitate is thoroughly stirred and boiled for some time. The precipitate is then separated. The solution of sodium nitrate and excess of sodium carbonate is evaporated to dryness (sodium carbonate being partly separated by crystallisation if desired), and the dry residue is ground with enough ferric oxide to prevent fusion at a high temperature. The mixture is then furnaced in an inclined retort described in Eng. Pat. 7273, 1895, when nitric acid and caustic soda are obtained as final products. —L. A.

(*B*.)—RESINS, VARNISHES.

Acaroid (Xanthorhœa) Resin, Yellow and Red. K. Hildebrand. Arch. Pharm. 1896 [12]; Chem. Rev. Fett u. Harz Ind. **4**, [4], 53.

The author finds that the yellow resin contains free cinnamic and paracumaric acids, the latter being also present (to the extent of 80 per cent.) as an ester of xanthoresinotannol ($C_{43}H_{45}O_9 \cdot OH$). No methoxyl group could be detected in this alcohol, and the acetyl- and benzoyl-derivatives demonstrated the existence of only one hydroxyl-group. Styracin (cinnyl cinnamate) and the phenylpropyl ester of cinnamic acid, together with paroxybenzaldehyde and vanillin—the latter, however, not being identified with certainty—are present in the yellow resin.

The red resin is characterised by the absence of cinnamic acid, styracin, and the cinnamic acid ester. Paroxybenzaldehyde was found, but the bulk of the resin consists of an ester of paracumaric acid with erythroresinotannol ($C_{40}H_{39}O_9 . OH$). Benzoic acid is present in the combined state.—C. S.

Shellac, Behaviour of the Resin Acids of, in Separating Fatty and Resin Acids by Gladding's and Twitchell's Processes. F. Ulzer and R. Defris. Zeits. anal. Chem. **36**, [1], 24.

See under XXIII., *page* 364.

(*C*.)—INDIA-RUBBER, &c.

PATENT.

India-rubber, Impts. in the Manufacture of, and in Machinery and Apparatus therefor. W. J. Clapp and F. S. Dauncey, Newport, Mon. Eng. Pat. 6830, March 28, 1896.

In order to render india-rubber proof against the action of "heat, steam, and other vapours, fluids, and liquids," the raw rubber is dissolved in a suitable solvent and mixed with from 2 to 10 per cent., or any other suitable quantity, of "silicate, slag wool, silicate cotton, Bessemer basic or other suitable slag, mica, platinum, alum, borax, pitch, tar, petroleum, or like materials." The mass is then raised to a suitable temperature according to the purpose for which the manufactured goods are to be applied.—L. A.

XIV.—TANNING, LEATHER, GLUE, SIZE.

Fermentation Phenomena in Tan Liquors. F. Andreasch, Imp. Research Station, Vienna. Der Gerber. **22**, [526], 174.

The following table gives the more important results, oakbark and sumac being selected as examples.

The values are given in grms. per 100 c.c. of liquor. The small differences result from the inexactness of the method for estimation of alcohol, and in the lactic acid, because of its instability. In the column headed "Class," I. = Original tan liquor; II. = Original tan liquor with addition of mineral nutriment; III. = Sterile, uninoculated solutions. In class III. no change occurred in these liquids; hence it is unnecessary to represent the figures in the table. (See next page.)—J. T. W.

Tanning, The Chromate Process of; Its Advantages and Disadvantages. F. Jean. Rev. de Chim. Ind. 1896, **7**, [83], 343—345.

Since its introduction into America in 1892, the chrome tanning process (in which the hides are soaked in an acid solution of potassium bichromate, subsequently in an acid bath of sodium sulphite, the result being that chromium oxide is deposited on the fibre) has made great advance there, and to some extent has been introduced into Europe —notably into Germany. The process certainly merits consideration on account of its cheapness, the comparatively short time required for its completion, and because the leather produced by it possesses superior tenacity, great resisting power to moisture and heat, and is not liable to crack. For these reasons (the validity of which is generally

Oak Bark. Tannin, 9·88 per cent. ; non-tannin, 7·21 per cent.

Time, 30 Days. Temperature, 30° C. — Changes by Fermentation with

Material.					Yeast (cultivated from Tan Liquors).			Lactic Bacterium I. of Tan Liquors.		
Kind of Solution.	Class.	Concentration.	Amount before Fermentation. Tannin.	Amount before Fermentation. Non-Tannin.	Tannin.	Non-Tannin.	Chief Product, Alcohol.	Tannin.	Non-Tannin.	Chief Product, Lactic Acid.
Original tannin solution	I.	1	0·9000	0·656	0·9000	0·4259	0·1045	0·9000	0·6164	0·0343
		2	1·8000	1·312	1·8000	0·8528	0·2000	1·8000	1·2276	0·0700
	II.	1	0·9000	0·656	0·9000	0·4265	0·1085	0·9000	0·1713	0·4023
		2	1·8000	1·312	1·8000	0·8530	0·2081	1·8000	0·3960	0·7601
Non-tannin solution prepared with hide powder	I.	1	..	0·656	..	0·4244	0·1050	..	0·5898	0·0550
		2	..	1·312	..	0·8500	0·2123	..	0·4739	0·0900
	II.	1	..	0·656	..	0·4250	0·1058	..	0·1514	0·4088
		2	..	1·312	..	0·8520	0·1158	..	0·3696	0·7823
Pure tannin solution, with glucose added	I.	1	0·9000	0·4000	0·9000	0·0034	0·1985	0·9000	0·4000	..
		1a	1·8000	0·4000	1·7988	0·0038	0·1980	1·8000	0·3984	0·0015
		2	0·9000	0·8000	0·9000	0·0064	0·3971	0·9000	0·7988	..
		2a	1·8000	0·8000	1·8008	0·0070	0·3980	1·8000	0·8000	..
	II.	1	0·9000	0·4000	0·9000	0·0030	0·1990	0·9000	0·0100	0·3240
		1a	1·8000	0·4000	1·7980	0·0040	0·1980	1·8000	0·0111	0·3188
		2	0·9000	0·8000	0·9000	0·0071	0·4000	0·9000	0·0223	0·6450
		2a	1·8000	0·8000	1·8000	0·0078	0·3989	1·8000	0·0246	0·6353
Pure tannin solution, with non-tannin added	I.	1	0·9000	0·6450	0·9000	0·4230	0·1050	0·9000	0·5811	0·0530
		1b	1·8000	0·6450	1·8000	0·4240	0·1046	1·8000	0·5809	0·0541
		2	0·9000	1·2900	0·9000	0·8510	0·2070	0·9000	1·1810	0·0900
		2b	1·8000	1·2900	1·8009	0·8520	0·2089	1·8000	1·1798	0·0883
	II.	1	0·9000	0·6450	0·9000	0·4160	0·1025	0·9000	0·1444	0·4155
		1b	1·8000	0·6450	1·8000	0·4130	0·1050	1·8000	0·1456	0·4108
		2	0·9000	1·2900	0·9000	0·8244	0·2010	0·9000	0·3450	0·7844
		2b	1·8000	1·2900	1·8000	0·8315	0·2088	1·8000	0·3484	0·7821

Sumach. Tannin, 23·35 per cent. ; non-tannin, 19·81 per cent.

Kind of Solution.	Class.	Concentration.	Amount before Fermentation. Tannin.	Amount before Fermentation. Non-Tannin.	Yeast. Tannin.	Yeast. Non-Tannin.	Yeast. Chief Product, Alcohol.	Lactic Bacterium I. Tannin.	Lactic Bacterium I. Non-Tannin.	Lactic Bacterium I. Chief Product, Lactic Acid.
Original tannin solution	I.	1	0·9000	0·7630	0·9000	0·5730	0·0961	0·9000	0·0360	0·6032
		2	1·8000	1·5260	1·8000	1·1360	0·1842	1·8000	0·5620	0·8000
	II.	1	0·9000	0·7630	0·9000	0·5700	0·0983	0·9000	0·0321	0·6188
		2	1·8000	1·5260	1·8000	1·1299	0·2900	1·8000	0·5422	0·8100
Non-tannin solution prepared with hide powder	I.	1	..	0·7630	..	0·5715	0·0980	..	0·0340	0·6244
		2	..	1·5260	..	1·1289	0·1921	..	0·5549	0·8055
	II.	1	..	0·7630	..	0·5695	0·0997	..	0·0360	0·6166
		2	..	1·5260	..	1·1254	0·1944	..	0·5484	0·8114
Pure tannin solution with glucose added	I.	1	0·9000	0·4000	0·9000	0·0054	0·1965	0·9000	0·3981	..
		1a	1·8000	0·4000	1·8009	0·0040	0·1970	1·8000	0·3990	..
		2	0·9000	0·8000	0·9000	0·0070	0·3971	0·9000	0·8000	..
		2a	1·8000	0·8000	1·8000	0·0075	0·3984	1·8000	0·7980	0·0010
	II.	1	0·9000	0·4000	0·9000	0·0040	0·1980	0·9000	0·0032	0·3210
		1a	1·8000	0·4000	1·8000	0·8048	0·1980	1·8000	0·0044	0·3244
		2	0·9000	0·8000	0·9000	0·0061	0·3945	0·9000	0·0114	0·6751
		2a	1·8000	0·8000	1·8000	0·0070	0·3911	1·8000	0·0151	0·6644
Pure tannin solution, with non-tannin added	I.	1	0·9000	0·7500	0·9000	0·5645	0·0900	0·9000	0·0250	0·6006
		1b	1·8000	0·7500	1·8000	0·5681	0·0911	1·8000	0·0265	0·6011
		2	0·9000	1·5000	0·9000	1·1240	0·1867	0·9000	0·5188	0·8144
		2b	1·8000	1·5000	1·8000	1·1258	0·1806	1·8000	0·5277	0·8056
	II.	1	0·9000	0·7500	0·9000	0·5673	0·0920	0·9000	0·0086	0·6153
		1b	1·8000	0·7500	1·8000	0·5630	0·0920	1·8000	0·0145	0·6100
		2	0·9000	1·5000	0·9000	1·1285	0·1800	0·9000	0·5193	0·8121
		2b	1·8000	1·5000	1·8000	1·1242	0·1841	1·8000	0·5000	0·8255

admitted) it is very suitable for certain purposes, *i.e.*, for engine belts, straps, and the like. For general purposes, however, chrome leather must be pronounced less suitable than leather produced with bark; and it is particularly in the boot manufacturing industry that it requires to be most carefully tested. Chiefly on account of its imperviousness to moisture and air, chrome leather is not likely to be used extensively in the manufacture of boots. Besides, it is stated that, in respect to elegance of appearance and finish, chrome leather boots compare unfavourably with those made from bark-tanned leather; that in shaping and trimming, the cut edges are not smooth, but rough and fibrous; and that difficulties are met with in the stitching and chasing processes. It is noted that Cavalier in 1853 used a chromate bath with ferrous sulphate to reduce the chromic acid.—H. T. P.

Formalin, Experiments with. W. Eitner. Der Gerber. **23**, [541], 67.

SINCE the introduction of formalin or formaldehyde as a trade product it has been proposed to use it for tanning leather as a substitute for alum.

The author finds that, although it has a hardening effect on the skin fibre, in no case can leather be produced by its use alone. It may, however, be used with advantage in certain tannages to "fix the swelling" produced by the sour liquors; the hides may then be worked in sweet extract liquors without losing the characteristic appearance of the Bohemian pine sole-leather. A solution of 1 kilo. of commercial formalin (40 per cent.) in 1,000 litres of water is used, and the hides are soaked in it for 24 hours after coming out of the sour liquors; the tanning may then be finished in the usual way or in extract liquors. In this way a very solid, hard sole-leather is produced, which is not brittle. The tanning is considerably hastened.

The chief use of formalin for tanners is as an antiseptic, though for preventing putrefaction other materials are cheaper. Hides infected with anthrax may be rendered harmless by painting with a $2\frac{1}{2}$ per cent. solution. The most remarkable result was that skins treated with a dilute solution remained quite fresh, without the slightest smell of putrefaction, and yet in four weeks they could be unhaired completely.

A bacteriological examination of the Malpighian layer showed absence of putrefactive bacteria, but presence of some ferment in large amount. The reaction was acid. This is regarded as a proof that the sweating process need not be accompanied by putrefaction, and that the alkaline products of putrefying bacteria are not necessary for the process, as has been supposed.

The best general use of formalin in leather manufacture, at present, is for preserving glazes and seasonings.
—J. T. W.

Albumin at Chinkiang, Manufacture of. U.S. Cons. Reps., March 1897, 386.

See under VI., *page* 327.

Tannin, Estimation of. [*The Tannoform Reaction.*] E. Aweng. Apotheker Zeit. 1896, 831.

See under XXIII., *page* 364.

Gelatin, Volumetric Method for the Estimation of, in Glue and Glue Liquors. F. Jean. Rev. de Chim. Ind. 1896, **7**, [83], 339.

See under XXIII., *page* 364.

PATENTS.

Tanning, Impts. in or relating to. Alexandre Levinstein, Paris. Eng. Pat. 10,263, May 13, 1896.

In rapid tanning processes in drums, if the temperature be high, the leather becomes brittle; if low, the tanning proceeds slowly.

The temperature is therefore kept between 20° and 40° C. by injecting steam or air into the drum whilst rotating, or by varying its speed of revolution by means of conical pulleys.—J. T. W.

Tanning Apparatus, Impts. in. Alexandre Levinstein, Paris. Eng. Pat. 10,390, May 14, 1896.

Details are given of the blower for introducing cold air into the tanning drum (see preceding abstract), and also of means for taking samples of extract, and for ascertaining the temperature of the interior while the drum is rotating. The drum can be rotated at three different speeds by means of the usual conical pulleys.—J. T. W.

Tawing Hides and Skins, An Improved Process of and Compound for. G. W. Adler, Philadelphia, U.S.A. Eng. Pat. 14,293, June 27, 1896.

The tawing liquor is prepared by dissolving 5 lb. of bichromate of potash in $7\frac{1}{2}$ lb. of sulphuric acid, diluted with double the volume of water; this solution is then reduced by the addition of about 1 lb. of white sugar, or $1\frac{1}{2}$ pints of alcohol, until effervescence ceases. The solution is then rendered neutral by the addition of 20 lb. of sodium carbonate dissolved in $7\frac{1}{2}$ gals. of water; the mixture is allowed to stand several hours until precipitation of the "chromium oxycarbonate" is completed.

Hydrochloric acid (6—7 lb.) is then added, so as just to dissolve the precipitated chromium compound. The density of the liquor produced is about 20° Baumé, and its composition is probably best represented by the formula—

$$Cr_2Cl_6 + Na_2SO_4 + K_2SO_4 + NaCl + NaCHO_2, \text{ or } NaC_2H_3O_2, \text{ or } Cr_2(CHO_2)_6, \text{ or } Cr_2(C_2H_3O_2)_6.$$

The skins are prepared in the usual way, and the tawing liquor diluted to 5°—6° B., and used as in the one-bath chrome process.

It can be used for pickled or for fresh skins. The sodium chloride and sulphate present modify the astringent effect of the chromium salts. The organic acid present acts in a similar way, and greatly aids the tanning action.—J. T. W.

XV.—MANURES, Etc.

Injurious Effect of Chili Nitrate of Soda. A. Stutzer. Deut. landw. Presse, 1896, No. 66.

An examination of fields which had received Chili nitrate of soda, showed that of the various crops so manured, the injury was confined almost entirely to rye. The greater the amount of nitrate applied, the greater the injury. The results of analyses of the nitrate of soda, indicated that its composition was normal, and the author supposes that the damage was caused by deficiency of moisture in the soil, and possibly, also, by the unequal distribution of the manure.

Lemmermann (Bied. Centr. 1897, **26**, 78) calls attention to the experiments of Sjollema (this Journal, 1897, 152 and 163), indicating that the damage is due to the presence of perchlorate in the nitrate of soda. He suggests that it would be of interest to ascertain in what form the perchlorate is present; and that by means of a heavy application of potassium salts, the perchlorate might be rendered sparingly soluble, and so comparatively harmless.
—N. H. J. M.

Phosphatic Slags, Mechanical Analysis of Basic. H. W. Wiley. J. Amer. Chem. Soc. 1897, **19**, 19.

See under XXIII., *page* 360.

PATENT.

Fish and other Offal or similar Refuse, Impts. in or relating to the Treatment of. The Fish Utilisation Syndicate, Ltd., and J. C. W. Stanley, London. Eng. Pat. 7268, April 2, 1896.

The fish offal is first sorted upon a sieve; extraneous matters are picked out, whilst blood, &c., drains through into a suitable receptacle. It is then boiled with water in a boiler having a perforated diaphragm, which keeps the solid matters below the surface of the water, but allows oil to rise and flow off into a settling tank, where it is separated from the rest of the water and subsequently used for making soap, &c. The solid matter is removed from the boiler, squeezed in a press to get rid of most of the water, then dried and sifted. The finer parts, which pass through the sieve, constitute *fibre guano*; what remains on the sieve is crushed fine and sold as *bone guano*. The water out of the boiler may be precipitated with alum cake.—L. A.

XVI.—SUGAR, STARCH, GUM, Etc.

Saccharogenic Diastase in the Beetroot, Hypothesis of a. G. Arachequesne. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1897, **14**, 670—673. (See also this Journal, 1897, 252.)

With regard to the hypothesis of a saccharogenic diastase to account for the difficulty of fermentation of certain beetroots, the author points out that it leaves a number of facts unexplained, particularly the premature running to seed, generally attributed to sudden changes of temperature, but which may be caused by the rootlets being torn or atrophied.

As to claims of priority, the existence of this diastase in the beetroot and other plants, was demonstrated by Pasteur and his pupils, and the theory was published long ago by Duclaux in his *Microbiologie*.—L. J. de W.

Sugar-Cane Juice, Clarification of. J. L. Beeson. J. Amer. Chem. Soc. 1897, **19**, 56—61.

The clarification of cane juice being mainly an empirical method, the nature of the bodies removed and the chemical changes involved not being understood, it was the object of this investigation to compare the juice expressed by the mill with that obtained from the same large sample of cane by the process of diffusion with hot water, and to compare these juices before and after clarification, and study the products removed. The total proteids and albuminoids were obtained by multiplying the total and albuminoid nitrogen by the factor 6·25, the difference being termed amides; the "gums" were obtained by subtracting the total proteids and ash from the "solids not sugars."

The diffusion juice was found to contain less solids not sugars, less total proteids, and less albuminoids and amides than the mill juice, indicating that the hot water had coagulated and left in the bagasse about one-third of the albuminoids and gums. Bagasse from the diffusion process showed, on analysis, more proteids than that from the mill. The diffusion juice, after clarification with lime, showed rather less albuminoids, but a slight increase in ash and gums, attended by a lowering of the coefficient of purity. The skimmings showed a relatively small amount of proteids, indicating that these bodies had been partly broken down by the clarification.

The effect of adding lime before applying heat was now tried. One litre of mill juice required 10 c.c. of lime water to remove acidity, when a dark green precipitate formed and continued to form until 190 c.c. had been added and a faint alkalinity was reached. To remove slight excess of lime from the filtrate, superphosphate solution was added until the juice began to brighten. On heating for 20 minutes at 90° to 95° C. a small coagulum was formed. Sulphur dioxide bleached the juice, but caused no further precipitate.

The lime-heat clarification showed a marked improvement, there being an increase of 4 per cent. in the coefficient of purity, the lime having removed nine-tenths of the total non-sugars eliminated. Of the gummy bodies precipitated by nitrate of mercury, and albuminoids by copper hydroxide, one-half consist of bodies not rendered insoluble by lime or heat. It is the further work of the sugar chemist to discover some cheap, non-poisonous agent which will remove this class of bodies from cane juice.—L. J. de W.

Massecuites and Syrups, Tables for Determining Water in. E. Gravier. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1896–7, **14**, 661—669.

Tables are given showing the water in massecuites and spun syrups for degrees of apparent purity from 93 to 60, based on a number of determinations in which the water was estimated by heating 5 grms. mixed with ground glass at 105° to 110° C. for 18 hours, other results being obtained by interpolation.

In using the tables, 50 grms. of massecuite or syrup are dissolved in water, and made up to 250 c.c., and the density obtained by Mohr's balance at 15° C., water at 15° C. being taken as unity. The apparent purity is found by help of Scheibler's tables (*ibid.*, Dec. 1890), the sugar being estimated on the same solution as the density.

The purity being known, it is merely necessary to find, in the column corresponding to it, the density, and to read off the percentage of water. The maximum error is 0·4 per cent.—L. J. de W.

Massecuites, Calculation of the Quantity of Syrup added to First Runnings. O. Mittestaedt. Bull. de l'Assoc. des Chim. de Sucr. 1897, **14**, 708—710.

As the practice of returning centrifugalised syrups is very general, it is of interest to be able to calculate the real quantity of first runnings massecuite after having added, in the pan, a certain quantity of first runnings syrup, and further, to calculate the amount of syrup added.

The first question is easily solved, if one knows the dry solids of the finished massecuite and that of the syrup added. If 20 tons of massecuite have been obtained, of 6 per cent. of water, and quotient 91·4, this corresponds to $20 \times 0·94 = 18·8$ tons of dry solids, with $18·8 \times 0·914 = 17·183$ of sugar, and $18·8 - 17·183 = 1·617$ of non-sugar.

If there were added, during the boiling, 1·8 tons of syrup of 78 per cent. dry solids and quotient of purity 74, there is introduced into the massecuite $1·8 \times 0·78 = 1·4$ tons of dry solids, containing $1·4 \times 0·74 = 1·036$ tons of sugar, and $1·4 - 1·036 = 0·364$ ton of non-sugar. Deducting these figures from those of the finished massecuite, we obtain 16·147 tons of sugar and 1·253 of non-sugar, which form the dry solids of the original massecuite from the beetroot. Its quotient of purity must have been $16·147 \times 100 \div (18·8 - 1·4) = 92·8$.

The calculation becomes more complex if the quantity of syrup added is not known. Neglecting the sugar destroyed by the boiling, the quotient of that part of the massecuite from the beetroot alone must be equal to that of the syrup boiled (Dg). The quotient of the finished massecuite (Fg) will depend on the quotient of the green syrup (Sg) and its quantity. Let x denote the quantity of added syrup contained in 100 of dry solids of massecuite, there will be $100 - x$ of original massecuite dry. We have thus the equation:—

$$100\ \mathrm{Fg} = (100 - x)\ \mathrm{Dg} + x \times \mathrm{Sg},$$

$$\text{whence } x = 100\ \frac{\mathrm{Dg} - \mathrm{Fg}}{\mathrm{Dg} - \mathrm{Sg}}$$

For example, if the quotient of the finished massecuite be $\mathrm{Fg} = 91·4$, that of the concentrated syrup $\mathrm{Dg} = 92·8$, and that of the added syrup $\mathrm{Sg} = 74·0$, we get, on putting these values in the above formula, $x = 7·45$ dry solids in the added syrup in 100 parts of finished massecuite, supposed dry. If the added syrup contained 78 per cent. of dry solids, the actual amount added is $7·45 \div 0·78 = 9·54$. If the finished massecuite contained 6 per cent. of water or 94 of dry solids, we have $(100 - 7·45) \times 0·94 = 87·0$ per cent. of dry solids derived from the concentrated syrup.

—L. J. de W.

Sulphurous Acid, Inversion of Sugar Solutions by. K. Stiepel. Bull. de l'Assoc. des Chim. de Sucr. 1897, **14**, 706—707.

The author has made a very long and interesting study of the inversion of sugar in pure and impure solutions, and arrives at the following conclusions:—(1.) In pure sugar solutions: Their inversion by sulphurous acid, and by all other known acids, takes place according to the law of inversion formulated by Wilhelmy and the law of Guldberg and Waage. Pure solutions of sugar undergo with sulphurous acid, at a very low temperature, very slow inversion. (2.) In impure sugar solutions there are to be distinguished: (*a*) The influence of the salts and non-sugars present. As far as these influences are concerned, similar relations exist for SO_2 in particular, as for other acids, according to the general observations of Spohr. (*b*) The inverting effect of the acid sulphite of potash formed. It is much weaker than that of free sulphurous acid. (*c*) The inverting effect of organic acids liberated from their combinations by sulphurous acid. Leaving out the secondary effects produced by these influences, it follows that the inversion by free sulphurous acid, even in impure solutions, takes place according to the law of Guldberg and Waage.

It is therefore certain that the smallest quantity of SO_2, even in impure solutions (juice, syrup, and molasses), produces inversion, even in the cold. Although the secondary influences of the non-sugars, acid sulphite, and free organic acids are so small at low temperatures as to be practically negligeable, the inversion may become very sensible at higher temperatures.—L. J. de W.

Saccharose, Inverting and Decomposing Influence of Heat on Aqueous Solutions of. B. Rayman and O. Šulc. Rozpravy české akademie, 1896, **5**, Cl. II., 33.

The rate of inversion is dependent on the nature of the containing vessel. Thus, the polarisation of a 20 per cent.

solution of cane sugar fell from + 11·66° to + 5·94° when the liquid was boiled in a glass flask over a flame for 61 hours, to + 6·82° when heated in a water-bath. In a platinum flask, the rotation fell from + 13·25° to − 3·42° in 25 hours, the rate of inversion being fairly uniform throughout. Concentrated solutions are decomposed more rapidly. Under pressure, inversion proceeds less rapidly (at 100° C.) than in open vessels. The decomposition products formed when aqueous solutions of levulose and dextrose are heated under pressure were examined. At 105° C., levulose suffers appreciable decomposition, and at 120° C. yields formic acid, furfurol, carbon dioxide, pyrolevulinic acid, and brown ($C_{25}H_{22}O_{10}$) or reddish-brown ($C_{20}H_{20}O_9$) humin substances. At 120° C., dextrose yields very little furfurol, mere traces of non-volatile acids, and no humin bodies. At the same time a substance, having smaller (dextro) rotatory power and reducing power than dextrose, appears to be formed. At 140° C., furfurol and small quantities of humin substance ($C_{24}H_{22}O_{11}$) are produced, whilst above 150° C. formic acid appears amongst the products. At 160° C., more humin substance ($C_{24}H_{20}O_9$) is formed, whilst at 180° C. the dextrose is entirely destroyed in about 3½ hours, about one-half of it being converted into formic acid and black humin bodies—

$$4C_6H_{12}O_6 = C_{23}H_{18}O_8 + 14H_2O + CH_2O_2.$$

Amido acids invert cane-sugar slightly at 100° C., distinctly at 105° C., under pressure, the effect being the less pronounced the larger the molecular weight of the acid used. —H. T. P.

Wood Gums, The Composition of. P. Truchot. Rev. de Chim. Ind. **8**, [86], 43—44.

In Germany, and more especially in America, great attention is paid at the present time to the preparation of gums extracted from wood and other ligneous bodies, as it is believed that these products might be utilised as substitutes for some of the more expensive gums now employed, for example, by the calico printers, &c. The gums obtained from maize cobs and birch bark have recently been studied by G. W. Johnson (see this Journal, 1896, 605).—I. S.

Beetroot Juice, Estimation of Lime in, by Alcoholic Soap Solution. N. Rydlewski. Bull. de l'Assoc. des Chim. de Sucr. 1897, **14**, 696.

See under XXIII., *page* 365.

Beetroot, Determination of Sugar in the. K. Kaiser. Bull. de l'Assoc. des Chim. de Sucr. 1897, **14**, 688.

See under XXIII., *page* 364.

Beetroot, Method of Analysis of the. L. Jesser. Bull. de l'Assoc. des Chim. de Sucr. 1897, **14**, 684.

See under XXIII., *page* 364.

Sugar in Beetroot, Comparison of Direct Methods of Estimating. J. Graftiau. Bull. Assoc. Belge des Chim. **10**, [9], 354.

See under XXIII., *page* 364.

Starch, Estimation of, in Frozen Potatoes. D. Saare. Zeits. f. Spiritusind. 1897, **20**, 51.

See under XXIII., *page* 365.

PATENT.

Saccharine Solutions and other Liquids, Impts. in and Apparatus for Purifying and Decolorising. J. Y. Johnson, London. Eng. Pat. 28,763, Dec. 15, 1896.

A convenient quantity of the saccharine liquid is introduced into a vessel provided with plates of suitable material for positive and negative electrodes. The tank is connected with a reservoir containing ozone and also with a vacuum pump. It is preferable to employ aluminium, the irregular and pitted surface of which has been filled up with alumina, for the positive electrode, because the electrolytically formed alumina has a decolorising effect on the liquid. Favorable results are also obtained with zinc pitted in the same way as the aluminium and filled with oxide of zinc. The negative plate is generally made of carbon, provided with openings, &c. in which the impurities are deposited together with alumina or oxide of zinc, which may subsequently be recovered.

The liquid to be treated should not have a density above 40° B., and, if dark, may be heated from 70° to 90° C. In carrying out the process, from 4 to 10 minutes are usually required to complete the treatment, if the electrolysis be carried on simultaneously with the passage of ozone through the liquid, and from three to five hours if the ozone treatment follows the electrolysis. A current of 50 to 60 ampères at 5 to 10 volts is used per square metre of electrode. The effect of the ozone is to immediately oxidise the impurities, thereby completing the defecation which has been partially accomplished by the electrolysis.

If a high degree of defecation be required, it is best to treat the electrolysed juice with ozone *in vacuo* in a second tank. The liquid is run in by means of a sprayer simultaneously with the introduction of ozone, the two being thus thoroughly incorporated.—J. L. B.

XVII.—BREWING, WINES, SPIRITS, Etc.

Fermentation: A Reply to a Criticism by Prof. Duclaux (this Journal, 1896, 664—665). A. J. Brown. J. Fed. Inst. Brewing, 1897, **3**, 99—110.

In a paper on the "Influence of Oxygen on Alcoholic Fermentation" (this Journal, 1892, 257), the author described some experiments which appeared not to agree with Pasteur's well-known theory that fermentation is a consequence of "life without air." In a subsequent paper on "The Specific Character of the Fermentative Functions of Yeast Cells" (this Journal, 1894, 824), evidence was brought forward to show that the argument on which Pasteur based his theory was unsound. This theory depends entirely on the question of his determinations of fermentative power being independent of time. The *fermentative power* of a yeast, according to Pasteur's definition, is expressed by the proportion of sugar decomposed, to the weight of yeast which has decomposed it. If, however, the time be taken into consideration, together with the yeast formed and sugar fermented, the *fermentative activity* is determined—a property quite distinct from the fermentative power.

The author considers that Pasteur, in calculating his fermentative powers, had overlooked the fact that a yeast cell, like other living organisms, does not produce energy directly, but merely transmits it. The energy necessary to carry on the life work of living organisms is derived from the aliment they consume; the action of their functions, so far as the organisms are concerned, is a continuous action, and may be compared to the working of an engine fed with fuel. The decomposition of sugar by a yeast cell is a continuous function so far as the cell is concerned, and consequently it must depend on time, like all continuous phenomena under similar conditions.

In an ordinary fermentation, yeast cells multiply rapidly at first, their rate of multiplication gradually slackening until it ceases. Meanwhile fermentation continues during the existence of each individual cell so long as there is fermentable sugar in the liquid. Since fermentation is a continuous function of the yeast cell, those cells which came into existence first will have a longer time at their disposal to decompose sugar than those which are formed later, but the time factor rules the process for each individual cell, however long or short an existence it may have. It will be apparent on these grounds that time controls the ultimate result.

The author is of opinion that the expression *fermentative power*, as defined by Pasteur, should be abandoned and *fermentative activity* be retained to express the fermentative power of an organism in unit time.—J. L. B.

Yeasts, Carbonaceous Feeding of Budding. Th. Bokorny. Dingler's Polyt. J. 1897, **303**, 115—119; 140—143; 163—167.

Naegeli and Loew have found (Sitz Ber. d. math. phys. Kl. Munich, July 1879) that simply constituted organic compounds, such as acetic acid, can afford the necessary carbonaceous food to yeast-moulds; and Loew states that, whilst formaldehyde behaves as a strong poison to certain bacteria, yet, as the author has shown, the sodium sulphite compound of formaldehyde can be assimilated. Acids, alcohols, carbohydrates, acid amides, and bodies related to albuminoids are carbonaceous foods to yeasts; and, in the opinion of Loew (Die chem. Kraftquelle im leb. Protopl., Loew and Bokorny, Munich, 1882), these bodies primarily split off a CH_2O group, which then becomes changed into more complex carbon compounds. The author concludes from his work that only those substances can be assimilated by yeasts resembling to some extent the constituents from which the yeast-cell wall is built up. The salts containing the necessary nitrogen, sulphur, phosphorus, potassium, and magnesium were made into a 0·2 per cent. solution, the organic compound being often in larger quantity. The reaction of the resulting solution should be slightly acid or neutral, but not alkaline, as moulds would then flourish. The author's experiments were carried out in an incubator at 25°—30° C. with the substances shown in the following table, which gives his results, together with those of other observers:—

Methyl alcohol (0·2 per cent.) Not a carbonaceous food for yeast.

Ethyl alcohol (0·2 per cent.) Not a carbonaceous food for yeast.

Amyl alcohol (0·2 per cent.) Not a carbonaceous food for yeast.

Propyl alcohol. Not a carbonaceous food for yeast.

Benzyl alcohol (0·2 per cent.)

Ethyleneglycol. Doubtful.

Glycerin (0·2 per cent.) Yeast flourishes freely.

Phenol (0·05 per cent.) Not a carbonaceous food for yeast.

Catechol (0·05 per cent.) Not a carbonaceous food for yeast.

Resorcinol. (0·05 per cent.) Not a yeast food.

Gallic acid (0·05 per cent.) Not a yeast food.

Tannin (0·05 per cent.) Not a yeast food.

Pyrogallic acid (0·05 per cent.) Not a yeast food.

Quinol (0·05 per cent.) Not a yeast food.

Orthoxylenol. Poisonous.

o- and *p*-cresol. Poisonous.

The nutritive value of the following organic acids was ascertained, neutral solutions of the salts being used:—

Propionic acid (0·2 per cent.) Not a carbonaceous food for yeast.

Succinic acid (0·2 per cent.) Not a carbonaceous food, and unfavourable to yeast.

Aspartic acid (0·2 per cent.) A yeast food.

Quinic acid [$C_6H_7(OH)_4COOH$]. Not a yeast food, but favours moulds and fission fungi.

Parahydroxybenzoic acid (neutralised). A carbonaceous food for bacteria, but not for yeast.

Acetic acid. A yeast food.—*Naegeli.*

Citric acid. A yeast food.—*Naegeli.*

Tartaric acid. A yeast food.—*Naegeli.*

Aldehydes.

Acetaldehyde. A poison to yeasts.—*Bokorny.*

Formaldehyde. Poisonous.—*Bokorny.*

Hydroxybenzaldehyde. Not a carbonaceous food.—*Bokorny.*

o-Nitrobenzaldehyde. Poisonous.—*Bokorny.*

Glyoxal. Not suitable as a mould food.—*Loew.*

Carbohydrates.

Cane sugar. Budding yeast flourishes freely. Favours the production of glycogen.—*Naegeli and Loew.*

Glucose. Is easily assimilated by yeast, with production of glycogen.—*E. Laurent.*

Glucose. A trace of yeast develops within four days in a 0·2 per cent. solution to a considerable sediment.—*Bokorny.*

Fructose (levulose). A good food for yeast.—*Bokorny.*

Galactose. A carbonaceous food for yeast.—*Bokorny.*

Milk-sugar (0·2 per cent.) A yeast food.—*Bokorny.*

Milk sugar (0·2 per cent.) Yeast produces from 1 to 40 per cent. solution of glycogen.—*E. Laurent.*

Rhamnose (0·2 per cent.) Is fairly easily assimilated by yeast.—*Bokorny.*

Sorbose (0·2 per cent.) Yeast grows feebly therein.—*Bokorny.*

Arabinose (0·2 per cent.) A yeast food.—*Bokorny.*

Maltose. In 1 to 5 per cent. solution is assimilated by yeast, with a considerable production of glycogen.—*E. Laurent.*

Inositol. Is feebly assimilated by yeast in 1 to 2 per cent. solution, without production of glycogen.—*E. Laurent.*

Mannose.—Assimilated by yeast.—*Bokorny.*

Xylose. Assimilated by yeast.—*Bokorny.*

Mannitol. Assimilated by yeast, with formation of glycogen.—*E. Laurent.*

Erythritol. Is feebly assimilated by yeast, without formation of glycogen.—*E. Laurent.*

Erythrodextrin. Is assimilated by yeast, with production of glycogen.—*E. Laurent.*

Salicin. Is assimilated by yeast, with production of glycogen.—*E. Laurent.*

Amygdalin. Is assimilated by yeast, with production of glycogen.—*E. Laurent.*

Amido Compounds.

Peptone. A good carbonaceous food for budding yeast.—*O. Loew.*

Asparagine. An excellent carbonaceous food for budding yeast.—*Birner.*

Asparagine. Is assimilated, with production of glycogen.—*E. Laurent.*

Leucin (1 per cent.) Is somewhat assimilated by yeast, with formation of glycogen.—*E. Laurent.*

Asparaginic acid (1 per cent.) Is assimilated, with formation of glycogen.—*E. Laurent.*

Glutaminic acid (1 per cent.) Is assimilated, with formation of glycogen.—*E. Laurent.*

Glutamine. Is assimilated, with slight formation of glycogen.—*E. Laurent.*

Casein. Assimilated, with formation of glycogen.—*E. Laurent.*

Egg albumin. Assimilation, with slight production of glycogen.—*E. Laurent.*

Aniline. Unsuited for a yeast food.—*E. Laurent.*

o-Toluidine neutralised (0·1 per cent.) Not a carbonaceous food for yeast. Good nitrogenous food for moulds.—*Bokorny.*

p-Anisidine (0·1 per cent.) A nitrogenous, but not a carbonaceous food for yeast.—*Bokorny.*

Nitraniline (0·1 per cent.) A nitrogenous, but not a carbonaceous food for yeast.—*Bokorny.*

Methylamine. Not a carbonaceous food for yeast.—*E. Laurent.*

Ethylamine. Not a carbonaceous food for yeast.—*E. Laurent.*

Propylamine. Not a carbonaceous food for yeast.—*E. Laurent.*

Formamide. Not a carbonaceous food for yeast.—*E. Laurent.*

Acetamide. Not a carbonaceous food for yeast.—*E. Laurent.*

Other Organic Compounds.

o- and *p*-nitrocinnamic acid. Not a carbonaceous food for yeast.—*Bokorny.*

p-Nitrotoluene. Not a carbonaceous food for yeast.—*Bokorny.*

Benzene. Not a carbonaceous food for yeast.—*Bokorny.*

—J. L. B.

P

Wine Yeasts, Researches on the Differential Characteristics of. C. Forti. La Bière, **5**, [1], 9.

The author having, from previous researches, been led to form the opinion that the morphological and physiological characteristics of wine yeasts are insufficient of themselves to determine the suitability of the yeasts to ferment musts of different composition, and that for this purpose practical experiments in fermentation are necessary, made a number of investigations with natural musts at different temperatures, as well as with varying quantities of sugar, degrees of acidity, kinds of antiseptic bodies, and nitrogenous matter; also with mixed yeasts and with wines still containing sugar. He finds that each yeast varies in behaviour as the temperature, nature of nitrogenous food, acidity, and alcoholic content of the medium are modified. So far as temperature is concerned an exposure to 35° C. for 5 to 15 days weakens all wine yeasts, the longer period proving fatal; and prolonged exposure to a temperature of 25° C. is also injurious. In presence of nitrogenous matter the yeasts adapt themselves in different degrees, each yeast needing a special amount of nitrogen varying with the kind of nitrogenous matter provided. The influence of the degree of acidity varies with the nature of the ferment, the temperature, and the amount of sugar. The yeasts differ in their behaviour towards sugar in presence of alcohol, some being able to perform their functions when the medium contains 12 or 13 per cent. of alcohol, whilst others are rendered inactive by a much smaller quantity. On the other hand, the production of spores at 25 C., and the appearance of the colonies on gelatin, form an insufficient basis for classification.—C. S.

Beer Yeast, Composition of the Fat present in. E. Gérard and P. Darexy. J. de Pharm. et de Chim. 1897, [6], 275—280.

The fat extracted from ordinary brewery yeast is brownish in colour, semi fluid, and distinctly acid in reaction. It consists largely of the glycerides of stearic and palmitic acids (in about equal proportions), together with traces of butyric acid. In addition, yeast fat probably contains small proportions of these acids in the free state.—H. T. P.

Distillery Work, Study of Several Fungi utilisable in. E. Sorel. Rev. Chim. Industr. **8**, 13—17.

The ragi employed in Java for the production of arrack consists of particles of the mycelium of Chlamydomucor Orizæ, accompanied by yeast cells and bacteria. This fungus, like Rhizopus Orizæ, has very little action on ordinary rice or potato starch, but converts cooked starch into glucose. Neither of the organisms has the power of producing alcohol, this function being discharged in the present instance by Monilia Javanica, which ferments dextrose, levulose, maltose, and raffinose (but not lactose), but ceases to act when the alcohol reaches 5 per cent. of the medium; and by Sacch. Vordermanni, a very active ferment, capable of eliminating 18 to 19 per cent. of sugar in three days at 25 to 30° C., and of retaining its power in presence of 9 to 10 per cent. of alcohol.

Japanese saké is prepared by the aid of the diastase secreted by Aspergillus Orizæ—an organism living on and saccharifying all classes of rice. (See this Journal, 1886, 170, and 1890, 754.)

Referring to the researches of Juhler (C. Bl. f. Bakt. 1895, Oct. 9), on the evolution of saccharomycetic yeasts from Aspergillus Orizæ, the author finds that when the fungus is rendered incapable of growing under normal conditions, the yeast is always developed, although under more favourable circumstances no yeast is produced. Thus, whilst conidia sown on malt extract containing 0·030 grm. of hydrofluoric acid per litre, gave yeast in three or four days, the production of yeast was retarded to 10 days in presence of twice the above quantity of antiseptic, and ceased altogether when a five-fold quantity was used; whereas an unproductive fragment of mycelium from a wort culture with 0·030 grm. of HF transferred to a medium with 0·060 grm. of the acid, although initially free from yeast, gave yeast cells on the following day, and when re-washed and placed in a wort with 0·090 grm. of HF, produced rapid fermentation and a considerable deposit of active yeast cells, itself finally disappearing after conversion into a compact tissue of short cells. The results obtained in experiments with aerated media also confirm the statement that the formation of mycelium always precedes that of the yeast, and that when the development of the former is arrested, the size of the filaments decreases, and they subdivide into budding cells having the properties of yeast. The absence of direct formation of yeast cells from the conidia, as observed by Juhler, is attributed to the difference in the media employed.

Further researches in connection with the converse development of the yeast cells into the original form of the mould confirm the results previously obtained by the author (Comptes rend. **121**, 948). He finds that the yeast becomes incapable of saccharifying starch in solution, but that it will give (even after an existence of over a year), with cooked rice in an almost dry state, a mycelium possessing diastatic power. The development of the mycelium is, however, slow, and unless the water, carbon dioxide, and alcohol formed are immediately removed, it becomes paralysed, and the untransformed yeast sets up fermentation. This was proved by experiment in a series of four flasks, aerated by a single current passed through each in succession, where the yeast developed conidia of Aspergillus in the first, a little mycelium in the second, but only produced alcohol and carbon dioxide in the remaining two; whereas on the interposition of condensers and washing tubes containing baryta water, between the flasks in a parallel series of experiments, conidia were developed in all four.—C. S.

Denaturing of Spirits. Zeits. f. Spiritusind. 1897, **20**, 19.

The members of the acetone group recommended by Lang for denaturing spirit may be divided into two classes, namely, (1) ketones of the type of ethylmethylketone, which are readily soluble in water; and (2) the acetone or ketone oils which are insoluble, or nearly so, in water. Experiments have shown that the complete removal of ethylmethylketone from spirit cannot be satisfactorily effected; and brandy spirit (for Cognac, &c.) to which this ketone has been added cannot be completely renatured either by filtration, rectification, or by treatment with ether, light petroleum, benzine, by treatment with acids, alkalis, paraffin oil, animal charcoal, &c. Filtration is impracticable, as at least 600 kilos. of charcoal would be required for the treatment of 100 kilos. of spirit. Experiments have also been made with crude ketone oil, distilling between 75° and 230°, and with the different fractionated constituents obtained from it. One per cent. of oil was used for denaturing the spirit, and it was found that when the constituent of lowest boiling point (75°—105°) had been used, a partial renaturing could be effected by careful rectification of the previously diluted spirit. This was not the case, however, when the crude oil or a fraction boiling above 150° had been used. In the experiments on filtration through charcoal, the denatured spirit was diluted to about 30 per cent., and passed through 5 kilos. of charcoal; each litre of filtrate was tested with phenylhydrazine, and those samples which gave no reaction were regarded as renatured. The following results show the weight of charcoal required to renature 100 kilos. of spirit to which 1 per cent. of oil had been added:—

	Kilos.
1. Denatured with methylethylketone required at least	600
2. Denatured with acetone oil (b. pt. 75°—230°) required at least	100—120
3. Denatured with acetone oil (b. pt. 75°—105°) required at least	140—150
4. Denatured with acetone oil (b. pt. 105°—150°) required at least	100—110
5. Denatured with acetone oil (b. pt. 150°—200°) required at least	70—80
6. Denatured with solvent naphtha required at least	9—10

In the case of spirit which had been denatured by the addition of 1 per cent. of ethylmethylketone and 2 per cent. of acetone oil, it is calculated that 1,000 kilos. of charcoal would be required for the complete renaturing of 100 kilos. The addition of chemicals, such as sodium sulphite, potash, or ammonia also fails to effect the removal of this mixture.

Lang's experiments have been repeated and, in the main, confirmed by Heinzelmann.—A. K. M.

Wine, Solvent Action of, on Metallic Zinc. L. A. Levat. Comptes Rend. 1897, **124**, [5], 242—243.

A strip of ordinary sheet zinc when placed in a bottle of red wine (sealed down) was found to be attacked to a marked extent, hydrogen being slowly evolved (sufficient at the end of 10 days' contact to blow the cork out of the bottle). After a month, the wine had become turbid and possessed a metallic flavour. The surface of the zinc was corroded and bright, but studded here and there with granules of a zinc salt. The use of zinc or galvanised taps and fittings for wine tuns, &c. must therefore be condemned. —H. T. P.

Wines, Loss of Colour in (La Casse), produced by a Soluble Oxidising Ferment of. P. Cazeneuve. Comptes Rend. 1897, **124**, [8], 406—408.

The malady known as *la casse des vins*, and which consists in the oxidation of the red colouring matter of the wine, has been attributed by Gouirand to the action of a diastase. Martinaud admits the presence of an oxidising ferment—*oxydase*—in ripe grapes, whilst Bertrand confuses this ferment with laccase. The author has examined a large quantity of a Beaujolais wine affected by this malady. By treatment with strong alcohol, a gummy precipitate was obtained, consisting mainly of the normal gum of wine impregnated with oxydase. The aqueous solution exhibits the fundamental properties of laccase, but it would be premature to assume the identity of the latter with the oxydase of wine which the author terms *œnoxydase*. This substance acts upon wine at temperatures even below 0°; it is instantly destroyed at 70°—75°. From a comparative study of the wines from different vineyards, the author is led to the opinion that the abundance of œnoxydase in the Beaujolais wines last year is to be attributed to certain vegetative conditions favouring its formation, rather than to the presence on the grapes of cryptogamic parasites which might secrete oxydase. The author's observations invalidate the conclusions arrived at by Laborde. (This Journal, 1897, 156.)—A. K. M.

Malt Wines. List. Chem. Zeit. 1897, **21**, [1], 1—5.

The statement that by Sauer's method, a product containing 16—17 per cent. of alcohol could be obtained by direct fermentation has been confirmed by the work of Dr. Moslinger, of Neustadt.

Sauer sterilises a 20 per cent. wort, warms to 50°, and sows it with a culture of lactic acid bacillus. When the lactic acid content reaches 0·8 per cent., the liquid is heated to 70° to stop the action, quickly cooled to 20°, and sown with a pure yeast culture derived from Tokay or Spanish grape fermentation. As the action slackens, more sterilised wort is added, and the sugar-content kept up by cane-sugar, till fermentation will go on no longer. The products come into the market as "Malton Tokay" and "Malton Sherry" respectively. The following figures have been obtained by Fresenius, Moslinger, and the author on analysis of the commercial article or of a privately fermented product.

—		Sherry.	Tokay.
Total extractive ..	F.	12·47	28·29
	M.	11·52	28·25
	L.	11·10	28·07
Sugar calculated as dextrose	F.	6·81	19·07
	M.	5·59	17·74
	L.	6·33	19·77
Alcohol, by weight	F.	13·36	9·92
	M.	13·71—14·34	9·61
	L.	14·71	10·59
Nitrogen (from peptones)	F.	0·067	0·0810
	L.	0·075	0·0942
Phosphoric acid ..	F.	0·084	0·1280
	L.	0·093	0·1362

These wines have been found of value medicinally, and must, from their purity, and their content in "extractive," alcohol, and sugar, be regarded in many cases as preferable to the sweet Southern wines.—J. T. D.

The Sparging Process and its Influence on the Yield of a Mash. W. Windisch. Wochenschr. f. Brauerei, 1897, **14**, 1.

In this article the author refers to two points, the importance of which has not been sufficiently emphasised in his previous papers. 1. A primary condition for obtaining a good yield depends upon the thorough draining of the first wort from the grains before any sparging liquor is added to the latter. 2. The second condition mentioned is that sufficient time be allowed for the sparging process. If this be hastened, the wort contained within the pores of the grains will not have time to diffuse into the sparging liquor, and a loss will thus occur.—A. K. M.

Beer, Japanese. A. Lang. Zeits. f. d. ges. Brauwesen, **20**, [2], 27.

The cultivation of barley in Japan has greatly increased of late years, and a large quantity is now used for malting and brewing. Two samples of Japanese beer, one from the Asahi and the other from the Yebisu brewery have recently been analysed with the following comparative results (after driving off the carbon dioxide):—

—	Saccharometer Reading.	Alcohol.	Extract.	Maltose.	Dextrin.	Total Acid (calculated as Lactic Acid).	Protein (N. 6·25).	Ash.	Phosphoric Acid.	Glycerin.	Calculated. Original Wort, Balling.	Apparent Attenuation.	Actual Attenuation.
	Balling.	%	%	%	%	%	%	%	%	%			
Asahi beer	3·59	4·60	5·642	1·725	..	0·133	0·454	0·214	0·0718	0·145	14·44	75·14	60·93
Yebisu.................	3·12	4·56	5·23	1·29	2·41	0·161	0·491	0·222	0·051	0·120	13·98	77·7	62·6

The concentration corresponds with that of Bavarian lager beer, and the high attenuation with that of North German beers. In flavour the Yebisu is vinous, whilst that of the Asahi beer recalls apple must (due to pasteurisation). The latter was not clear and had deposited a sediment consisting of glutin without any living organisms.—C. S.

Mechanical Clarification of Beer by Wood Chips and Filtration. Amer. Brewer's Review, **10**, [7], 245—246.

The use of chips and filters is very extensive in American breweries and gives satisfactory results. German brewers, however, according to a recent address by Dr. Will at Munich, while adopting chips, do not take kindly to the use of filters. Chips of hazel or beech wood are the most commonly used and act as a good clarifying medium, holding the sediment down fast, so that if they are properly arranged in the cask the liquid can be drawn off clear to a lower level than in such casks as have no chips. The aluminium chips recently introduced obviate the risk of infection by the use of dirty wooden chips, and have been proved to be without effect on the flavour of the beer. According to Dr. Will the filtration of beer causes a deterioration of taste, and the beer loses in fulness and in the consistency of the foam. From a biological point of view the beer also suffers deterioration, since the filter retains only the large vigorous yeast cells whilst wild yeasts

and bacteria, for the most part, pass through without obstruction and afterwards develop all the more vigorously owing to the absence of yeast.

These views of Dr. Will are by no means, however, borne out by the experience of United States brewers, which is that the beer suffers no detriment whatever by filtration.
—J. G. W.

Butyl-Alcoholic Fermentation. O. Emmerling. Ber., 1897, [4], 451—453.

The author previously (Ber., 1896, 2726) stated that he could not obtain the bacillus described by Fitz which produced butyl-alcohol from glycerin; he has now, after examining a number of materials, obtained the bacillus from hay from Alsace. As it was only present in a comparatively small proportion in the hay infusion, this was increased by growing the mixed bacteria in a 5 per cent. glycerin solution *in vacuo*.

As *bacillus subtilis* is ærobic, whilst the butyl-alcohol bacillus is anærobic, the proportion of the latter was increased, and after three cultivations, plate cultivations were made and the bacilles which was sought, obtained in a pure state.

The bacillus agrees fairly well with Fitz's description being slightly elliptical rods; it gave 6 per cent. of butyl-alcohol from glycerin, and 10 per cent. from manitol, whilst when acting on dextrose only ethyl alcohol was produced. The author thinking that this organism might be identical with that described by Beyerinck (C. B. Bact. 15, 177) as *granulobacter saccharobutyricum*, has compared the two, but finds that though resembling one another in appearance, they possess very different functions.

The bacillus was also obtained from rotten wood, but is not the one which causes the destruction of the cellulose.
—A. L. S.

Mould Fermentation. O. Emmerlich. Ber. 1897, [4], 454—455.

As is well known, moulds, when grown under anærobic conditions in sugar solutions, act as yeasts and produce alcoholic fermentation. The author considered it would be of interest to determine if glycerin and succinic acid were produced.

He employed *Mucor Racemosus* growing in a cane-sugar solution and replaced the air by hydrogen. After three weeks' fermentation at 25° C. the fermented solution was found to contain alcohol, glycerin, and succinic acid in the proportion of 22·0, 1·83, and 0·31 respectively.—A. L. S.

Extracts. A. Rogers. J. Fed. Inst. Brewing, 1897, 3, 129—133.

An average malt will yield 78 per cent. extract, but in practice about 70 per cent. is obtained, the remaining 8 per cent. of the starch being incompletely modified. The author recommends that fluted rollers should be used in the malt mill, as they help to catch smaller grains that might otherwise escape; also that the mash tun should be provided with rakes. The lower the striking heat of the water can be arranged to suit the resulting beers, the higher will be the extract obtained. For brewing a strong beer with a full tun, the following method is suggested:—

The mash is run into the tun with a minimum quantity of one and a half barrels of water to a quarter of malt. It is allowed to stand the usual time and no underlet used, unless the initial heat requires to be raised. The taps are then set, the tun drained dry and the goods sparged. This operation is repeated, the sparge, however, not running while the taps are flowing, care being taken that when this is finished no wort is left in the tun. Under these conditions a full extract is said to be obtained.—J. L. B.

Corks [Beer and Wine], Dangers occasioned by. H. Will. Zeits. f. d. ges. Brauw., 20, 93—96.

Beer, unlike wine, is not stored long enough in bottles to become "corked," but may, if the corks are not scalded, be contaminated by extractive matter which causes turbidity, and may also be infected with wild yeast and fungi contained in the cracks and fissures of twice used corks. As pointed out by Wortmann, at the late Congress of German wine producers, the porous nature of cork permits diffusion so that export beers, even though pasteurised, may, by the intrusion of oxygen, have any dormant micro-organisms present revived with disastrous results.

Wine left in bottle for 25 or 30 years has been found when uncorked, to contain living organisms in full activity and especially mould fungi capable of eliminating alcohol and of imparting to the liquid, unpleasant flavours and odours, even when present in small numbers. Similar effects may be produced in beer from the presence of mycoderma, a few individual organisms sufficing to develop a musty flavour.

Wortmann investigated the causes of "corkiness" in wine and found that it may either be due to organisms in the wine without the cork being more than a passive assistant (by permitting the admission of oxygen), or else directly to defective corks without the aid of any organism. In the first instance, however, mould fungi may penetrate to the wine by pushing their fibres through the natural channels existing in the cork, and Wortmann found infection from various kinds of penicillium, dematium yeasts, racodium cellare, and cladosporium herbarum, communicated in this way, each of which developed musty and unpleasant flavours differing with the kind of wine exposed to the infection. A similar case of infection by penicillium has been observed in bottled beer, a strong growth appearing on the underside of the cork, and a mycelium developing in the beer, which was quite spoiled.

It is therefore advisable to select corks with great care, rejecting those in which the natural fissures run longitudinally, and controlling the scalding process so as to preserve the elasticity of the material.

When wine is spoiled by defective corks without the agency of any organism, the effects are due to decomposition of the matter contained in the fissures and the gradual extraction of the products by the adjacent liquid. This may be prevented by steeping the corks in paraffin, or paraffin and wax.—C. S.

White Wines, Testing for Coal-Tar Colours in, and the Difference between these Colours and Caramel. A. d'Aguiar and W. da Silva. Comptes Rend. 1897, 124, [8], 408.

See under XXIII., *page* 361.

Potassium Bitartrate, Estimation of the, contained in Wine. H. Gautier. Comptes Rend. 1897, 124, [6], 298—300.

See under XXIII., *page* 365.

PATENTS.

Rice and other Grain or Seed for Brewing and other Purposes, Impts. in the Treatment or Preparation of; and in Means or Apparatus to be employed therein. A. Perry and H. Perry, Bow, Middlesex. Eng. Pat. 5615, March 12, 1896.

The object of this invention is an improved process for "flaking" rice or other grain in order to render it more suitable for use in distilling, brewing, &c.

The rice or other grain is fed into the upper part of a perforated cylinder immersed in a tank of boiling water, and in order that the scalding may be regularly performed the cylinder is divided by a horizontal partition which does not quite reach to the end. The material which is being treated is conveyed by screws through the upper chamber thus formed until it falls through the opening left by the short partition into the lower portion of the cylinder, and is then conveyed by screws until it is delivered into an elevator at the other end of the cylinder.

The scalded material is then conveyed in the same manner through another perforated cylinder immersed in cold water; this treatment removes the stickiness of the grain, and enables it to be handled and passed through the machinery without trouble.

The final operation consists in passing the grain between highly heated pressure rollers of large diameter. The rollers are kept hot by passing heated sand through them, the sand as it passes from the rollers being returned to the furnace, and after passing through these is again led through the rollers.

The flaked grain is removed from the hot rollers by scrapers and carried away on an endless belt. The machinery is fully described by the aid of drawings.
—A. L. S.

Wines and Spirits, Apparatus for Removing Saccharine and other Impurities from. From J. T. Bennett, Cape Town. C. H. McEuen, London. Eng. Pat. 22,403, Oct. 9, 1896.

This apparatus consists of a funnel-shaped vessel provided with an air-tight cover, and inlet and outlet valves. The latter is in combination with an open vat, also fitted with an inlet and draw-off. In the operation, the vat is filled with water to a height above the outlet of the funnel, whilst the funnel itself is filled with the wine or spirit to be purified. The inlet tube is then closed. Under these conditions the wine and water, though in contact, will not mix, but the heavier saccharine or vegetable impurities will descend into the vat.—J. L. B.

Malt Liquors, Impts. in Apparatus and Process for Treating. A. E. Feroe, New York, U.S.A. Eng. Pat. 21,022, Sept. 22, 1896.

The beer having arrived at that stage of the fermentation when it is nearly ready to rack into casks, it is confined under a pressure greater than that produced by fermentation, and allowed to deposit the yeast. It is then cooled to cause the semi-soluble albuminoids to separate, and the beer still under pressure, passes upwards through a filter. The beer is then racked into casks without loss of pressure. The inventor has devised an elaborate apparatus for performing these operations, which is fully described.—A. L. S.

Mash-Tuns and Conversion Vessels for Brewers' and Distillers' Use, Impts. in or connected with. [*Lining with Silver.*] R. D. Bailey, Gloucester, and L. P. Ford, London. Eng. Pat. 27,275, Dec. 1, 1896.

The inventors propose to coat with silver all metallic parts of mash-tubs or conversion vessels.—A. L. S.

Alcoholic Beverages, A New or Improved Process and Apparatus for Rapidly and Economically Ageing or Maturing. F. C. Haeck, Brussels. Eng. Pat. 1162, Jan. 15, 1897.

The alcoholic liquid to be matured is subjected to a progressive and prolonged heating (40°—55° C.) in an apparatus warmed by hot-water pipes and provided with a cover, whereby the volatile substances which evaporate may be removed from the heater. There are also discharge orifices which permit the separate withdrawal from the apparatus of the treated liquid, the noxious matters of lower specific gravity than the liquid (propyl, butyl, and amyl alcohol), and the deposited dregs. The objectionable constituents having been thus separated, the liquid is drawn off into hermetically closed barrels and allowed to remain at a low and constant temperature for several weeks, when it is filtered or clarified, and bottled.—J. L. B.

XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

(*A.*)—FOODS.

Cheese, Estimation of Nitrogen in. B. A. van Ketel and A. C. Antusch. Nederl. Tijdschr. Pharm. 1897, 82.

See under XXIII., *page* 366.

Tea, Analysis of. Aglot. Rev. Chim. Ind. 8, 17.

See under XXIII., *page* 365.

Fat in Milk, Estimation of. H. Fresenius. Zeits. Anal. Chem. 36, [1], 31.

See under XXIII., *page* 366.

PATENTS.

Albuminous Substances, A Process for the Preparation and Extraction of, from Animal and Vegetable Mixtures and Products. D. Finkler, Bonn, Germany. Eng. Pat. 5749, March 14, 1896.

This process consists in converting into soluble products, or completely decomposing, such substances contained in albumin as are unpleasant and injurious to health. This is effected particularly by heating or boiling the albumin with hydrogen peroxide of such a strength that it is without action on the albumin itself; the soluble product may then be removed by washing, leaving albuminous substances, which, when well dried, are free from smell and taste, rich in albumin, and nourishing as food.—J. L. B.

Coffee, Impts. in the Manufacture of. H. Boisselier, Middlesex. Eng. Pat. 7904, April 15, 1896.

Equal weights of washed coffee, and an oil or fat melting at about 90° F., are mixed and ground between two horizontal stones; when a sufficient degree of fineness has been attained, there results a semi-liquid paste, perfectly smooth and free from grit. This is collected whilst warm, placed in bags or cloths, and submitted to hydraulic pressure, whereby the whole of the fat is removed. The coffee remaining in the cloths is pulverised and sifted, and yields a product readily miscible with hot water for making the beverage.
—J. L. B.

Food Products and Beverages, An Improved Process for Sterilising and Preserving. W. Nägeli, Mombach, Germany. Eng. Pat. 6496, March 24, 1896.

An acid (or alkali) is placed in the preserving vessel whilst being heated in the sterilising chamber. A basic (or acid) substance is inserted in the stopper of the tin or glass containing the sterilised food, in such a manner that on turning the bottles or tins, combination of the acid and base takes place. This process can also be used for saturating non-alcoholic beverages with carbonic acid, the bicarbonate being placed in the stopper and dissolved after the sterilisation and closing of the vessel. By the distillation of fermented beer, and by its subsequent sterilisation and saturation with carbonic acid as described above, non-alcoholic beer may be prepared.—J. L. B.

(*B.*)—SANITATION; WATER PURIFICATION.

Ozone, Atmospheric; Estimation of, on Mont Blanc. M. de Thierry. Comptes Rend. 1897, 124, [9], 460.

See under XXIII., *page* 360.

PATENTS.

Water Softening and Purifying Apparatus, Impts. in. F. H. Tyacke, Old Broad Street, E.C., and C. E. Gittins, Iverson Road, West Hampstead. Eng. Pat. 5849, March 16, 1896.

The apparatus comprises a tank to contain the purifying reagent, a mixing and settling tank having vertical partitions extending alternately from the top and bottom so as to form a zig-zag passage for the water, a storage tank for the softened and purified water, an elevator adapted to discharge the reagent in a suitable proportion into the mixing and settling tank, a water motor (preferably a bucket wheel) adapted to drive the elevator and actuated by the water to be purified, and a supply valve automatically controlled by a float located in the storage tank, for admitting the water to the motor and mixing tank.—R. A.

Sewage and other Waste Liquids, The Utilisation of By- and Waste Products [Flue Dusts] from Iron and Steel Industries for the Manufacture of Improved Precipitants for the Purification of. W. E. Adeney and W. K. Parry, both of Dublin. Eng. Pat. 5955, March 17, 1896.

The flue dusts produced in the manufacture of iron, ferro-manganese, and steel, are digested with hydrochloric or

sulphuric acid in sufficient proportion to dissolve the chief basic substances present. The treated mass may be dried, and applied direct for precipitating sewage and the like; or the soluble salts may be dissolved and separated from the residue, and applied in like manner. "Besides treating the flue dust with sulphuric acid or with hydrochloric acid, it may be fused with bisulphate of soda."

Sewage precipitants may also be obtained by mixing manganiferous flue dusts, containing higher manganese oxides, with crude sulphates or chlorides of iron in about equal proportion. The action of the higher oxides of manganese, when mixed with wet sewage sludge, is described in Eng. Pat. 21,635, 1894 (this Journal, 1895, 983).—E. S.

Filtering Material and Process for Making the Same, An Improved. F. Jurschina, L. Schnitzer, and F. Hirsch, all of Vienna. Eng. Pat. 6934, March 30, 1896.

See under I., *page* 315.

Purifying Water by Boiling it in Open Vessels, Method and Apparatus for. A. Dervaux, Brussels. Eng. Pat. 24,474, Nov. 2, 1896.

The water is boiled in an open vessel by means of steam admitted near the bottom, the steam being continually condensed before it reaches the surface of the water, by admitting a fresh supply of the water to the vessel. The water may be admitted either at the top or the bottom of the vessel, the purified water being drawn off through a pipe at the side.—R. A.

(C.)—DISINFECTANTS.

Ferrous Sulphate, Employment of, for the Destruction of Cryptogamic Parasites of the Vine. Croquevielle. Comptes Rend. 1897, **124**, [8], 418–419.

The following treatment to be applied to the vine in the winter is recommended as a preventive of the maladies known as *black rot, oidium, mildew, anthracnose,* &c.:—1. The stems are washed or sprinkled with a solution containing at least 10 per cent. of sulphate of iron. 2. Powdered sulphate of iron is applied to the soil in quantities varying from 500 to 1,000 kilos. to the hectare, according to the nature of the soil.—A. K. M.

Formaldehyde, Methods for the Estimation of. G. Romijn. Zeits. Anal. Chem. **36**, [1], 182.

See under XXIII., *page* 366.

PATENT.

Albumin and Protein Bodies, Production of Formaldehyde Compounds of the. Dr. A. Classen, Aix-la-Chapelle, Germany. Eng. Pat. 7712, April 11, 1896.

By the action of formaldehyde, or of substances capable of yielding it, on albumin or protein bodies, at a temperature of 110°—130° C., compounds are formed which are quite innocuous and are gradually decomposed by tissues and secretions, giving off formaldehyde. For instance, 50 c.c. of purified albumin or blood serum are mixed with 50 c.c. of a 40 per cent. formaldehyde solution and evaporated on a water-bath until the mass appears dry. It is then heated in a digester for 24 hours to 120° C., finely powdered, stirred up again with formaldehyde solution, and treated a second time in the digester. The product obtained is finally heated for some time to 120°—130° C., after which it is freed from formaldehyde by washing with water. The products are said to possess a durable disinfecting action and animal blood, when treated therewith, even after standing several days at 40° C., maintains a light red tint and remains perfectly odorless.—T. A. L.

XIX.—PAPER, PASTEBOARD, Etc.

Paper-making, The Results of Grinding Fibres for. W. Herzberg. Mitt. k. t. Versuchsanst. zu Berlin, **14** [5 and 6], 306—308.

As is generally known, fibres, especially linen, when ground in the beating engine with blunt blades and dense stuff, are so crushed that it is almost impossible to state their origin. This grinding is done mostly in order to obtain strong writing paper, cigarette paper, &c., the pulp being known as "greasy." In blotting- and similar papers a greasy pulp is carefully avoided, the grinding being performed with sharp blades, and with thin stuff so as to separate the fibres without crushing them.

The appearance of the fibres is different in different cases, owing to the various methods and degrees of grinding.

An examination shows that the fibre is ground to such a degree, in some cases, as to make it difficult to find any whole filament.

By the use of numerous micrographic reproductions, it would, no doubt, be possible to obtain normal standards of grinding, which would be of great utility to paper-makers, as it would be possible to compare results without taking a micro-photograph each time.

In the table following, are given the strength of the papers compared with those of the fibres:—

Name of Paper.	Composition of Pulp.	Middle		Resistance against Crumpling.
		Breaking Length.	Stretching.	
		M.	Per Cent.	
Dutch bank-note paper	Cannot be ascertained with certainty on account of grinding. Possibly linen.	7,275	11·3	Extremely strong.
Normal I	Linen, some cotton fibres, rather strongly ground.	6,215	4·7	Very strong.
Document paper made from unbleached rags.	Linen, and a little cotton	8,425	5·6	Extremely strong.
Normal I	Linen and cotton	6,050	4·2	Very strong.
"	Cotton, with a little linen.....................	6,825	7·2	Extremely strong.
Photographic paper...........	Linen, with very little cotton	2,600	3·8	Weak.
" "	Linen, with very little cotton	4,350	4·5	Weak.
Wrapping paper	Manilla hemp	6,750	4·5	Extremely strong.

The three normal papers were made in different mills.
—S. P. E.

PATENTS.

Paper, Process for Increasing the Suppleness or Pliability of. O. Tietze, Ratingen, near Düsseldorf, Germany. Eng. Pat. 1497, Feb. 28, 1896.

Hard paper is rendered pliable and more suitable for concert and other programmes, &c., by impregnating it with soap made from castor oil, or a mixture of castor and other oils, and resin. The paper is soaked in, or brushed over with a strong aqueous solution of the soap, and dried. By varying the relative proportions of fatty oil and resin, various degrees of suppleness may be produced.—L. A.

Fabrics from Paper Pulp, Impts. in the Manufacture of. C. Brodbeck, Paris. Eng. Pat. 23,598, Dec. 10, 1895.

See under V., *page* 327.

Alkali-Cellulose, Impts. in the Manufacture of. C. F. Cross, London. Eng. Pat. 4713, March 2, 1896.

The cellulose fibre or pulp is treated for some time with dilute hydrochloric or sulphuric acids, until the cellulose

is in a brittle condition. It is then washed, and treated in an edge-runner with caustic soda, in such proportions as to give a compound containing 40 to 50 per cent. of cellulose, 10 to 12 per cent. of caustic soda, and 38 to 50 per cent. of water. The product is then in a pulverulent condition, favourable for treatment with carbon bisulphide for the preparation of thiocarbonate. It has approximately the composition $C_6H_{10}O_5NaOH$.—S. P. E.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

Lovage, Essential Oil of. E. Braun. Archiv. der Pharm. **235**, 1.

Lovage oil is found to contain (*a*) a terpene, $C_{10}H_{16}$, resembling limonene, but not giving crystalline compounds with the halogen acids; (*b*) cineol, $C_{10}H_{18}O$; (*c*) isovalerianic acid; (*d*) acetic acid, as an oxidation product; and (*e*) benzoic acid. The oil commences to boil at 170°, but begins to decompose at 200° C.—J. O. B.

Belladonna, Alkaloidal Value of. Kremel. Pharm. Zeit. **41**, 686.

An examination of the individual parts—roots, stem, leaves, and fruit—of a belladonna plant, gathered in July, and bearing some unripe fruit and flowers, resulted as follows:—

The root gave	1·750 per cent. of alkaloid.
The stems gave	0·616 „ „
The leaves gave	0·700 „ „
Unripe fruits gave	0·600 „ „

These results apply to dry material.

The fluorescent substance, chrysatropic acid, was also obtained from all parts of the plant, by washing the acid extracts with chloroform. Subsequently, a part of the fresh root remaining over from the above analysis, was dried at 100° C., losing 81 per cent. of water, and was then examined for its alkaloidal percentage. This last was found to be 0·95 per cent.—a fact indicating that 45·71 per cent. of its alkaloids had been decomposed in the process of drying; a piece of the same root upon re-examination after having been kept for the space of one year, yielded only 0·70 per cent. of alkaloid—a further loss of 26·26 per cent. However, the decomposition of alkaloids brought about by keeping the drug, would seem not to go beyond a certain limit. Samples kept for a year showed on an average between 0·50 and 0·70 per cent., others kept for eight years 0·60 per cent., and still others, in the form of powder, kept for a period of ten years also showed 0·60 per cent. of alkaloids.

A solid extract of belladonna with 2·60 per cent. of alkaloids was kept for a space of three years, at the end of which time the amount of alkaloids was found to be 2·45 per cent.

With a view of ascertaining the relative percentage of alkaloids contained in belladonna roots gathered at different periods of the year, specimens were gathered in June and October from the same spot. After drying, both were examined for the quantity of alkaloid, as well as for that of extractive matter. The former yielded 0·88 per cent. of alkaloids, and the latter but 0·225 per cent. That gathered in June gave 26·6 per cent. of solid extract, assaying 3·32 per cent. of alkaloids; that in October gave 16·6 per cent., with but 1·30 per cent. of alkaloids.

—J. O. B.

Apiol. W. Dulière, Jonas, and François. J. Pharm. Chim. 1897, **5**, 229.

Very dissimilar substances are sold under the name of apiol, varying in density, colour, action of solvents and reagents, and action of heat, which decomposes adulterated apiol, the true apiol being volatile. Essential oil of parsley contains apiol and a terpene in varying proportions; according to Dulière some essences contain no apiol. Jonas states that whilst the density of apiol, according to the Belgian Pharmacopœia, is 1·07—1·08, that of the French product is much lower. The property of apiol of sinking in water does not serve for the detection of impurities, a sample mixed with an equal volume of olive oil behaving, in this respect, exactly like pure apiol.

François has studied the properties of apiol prepared in the following manner:—1 kilo. of ground parsley fruit was twice macerated with 1 litre of 92 per cent. alcohol for 24 hours, the alcoholic solution shaken with 100 grms. of animal charcoal for 24 hours, and filtered; then one quarter of the liquid was distilled off, the remainder being mixed with its own weight of chloroform, and left to settle for 24 hours. After filtering, half the liquid was distilled off. The residue was warmed at 100°—110° C. until chloroform and alcohol were driven off, and to the cooled liquid one-eighth of its weight of litharge was added, the mixture digested for 24 hours, and filtered through a thick layer of animal charcoal. Thus obtained, apiol is a complex product, on fractional distillation 5—6 per cent. of a light yellow oil of strong odour and pungent taste passing over at 150°; this crystallised on cooling, and is the German "pure crystallised apiol." At 170°, a fatty oil of slight odour and bitter taste, and non-crystallisable, distils over. According to François, this is apiol. At about 190°, a brown-coloured mixture of resinous substances passed over.

The apiol of commerce is generally coloured green by chlorophyll, due to imperfect purification, or use of ether instead of chloroform. The principal adulterants are castor oil, linseed oil, glycerin, and gurjun balsam. The density is 1·08 at 15° C.; it is insoluble in water and benzene, gives a cloudy solution in alcohol; is soluble in ether and chloroform, and very soluble in acetic acid. If apiol be allowed to run down the sides of a tube into nitric acid, at the point of contact a blood-red coloration is produced, turning to rose and then to yellow.—A. C. W.

Citral, Action of Alkalis on; Methylheptenone Preparation of. A. Verley. Bull. Soc. Chim. 1897, **17**, 175.

By boiling with weak alkalis, citral is decomposed into acetaldehyde and methylheptenone; the latter substance may thus be readily prepared from the citral of lemon-grass oil. 500 grms. of citral, 500 grms. of potassium carbonate, and 5 litres of water were boiled for 12 hours with a reflux condenser; acetaldehyde is evolved during the whole course of the reaction, and may be collected by allowing the condenser to become slightly warm and attaching a receiver cooled by ice. When the operation is over the product is steam-distilled, and the crude ketone, on fractionation *in vacuo* with a distilling column, separates into methylheptenone (five-sixths) and unaltered citral. The ketone is formed quantitatively, but the aldehyde is partially resinified; only traces of organic acids were left in the flask.

$$(CH_3)_2C{:}CH.CH_2.CH_2.C(CH_3){:}CH.CHO + H_2O = CH_3.CHO + (CH_3)_2.C{:}CH.CH_2.CH_2.CO.CH_3.$$

Methylheptenone prepared by this method is identical with that obtained by Wallach, by Tiemann and Semmler by the careful oxidation of citral, and with that extracted by Barbier and Bouveault from lemon-grass oil. The citral extracted from lemon-grass oil by means of sodium sulphite contains 10 per cent. of methylheptenone; this may be separated by fractionation in a vacuum with a distilling column, but has not the same odour or boiling point as that prepared by the above method; by transformation into the oxime, purification of this by distillation *in vacuo*, and subsequent decomposition, the methylheptenone is obtained quite pure and boiling at 168° C.—A. C. W.

Anethol and its Homologues. C. Moureu and A. Chauvet. Comptes Rend. 1897, **124**, [8], 404—406.

The authors describe a modification of Perkin's method of synthesising anethol, and the preparation of two homologues of the latter, *viz.*, parabutenylanisol and para-isopentenylanisol. The odour of aniseed is characteristic of all these compounds, and is also possessed by an isomer of butenylanisol obtained by Perkin; this property is attributed to the para position of the two side chains and to the peculiar unsaturated character of the hydrocarbon chain, $- CH = CH - R$. —A. K. M.

Essences, Artificial, Ionone and Hawthorn. Tiemann. Le Goudron, 1, [2], 11.

1. *Ionone (Artificial Essence of Violets).*—Equal parts of citral and acetone are agitated with barium hydroxide solution for several days, the mass being then taken up by ether, which is afterwards decanted and the residue distilled, the fractions passing over between 138° and 155° (under 0·012 in. pressure) being collected. The unconverted acetone and citral are removed by steam and the remainder distilled under the same pressure, collecting the fractions boiling between 143° and 145°. 20 parts of this portion, *pseudo-ionone*, are heated to boiling, with 100 parts of water, 100 of glycerin, and 0·5 part of sulphuric acid. After cooling, agitating with ether, and evaporating the latter, the residue is distilled and the fractions boiling between 125° and 135° constitute *ionone* (sp. gr. 0·935).

2. *Artificial Essence of Hawthorn* is prepared by heating together, on the water-bath, paroxybenzoic aldehyde, methyl di-iodide, and a solution of potash in methyl alcohol. This essence boils at 218°, and its purity is best assured by the distillation test.—C. S.

Basil, Essential Oil of, Its Chemical Composition. Dupont and Guerlain. Comptes Rend. 1897, 124, [6], 300—302.

This oil is obtained by distillation from the leaves of *Ocymum basilicum L.*, a plant of oriental origin, but which has been acclimatised in the South of France. The fresh leaves furnish about 0·04 per cent. of the essence, or when dried about 1·5 per cent. Essence of basil forms a slightly oily, yellowish liquid, possessing a strong characteristic odour. Its specific gravity at 15° C. is 0·9154. It is levorotatory (− 7° 40′ in 100 mm. tube). On distillation about four-fifths of the oil pass over between 190°—220° C., leaving behind a viscous brown substance which retains the odour of the original oil. The volatile portion on fractionation yielded two main fractions: one constituting about 60 per cent. of the essence (195°—200° C.), consisting of *l*-linalool ($C_{10}H_{18}O$), identical with the linalool obtained from oil of lavender, &c.); the second (205°—215° C.) possessed an odour resembling that of estragol (para-methoxy-allyl-benzene), and was found to contain a large proportion of that body. The estragol, although not separable as such, was clearly identified by previous conversion into the isomeric anethol, and isolation of the latter.—H. T. P.

Turpentine, The Rectification of: Oil of. E. Kremers. Pharm. Rev. 1897, 15, 7.

For pharmaceutical purposes, oil of turpentine is usually required to be rectified. In a series of tables, the author reproduces the results of investigations dealing with this question. In some of the experiments, portions of crude oil (1,000 c.c.) of sp. gr. 0·872 at 20° C., and rotatory power $(\alpha)_D$ = + 11°·616 were agitated with an equal volume of milk of lime, and after an interval of at least 12 hours, distilled in a current of steam, 4 fractions of 200 c.c. each being collected, and the residual 200 c.c. rejected. The results obtained in one experiment are subjoined:—

Fraction.	Sp. Gr. at 20° C.	$(\alpha)_D$.
		°
1	0·864	+ 14·180
2	0·866	+ 12·297
3	0·8702	+ 11·429
4	0·8722	+ 10·700

The decrease in volatility is, therefore, accompanied by a diminution of rotatory power. In another similar experiment the whole (mixed) distillate was dried over anhydrous sodium sulphate. The oil possessed an agreeable odour, its sp. gr. was 0·864 at 20° C. $(\alpha)_D$ = + 12·177°. Rectification over milk of lime, in the manner described, results, therefore, in the production of an oil of excellent quality. At the same time it is pointed out that the rectified oil when stored in the customary way is subject to rapid resinification.—H. T. P.

Fennel, Commercial Varieties and their Essential Oils. J. C. Umney. Pharm. J. 1897, 54, 225.

The chief chemical and physical differences of the oils of various commercial varieties of fennel fruits are tabulated as follows:—

Source.	Percentage Yield of Oil.	Optional Rotation 100 mm.	M. Pt. after Solidifying.	Sp. Gr.	Per Cent. of Fenchone.
		°	°		
French sweet fennel	2·10	+ 16·0	12·5	0·976	None.
French bitter fennel	+	+ 16·5	11·7	0·980	"
Persian	1·70	+ 14·0	11·2	0·977	3·4
Indian	0·72	+ 21·0	8·2	0·968	6·7
Japanese	2·70	+ 15·5	10·0	0·975	10·2
Galician	4·40	+ 20·0	6·2	0·965	18·1
Russian	4·80	+ 23·0	4·4	0·967	18·2
Saxon	4·70	+ 22·0	6·1	0·974	22·5

The author finally giving it as his opinion that the Russian, Roumanian, Galician, Japanese, and Saxon varieties of the fruit, are best adapted for pharmaceutical use, with a preference for the last-named. The Indian fruits, which have been considerably used in this country, are thus shown to be of little value, owing to their richness in oil and the presence of fenchone in their oils.—J. O. B.

Peppermint Oil, Occurrence of Sulphur Compounds in American. C. Kleber. Pharm. Rev. 1896, 14, 269.

The author conclusively proves that methyl sulphide is an ever-present constituent of American oil of peppermint. It may be detected by distilling about 50 c.c. of the oil, an efficient condenser being used, until a few drops have passed over. The distillate, when agitated with mercuric chloride solution, yields a white crystalline precipitate. Other sulphur compounds besides methyl sulphide are probably present; for during the rectification of crude peppermint oil, a penetrating disagreeable odour may, after a time, be noticed, resembling that produced when organic sulphur compounds are decomposed by heat.—H. T. P.

Coumarin, Purification of. E. Claassen. Pharm. Rev. 15, 28; and Pharm. J. 1897, 58, 161.

Crude coumarin is placed in a capacious copper distilling vessel or a good Bohemian flask, petroleum spirit of low specific gravity added, and the liquid boiled for 5 or 10 minutes, the still being fitted with a reflux condenser. The liquid is then poured into a warm bottle, and on cooling, crystals of coumarin separate. The crude coumarin is again treated with petroleum spirit, and the process repeated until it is exhausted. Any coumarin remaining in the spirit may be removed by shaking out with 5 per cent. sodium hydrate solution, and subsequently precipitating with hydrochloric acid.—A. S.

Bromphenacetine. O. Hodurek. Ber. 1897, 477—480.

The Fabrik vorm. Hofmann and Schoetensack prepare bromphenacetine in the following manner:—To a solution of 100 grms. of phenacetine in 1 litre of glacial acetic acid, 250 c.c. of strong hydrochloric acid are added, and then gradually a solution of bromine in hot caustic soda until a permanent bromine reaction is obtained. On dilution with an equal volume of water the greater part of the bromphenacetine separates. Heated with concentrated hydrochloric acid bromphenacetine is converted into the hydrochloride of *o*-brom-*p*-phenetidine—

$C_6H_3(OC_2H_5)Br.NH_2HCl[OC_2H_5:Br:NH_2HCl = 1:2:4]$.

Heated with acetic anhydride, it is converted into the diacetyl compound $C_6H_3(OC_2H_5).Br.N(C_2H_3O)_2$.—A.C.W.

Guaiacum Resin. J. Herzig and F. Schiff. Ber. 30, 378—380.

The formula of the acid of this resin according to the authors, is $C_{18}H_{18}(OCH_3)_2(OH)_2$, whilst Doebner and Luecker only admit the presence of one hydroxyl group (Ber. 28, 866).—A. C. W.

Spanish Lavender, Essence of. E. Charabot. Bull. Soc. Chim. 1897, **17**, [6], 378—380.

The following are the results of the examination of samples of essential oil of Spanish lavender prepared in 1895 and 1896:—

—	1895.	1896.
Density at 15° C.	0·916	0·912
Rotation (100 mm.)	+ 16° 25′	+ 13° 20′
Esters, calculated as $C_{10}H_{17}.O.CO.CH_3$.	3·15 per cent.	3·4 per cent.
Free alcohol, calculated as $C_{10}H_{17}.OH$.	44·5 „	50·5 „

French oil of lavender is levorotatory and contains 35—40 per cent. of linalyl acetate.

After a series of fractionations, the author has separated borneol from the oil, this appears to be the principal alcoholic constituent.—A. C. W.

Chloral, Industrial Preparation of. A. Trillat. J. Pharm. Chim. 1897, **5**, 218—222.

The process is divided into three stages—preparation of chloral alcoholate, conversion of this into chloral, and formation of the hydrate.

Chloral Alcoholate.—In a large glass balloon 60 kilos. of alcohol, as completely dehydrated as possible, are subjected to the action of a current of chlorine, the escaping hydrochloric acid being absorbed by water. The introduction of the chlorine lasts a considerable time—in some works 10—14 days. At first the flask must be cooled; if the temperature be not properly regulated the yield is much reduced. After a time the action becomes less violent; the mixture is then warmed to 60° C., and finally to 100° C. When the liquid is completely soluble in water, the current of chlorine is stopped, and, on cooling, a white mass of the alcoholate is obtained. In this operation, Springmühl recommends the use of 1 per cent. of iodine as a chlorine carrier; Page recommends an addition of crystallised ferrous chloride. In the former case the ethyl iodide formed is easily recovered.

Chloral.—In the ordinary process the chlorinated liquid is not allowed to cool, and is transferred to an enamelled still holding 150—200 kilos., where an equal volume of strong sulphuric acid is added in small quantities. The temperature is raised to boiling, the condensed vapours being returned to the still. During ebullition there is an evolution of hydrochloric acid gas, the cessation of which indicates the termination of the decomposition of the alcoholate. Fractional distillation follows, this ceasing when the thermometer has risen to 100° C. The distillate is again rectified, the portion coming over above 94° C. being pure chloral.

Chloral Hydrate.—The chloral is shaken with the theoretical quantity of water, and the product of the reaction at once poured over a smooth surface, when it solidifies; in this state it is sent into commerce.

To obtain crystals, the warm mass (heat is disengaged in the reaction) is mixed with one-third its volume of chloroform, and the mixture let cool in closed vessels: crystallisation is complete in a week. The mother-liquors serve for a second crystallisation. The crystals are dried at the ordinary temperature.

Flackinges recommends crystallisation from warm carbon bisulphide; Martius uses benzene.

Other methods which have been described, differ from the preceding in that the chlorinated mixture, after solidification and pressure, is sublimed or distilled with calcium chloride. Chloral hydrate should be free from alcoholate, which has not the same physiological properties; and, warmed with twice its volume of water, it should give a clear solution, free from oily drops and not precipitated by silver nitrate. When carefully heated, it should volatilise completely, and the vapours should not be inflammable. Gently warmed with 3 vols. of strong sulphuric acid, the mixture should remain clear. On warming the hydrate of chloral with potash solution, turbidity is caused through separation of chloroform; the clear portion is decanted and iodine dissolved in potassium iodide, added. So soon as the liquid begins to turn yellow, it is cooled, when the presence of alcohol, and consequently of chloral alcoholate, is indicated by the formation of a precipitate of iodoform. Chloral hydrate may be quantitatively analysed by determining the amount of soda required to convert it into chloroform.—A. C. W.

Bismuth Iodogallate, Preparation of. J. de Pharm. d'Anvers, **52**, 326; and Pharm. J. 1897, **58**, 167.

30·4 grms. of subnitrate of bismuth are dissolved in 100 grms. of nitric acid, and to the solution 500 c.c. of warm water are added. To this is added, with constant agitation, a clear solution of 16·6 grms. of potassium iodide and 18·8 grms. of gallic acid in 300 c.c. of distilled water. The precipitate is collected, washed with a saturated solution of gallic acid, and dried.—A. S.

Hypo-iodous Acid and Hypo-iodites. R. L. Taylor. Chem. News, 1897, **75**, 97.

When a little alkali is added to an aqueous solution of iodine, there is practically no iodate formed, 90 to 95 per cent. of the iodine undergoing the reaction expressed by the equation $2KOH + I_2 = H_2O + KIO + KI$.

The solution bleaches indigo much more rapidly than chlorine or hypochlorites, but does not bleach litmus. It gives a precipitate with cobalt nitrate, which blackens on standing, and an immediate brown precipitate with manganous salts and lead salts. Stronger solutions are obtained by using iodine water containing a little suspended iodine. All the solutions are decomposed completely by boiling for three or four minutes. It is stated that Lunge and Schoch obtained by the action of iodine upon lime in presence of comparatively little water, a bleaching liquid which resisted a boiling temperature for hours, but the author concludes that their observations were only correct so far as they agreed with those of Schönbein.

Free hypo-iodous acid is formed by the action of precipitated mercuric oxide upon iodine water. The filtered (colourless) solution possesses only the feeblest possible bleaching properties, but the addition of a little alkali changes it at once into as strong a bleaching solution as Schönbein's. It was found that 40 to 45 per cent. out of a possible 50 per cent. of the iodine used, existed in the solution as hypo-iodous acid. With iodine water containing a little suspended iodine, a stronger solution is obtained, which soon decomposes, turning brown, owing to the liberation of iodine.—A. S.

Antipyrine (Dimethylphenylpyrazolone): Its Compounds with Phenols. G. Patein. Comptes Rend. 1897, **124**, [5], 233—235.

The conclusions arrived at are:—

1. Monomethylphenylpyrazolone does not combine with phenols or phenolic acids.

2. Since one of the two N atoms existing in the mono- and dimethylpyrazolones, is in each case linked in precisely the same way, the fixation of phenols by antipyrine must be a function of its second N atom.

3. For this reason, Meyer's view, according to which antipyrine is represented as a kind of betain, must be considered untenable, for a compound of this nature could not combine with phenols.—H. T. P.

Alkaloids, Determination of. E. H. Farr and R. Wright. Pharm. J. 4, 202.

See under XXIII., *page* 366.

Guaiacol, Determination of, by Demethylation. M. Adrian. Nouveaux Rem. **13**, 97.

See under XXIII., *page* 367.

Kola, The Caffeïne Compound in. J. W. T. Knox and A. B. Prescott. J. Amer. Chem. Soc. 1897, **19**, 63.

See under XXIII., *page* 366.

Acetone, Estimation of. L. F. Kebler. Amer. J. Pharm. **69**, 65; and Pharm. J. 1897, **58**, 161.

See under XXIII., *page* 367.

Acetanilide, Exalgine, Phenacetine, and Methacetine; Separation of, by Microchemical Tests. Schoepp. Pharm. Zeit. **42**, 106.

See under XXIII., *page* 361.

Citric Acid, A Synthesis of. W. T. Laurence. Proc. Chem. Soc. 1897, [176], 65—66.

Ethylic citrate was obtained synthetically by the condensation of ethylic bromacetate with ethylic oxalylacetate in the presence of zinc, as indicated by the following equations, in which $Et = C_2H_5$:—

(1) $COOEt.CH_2Br + COOEt:CH_2.CO.COOEt + Zn = COOEt.CH_2.C(OZnBr)(CH_2.COOEt).COOEt$;

(2) $COOEt.CH_2.C(OZnBr)(CH_2.COOEt).COOEt + H_2O = COOEt.CH_2.C(OH)(CH_2.COOEt).COOEt + ZnO + HBr$.

The yield of ethylic citrate is very poor, owing to other reactions proceeding simultaneously. To further confirm the formation of ethylic citrate, it was converted into the calcium salt of citric acid, and a substance obtained showing the characteristic properties of calcium citrate. The same salt was also obtained by heating the zinc compound formed in equation No. 1 with alcoholic potash and precipitating the calcium citrate from the hot solution.

The results were all confirmed by analysis. The above synthesis of citric acid seems to be of interest as being more direct and simple than the synthesis from sym. dichloracetone or from ethyl-γ-cyanaceto-acetate.

PATENTS.

Aceto-Derivatives of the Simpler Aromatic Amines, Process for the Manufacture of. R. W. James, London. From W. J. Matheson and Co., Ltd., New York, U.S.A. Eng. Pat. 304, Jan. 5, 1897.

Instead of obtaining the acetyl derivatives of aromatic amines, such as aniline, by heating these latter with acetic anhydride or acetic acid, the patentee proposes heating together a salt of the amine, such as the hydrochloride or sulphate, together with a salt of acetic acid. For instance, 129 kilos. of aniline hydrochloride and 82—84 kilos. of sodium acetate are heated for 12—24 hours to 125° C. under an inverted condenser in a vessel provided with an agitator. The melt is then poured into water, when the acetanilide separates out; or this product may be dissolved out by means of methyl alcohol or benzene, which leaves the inorganic salts behind. It is stated that it is desirable to recover by distillation the acetic acid and aniline which were not converted into acetanilide.—T. A. L.

Hydrocarbons [Camphor Substitute], such as Turpentine or the like, for the Production of Crystalline Substances, and Apparatus for Treating and Manufacturing same; Impts. in the Treatment of. H. B. McKenna, Stoke Newington, London. Eng. Pat. 3365, Feb. 14, 1896.

By the action of hydrochloric acid gas and air on American turpentine or other suitable hydrocarbon, crystalline substances are produced, which deposit in the apparatus described, and of which a drawing is appended. In order to purify the substance, it is distilled with steam and either pressed into cakes or sublimed like Japanese camphor. The crystals may also be placed in conical vessels, such as are used in sugar refining, and subjected to the operation known as claying, which consists in allowing a saturated solution of the camphor substitute to percolate through the crystals, by which means oil, moisture, or any soluble impurities are removed.—T. A. L.

Oxygen Gas, Impts. in Apparatus for making. A. Sweetser, West Dulwich. Eng. Pat. 6408, March 23, 1896 (see also this Journal, 1896, 213).

The improvements claimed include the introduction of a filter between the generator and outlet pipe or jet, to prevent clogging; the provision of automatic means for regulating the descent of the generator and the consequent exposure of fresh portions of the charge to the heat of the lamp. The generator and tubes are lined with non-oxidisable material or paint, and the charge may be coated with similar material or enclosed in a cover to ensure regularity of heating. Provision is also made for the sounding of an automatic signal when the charge is exhausted and the reservoir descends below a certain height; also for the opening of a safety valve when it is fully charged (to prevent excess pressure); and for lowering the flame, as the reservoir rises, and *vice versâ*.—C. S.

Amido-1-alphyl-2-alkyl-5-pyrazolones, Manufacture of New Derivatives of. O. Imray, London. From "The Farbwerke vorm. Meister, Lucius, and Brüning," Höchst a/M., Germany. Eng. Pat. 7709, April 11, 1896.

Hitherto the only derivative of amido-1-alphyl-2-alkyl-5-pyrazolone known, has been the 4-acetamido-1-phenyl-2-3-dimethylpyrazolone. New derivatives of 4-amido-1-alphyl-2-alkyl-3-methyl-5-pyrazolone and 1-*p*-amidophenyl-2-alkyl-3-methyl-5-pyrazolone are obtained by the introduction of alcohol and acid radicles. Thus, 1-*p*-amidophenyl-2-alkyl-3-methyl-5-pyrazolone or alkoyl derivatives thereof are obtained by treating the derivatives of 1-*p*-alkoylamidophenyl-3-methyl-5-pyrazolone having the general formula—

$$\begin{array}{l} C_2H_3O.NH.C_6H_4.N.C.OR \\ \quad\quad\quad\quad\quad\quad\quad\;\; | \quad \diagdown CH \\ \quad\quad\quad\quad\quad\quad\quad\;\; | \quad \diagup \\ \quad\quad\quad\quad\quad\quad\quad\;\; N:C.CH_3 \end{array}$$

with alkyliodides and subsequently with alkalis. The corresponding amido compounds are then obtained on hydrolysis with acids. All these new alkyl and alkoyl derivatives of amido-1-alphyl-2-alkylpyrazolone are basic compounds forming salts with acids. For example, 1-phenyl-2-3-dimethyl-4-dimethyl-amido-5-pyrazolone is obtained by heating 1 kilo. of 1-phenyl-2-3-dimethyl-4-amido-5-pyrazolone with an equal weight of methyl alcohol and 1·4 kilos. of methyl iodide for 1 hour to 90° C. After evaporating off the methyl alcohol, the base is liberated by an alkali, extracted with benzene, and crystallised from acetic ether and petroleum ether, and melts at 108° C. A number of other alkyl derivatives have been obtained from different 4-amido-1-alphyl-2-alkyl-3-methyl-5-pyrazolones, of which the melting points are given in the specification. By first methylating and subsequently benzoylating 1-phenyl-2-3-dimethyl-4-amido-5-pyrazolone, the 4-benzoyl-methylamido derivative is obtained (m.p. 137° C.). This, on hydrolysis, yields the 4-methylamido derivative, of which the picrate melts at 172° C. When *p*-acetylamidophenylhydrazine and ethylaceto-acetate (equimolecular weights) are heated in an alcoholic solution, evaporated, and heated to 140°—150° C., the product, on crystallisation from dilute alcohol, gives *p*-acetamidophenylmethyl pyrazolone melting at 197° C., which differs entirely from the product obtained according to Ger. Pat. 85,883, and has a different formula and constitution. The new product, when formed in an aqueous solution in presence of an acid, gives in addition the ethyl ether, which when anhydrous, melts at 132° C. The acetyl ether obtained by boiling with acetic anhydride or by shaking with acetic anhydride in presence of soda lye, crystallises in yellow laminæ, and melts at 161° C. By heating the ethylacetyl ether of 1-*p*-acetamido-phenyl-3-methyl-5-pyrazolone with methyl iodide in benzene solution, iodides of ammonium bases are formed, which, by the action of acids or alkalis, yield 1-*p*-acetylamido-2-3-dimethyl-5-pyrazolone. This compound, when heated on the water-bath with an excess of concentrated hydrochloric acid, is hydrolysed, and on subsequent neutralisation gives *p*-amidophenyl-2-3-dimethyl-5-pyrazolone, which is easily soluble in water, alcohol, and chloroform, but sparingly soluble in benzene; it melts at 210° C. It differs from the similar acetyl derivative in not giving a green nitroso compound with nitrous acid. The dimethyl derivative melts at 135° C., the benzoyl compound at 225° C., and the urethane at 196° C.

—T. A. L.

Oxy-phenoxacetic Acids, A New or Improved Process for Manufacturing. L. Lederer, Munich, Germany. Eng. Pat. 28,968, Dec. 17, 1896.

The method consists in reacting with hydrochloric or hydrobromic acid on alkoxylated phenoxacetic acids. Thus, 1 part of guaiaoxacetic acid is heated for several hours with 3 parts of concentrated hydrochloric acid, under pressure, to 100° C. On cooling, hydroxyphenoxacetic acid (pyrocatecholacetic acid) crystallises from the solution. It forms plates which melt at 139° C., and is converted on distillation into its anhydride, which melts at 58° C. The aqueous solution gives a dark blue colour with ferric chloride. The oxyphenoxacetic acids are said to possess "excellent curative properties."—T. A. L.

XXI.—PHOTOGRAPHY.

PATENTS.

Tetranitro Cellulose (Collodion Pyroxylin), Manufacture of Solutions of. E. Bronnert and T. Schlumberger, Mulhouse, Alsatia. Eng. Pat. 1858, March 28, 1896.

Although tetranitro cellulose is insoluble in strong ethyl alcohol, it becomes soluble as shown by Eder (Ber. 1880, **13**, 184) when small quantities of certain organic and inorganic acids and their ethers or salts are dissolved in the alcohol. The patentees prepare a solution of tetranitro cellulose by dissolving it in alcohol containing the following substances:—Free oxalic, citric, tartaric, lactic or levulinic acids; the salts of these acids, or of hydrochloric or alkylsulphuric acid with the alkalis, alkaline earths, &c.; also the ethers of these acids. The danger of drying tetra nitrocellulose may be avoided by treating the wetted material with a solution of calcium chloride, followed by pressing and drying and solution in alcohol as above described.—J. L. B.

Photographic Prints, Impts. in Chemical Compositions for Preparing the Surface of Suitable Material for. E. P. Schoenfelder and E. Kehle, Newark, Essex, New Jersey, U.S.A. Eng. Pat. 15,852, July 17, 1896.

Collodion is prepared by dissolving 300 grains of pyroxylin in 1 lb. of ether and 1 lb. of absolute alcohol. Of this collodion, $1\frac{1}{2}$ oz. are thoroughly mixed with the following:—10 drops of a solution containing 15 grains of chloride of gold, platinum, or allied metal in $1\frac{1}{2}$ oz. of alcohol (40 per cent.); 3 grains of an organic, *e.g.*, citric, acid in 3 drops of alcohol; 15 grains of silver nitrate in 50 drops of alcohol; $1\frac{1}{2}$ grains of a chloride, *e.g.*, strontium chloride, in 3 drops of alcohol; 2 drops of a softening chemical, *e.g.*, glycerin or castor oil; enough ammonia; 2 to 5 drops of gold bromide. This emulsion applied to the surface of any suitable material leaves a sensitive film, and photographs printed upon the film, when fixed with "hypo," do not require toning, as the latter effect is produced simultaneously with the printing.—L. A.

XXII.—EXPLOSIVES, MATCHES, Etc.

PATENT.

Explosive Compounds, Impts. in. A. V. Newton, London. From A. Nobel, Paris. Eng. Pat. 6431, March 23, 1896.

The object of the invention is to produce an explosive compound which will not act too violently on coal to be blasted, but cause a rending instead of a shattering action. The new powder is a kind of gunpowder, in which charcoal is replaced by a combustible giving a greater body of gas on explosion, and at a lower temperature, so as to be less liable to ignite any firedamp that may be present. Dextrin may be used in the substitution, and inert substances may be added, such as alum or sodium bicarbonate; or combustibles or oxidisers, in excess of their "chemical combustion proportions." A powder may be made by mixing and granulating 100 parts of potassium nitrate, 12 parts of sulphur, 75 parts of dextrin, and 40 parts of sodium bicarbonate. When a powder is wanted to be fired by detonative ignition, it may be made from 60 parts of potassium chlorate, 40 parts of dextrin, and 50 parts of sodium bicarbonate, granulated.

High explosives, such as picric acid or picrates, or ballistite, may also be incorporated with combustibles in excess, or with inert substances, and granulated.—E. S.

XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

Filter Pump, A New. J. Wetzel. Ber. 1897, 537.

In this pump the tube widens after the first constriction and then narrows, as is shown in Fig. 1. The water thus twice

Fig. 1.

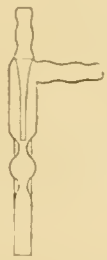
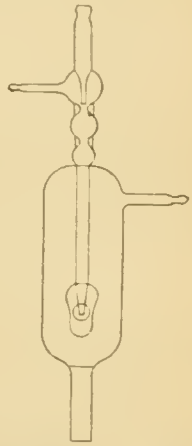


Fig. 2.



exerts its action. The current must be so regulated that the bulb between the two constrictions does not become filled with water. Using water at 5° C., a vessel of 3 litre capacity was evacuated down to a pressure of 7 mm. of mercury, in about one-third of the time and with the use of about one-third of the water required to produce the same effect with a pump of the old form. Fig. 2 shows the pump as constructed for evacuation and delivery of compressed air.—A. C. W.

Filter Paper, Acidity of. Maguier de la Source. Ann. de Chim. Analyt. 1897, **2**, 82.

Ordinary white filter paper is found to be very faintly alkaline. A paper 12 cm. in diameter was found to neutralise 0·0015 grm. of H_2SO_4. This, however, is not due to the combination of the acid with the cellulose, as stated by Ganz (Zeits. für angew. Chem. 1889, 669), but to traces of earthy impurities. French Berzelius paper is perfectly neutral. The Berzelius paper of Schleicher and Schüll, which has been extracted by hydrochloric and hydrofluoric acid, has a distinct acid reaction, equivalent for a 12-cm. paper to 0·0014 grm. H_2SO_4. Some Swedish Berzelius paper, 50 years old, had an acidity equivalent to 0·0003 grm. The same paper washed with very dilute HCl, and then with distilled water until the apparent removal of all trace of acid, washing being continued for 25 days, and the washed paper being dried over caustic potash, gave a decided acid reaction, equivalent to 0·0008 grm. of H_2SO_4 per filter; even after prolonged treatment with boiling water it gave an indication of acidity up to 0·0002 grm. It is thus evident that acid-washed paper always retains a trace of acid. The matter is of some importance in certain acidimetric operations, such as the determination of potassium bitartrate or other acid salts with a relatively high molecular weight.

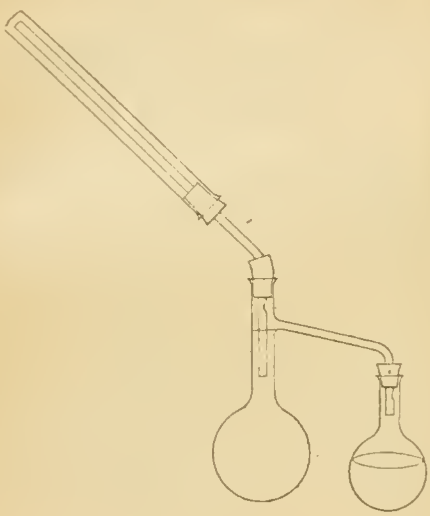
—J. O. B.

Steam-Distillation, Some Apparatus for. F. E. Matthews. Proc. Chem. Soc. 1897, [174], 18—19.

In this paper several forms of apparatus for automatically distilling substances by steam are described.

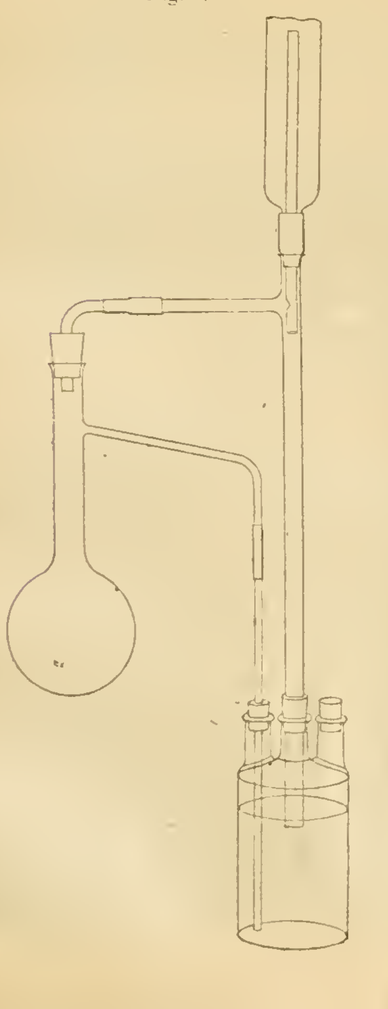
Some solids of high melting point may be separated by boiling the substance mixed with water in a flask fitted with a reflux condenser; the solid substance adheres to the inside of the condenser, whence it can be removed from time to time.

Fig. 1.



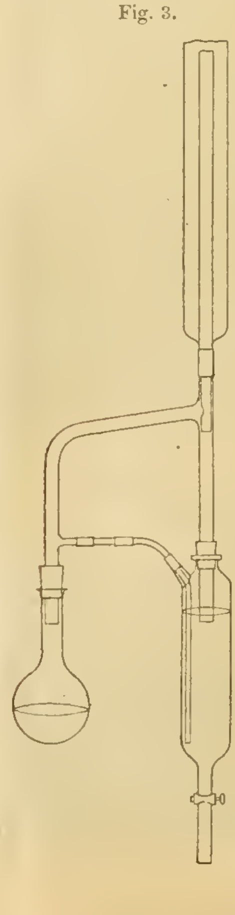
For liquids heavier than water, the flask in which the mixture is boiled is connected with the side tube of an ordinary distilling flask (see Fig. 1); this distilling flask, filled with water up to the side tube, serves as the receiver. Into the neck of the receiver, and passing below the surface of the water, a bent Liebig's condenser is fitted, which has the peculiarity of having a hole made in it a short distance above the level of the water in the receiver. On boiling the mixture in the flask, the vapours pass up the side tube of the receiver into the upper portion of its neck, and thence through the hole into the receiver, when condensation takes place. The condensed liquids run down the condenser to the water level in the receiver, where a drop of the heavy fluid sufficiently large to sink, is formed from time to time. The condensed liquids displace their own volume of water, which flows from the receiver through the side tube back again to the boiling-flask. In all cases in which vapour is passing in one direction in a tube, and water in the other, the advantage of perforating the tube near its lower end is pointed out.

Fig. 2.



For liquids lighter than water, the apparatus (see Fig. 2) consists of the boiling-flask, which is an ordinary distilling-flask; this is connected by the upper opening to an upright tube furnished with a ┳-piece. The top of the upright tube is connected to the condenser; the lower end dips two or three inches into a Woulff's bottle, containing water in sufficient quantity, which serves as the receiver. Through another neck of the Woulff's bottle, a second tube passes from the bottom of the bottle and is connected to the side tube of the boiling-flask. The mixed vapours pass from the boiling-flask into the upright ┳-tube, thence into the condenser; there becoming condensed, they fall down into the ┳-tube, producing a column of liquid which forces water from the bottom of the receiver back into the boiling-flask through its side tube.

Fig. 3.



A modification of this apparatus (see Fig. 3) dispenses with the necessity of having an india-rubber connection exposed to the hot vapour. In this modification the boiling flask is an ordinary plain flask. This is connected to the condenser by a side tube blown on to the upright tube. The water returns to the boiling flask through another ┳-tube, blown on to the side tube of the upright tube. For conveniently emptying the receiver without dismantling the apparatus, a separating funnel with two necks may replace the Woulff's bottle of the previous apparatus.

Many liquids bump badly when boiled with water; this can generally be avoided by introducing a zinc-platinum couple into the boiling-flask. The temperature of the water in the boiling-flask may be raised by dissolving suitable substances such as sulphuric acid or calcium chloride in it, or liquids of higher boiling point may be used.

—A. S.

Note on a Method for Determining Melting Points. E. H. Cook. Proc. Chem. Soc. 1897, [177], 74—76.

Notwithstanding the theoretical simplicity of taking a melting point, it is surprising that in commercial work considerable differences frequently occur between analysts when reporting upon such a substance, for example, as paraffin scale. Probably most, if not all, of these differences are caused by the different methods employed. Thus it is well known that the "English test," which consists in allowing the wax to solidify in a test-tube in which the thermometer is placed, gives results from $2\frac{1}{2}°$ to 3° F. lower than the "American test," in which the wax is melted in an open dish. Both these methods again differ slightly from the capillary-tube plan, and in this process a different result is obtained when an open tube is used than when it is closed. There are, in fact, many precautions which are necessary to be observed if concordant results are to be obtained, and it is much to be desired that some distinct and definite regulations should be made with reference to the matter.

The apparatus employed is a beaker filled to the brim with water; inside this, and separated from it on all sides, is a smaller one. The smaller beaker is partly filled with mercury, in which is placed a thermometer. A stirrer is used to keep the water in the large beaker of uniform temperature. A cardboard or other disc, covers the smaller beaker when the operation is in progress. The whole is heated from below by means of a sand bath. When the melting point to be determined is under 30°, it is better to replace the sand bath by an evaporating dish containing water.

The process is conducted as follows:—The material the melting point of which is to be taken, is placed on three or or four small pieces of thin ferrotype plate or other thin metallic sheet, or on the cover glasses which are used for microscope slides. If ferrotype or other metallic slips are used, care must be taken to remove the varnish or other coating in order that good metallic contact can be had with the mercury. The slips, with the substance on them, are now placed on the surface of the mercury and the heat applied until the substance melts. The solidifying point is obtained by raising the temperature above the melting point and allowing the beaker to cool, noting the thermometer when the first solidification takes place.

For temperatures between 100° and 200°, the larger beaker is filled with paraffin wax.

The following precautions have been found to be necessary:—(1.) The temperature must be made to rise very slowly. (2.) The liquid in the outer beaker must be frequently stirred. (3.) Not less than 2·5 cm. in depth of mercury must cover the inner beaker. (4.) Sufficient volume of water must be allowed between the two beakers. The minimum distances to give good results are 1 in. between them laterally and $1\frac{1}{2}$ in. at the bottom. (5.) The inner beaker must be immersed a sufficient depth in the water. This point is of great importance, the least distance between the top of the mercury and the top of the water being 3 in. A greater distance is, however, to be preferred. (6.) The whole apparatus should be protected from draughts. (7.) The disc should be kept on the smaller beaker during the determination.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Barium Thiosulphate for Standardising Solutions in Iodometry, Alkalimetry, and Acidimetry. M. Mutoianski. Zeits. anal. Chem. **36**, 220—221.

Barium thiosulphate is made by mixing hot highly concentrated solutions of 50 parts of sodium thiosulphate and 40 parts of barium chloride. The precipitate which forms is washed, first with lukewarm water, next with cold water, then with 95 per cent. alcohol, and finally with ether. It is then dried at the ordinary temperature on blotting-paper, and is ready for use; 266·7 parts of the salt are equivalent to 126·54 parts of iodine.

Its convenience for standardising iodine solutions depends upon the fact that an aqueous solution saturated at 17·5° C. forms a $\frac{1}{100}$ normal solution exactly. Such a solution deposits none of the salt on standing for some days at 15° C., although of course at lower temperatures its strength is altered. It is very quickly made by adding an excess of the barium thiosulphate to a quantity of water maintained exactly at 17·5° C., shaking for a quarter of an hour, and then filtering from the undissolved excess of the salt. It may also be used to standardise normal acid solutions (hydrochloric and sulphuric), and hence also for controlling normal alkali solutions. The equations which indicate its application to this purpose are—

$$KIO_3 + 5KI + 6HCl = 6KCl + 3H_2O + 6I.$$

$$KIO_3 + 5KI + 3H_2SO_4 = 3K_2SO_4 + 3H_2O + 6I.$$

The liberated iodine is determined by titrating with the $\frac{1}{100}$ normal solution of barium thiosulphate, using starch as indicator, and the strength of the acid is calculated therefrom.—H. S. P.

Thoria in Thorite, Estimation of. E. Hintz and H. Weber. Zeits. Anal. Chem. **36**, [1], 27–31.

Decompose 1 grm. of the thorite with concentrated HCl, evaporate to dryness, take up with 2 c.c. of HCl and water, filter off silica, and pass H_2S to remove Cu and Pb. If any precipitate obtained will not filter off clear, add a few drops of copper chloride solution, when the increased precipitate will filter perfectly. Expel H_2S by boiling, dilute to 200 c.c., and precipitate hot with 1 grm. of oxalic acid in solution. After two days' standing, filter, wash with water, and digest the precipitate several hours on the water bath with 60 c.c. of cold saturated ammonium oxalate solution. Dilute to 300 c.c., allow to stand two days in the cold, filter, and wash with water containing a little oxalate; heat the filtrate and add 5 c.c. of concentrated HCl. The residue which was insoluble in the ammonium oxalate should be digested a second, third, and even a fourth time with 20 c.c. of that reagent; filtered, and the filtrate acidified as above. After two days, filter off the thorium oxalate precipitated by the HCl, wash with acidified water, ignite, and weigh. As the precipitate always contains some cerium and yttrium oxides, dissolve by boiling with HCl; or fuse with $KHSO_4$, dissolve, precipitate with ammonia, filter and dissolve the hydrate in HCl. Evaporate to dryness, dissolve in water and a drop or two of HCl, dilute to 300 c.c. and boil for a few minutes with 3 to 4 grms. of sodium thiosulphate. Cool, filter, and add ammonia; filter, dissolve hydrates in HCl, evaporate to dryness, take up with a little water and precipitate boiling with concentrated solution of ammonium oxalate. Dilute and allow to stand some time in the cold, filter off the cerium and yttrium oxalates, ignite, weigh, and deduct from the weight obtained above to find the weight of pure ThO_2.

When the thorite is very impure, the repeated extraction of the oxalate precipitate with ammonium oxalate solution is insufficient. In that case ignite the insoluble residue and dissolve with concentrated HCl; or fuse, precipitate hydrates and dissolve the washed precipitate in HCl. Evaporate to dryness, take up with a few drops of weak HCl, dilute to 100 c.c., and precipitate boiling with thiosulphate. Dissolve the precipitate in HCl, drive off excess of acid by evaporation, precipitate with oxalic acid, and add this precipitate to the main ThO_2 precipitate. As the thiosulphate precipitation is not always complete, add ammonia to the filtrate obtained from it, dissolve the hydrates in HCl, evaporate to dryness, take up with water and precipitate boiling with concentrated ammonium oxalate. Dilute, allow to stand 24 hours, filter and precipitate the remainder of the ThO_2 with ammonia, adding this also to the main thoria precipitate.

The authors have examined the reactions upon which the scheme is based. They find that the precipitation of thoria in slightly acid solution by oxalic acid is practically perfect, cerous salts behaving similarly. With regard to the solubility of thorium oxalate in ammonium oxalate, C. Glaser has stated (this Journal, 1896, 675) that "ammonium oxalate precipitates thoria, the precipitate is soluble in excess on boiling, but re-precipitates on cooling." The authors' experiments show (1) that precipitated thorium oxalate is perfectly soluble in hot concentrated ammonium oxalate, the solution remaining clear after dilution and two days' standing; (2) that addition of hot, concentrated ammonium oxalate solution to thorium solution containing a little free

acid gives a clear solution which remains so on dilution and cooling even after standing several days; (3) that if one neutralises considerably a thorium solution, containing free acid, by ammonia, on adding concentrated ammonium oxalate to the hot solution it remains clear, but on standing for 12 hours it becomes opalescent and deposits some precipitate; but the most of the thorium oxalate remains dissolved. Only conditions (1) and (2) arise in the above scheme. The authors find the precipitation of thorium from its ammonium oxalate solution by means of HCl or ammonia is practically complete, and is more convenient than the more correct method of evaporating, expelling the ammonium oxalate by ignition, &c.—H. B.

Liquefied Ammonia, Testing Commercial. H. Bunte and P. Eitner. J. für Gasbeleucht. 1897, **40**, 174—175.

Owing to incorrect analyses, the impression has prevailed latterly that the liquefied ammonia sold in iron cylinders contains considerable quantities of water. Such could hardly be the case, as the gas, before compression, is thoroughly dried with caustic lime. The difficulties of testing liquefied ammonia were overcome in the following method:—A pipette of about 75 c.c. capacity, having a stop-cock on each stem, and at one end a smoothly-faced collar, was weighed. The cylinder containing the ammonia was placed valve lowermost, and to the outlet was attached by a union-joint a brass tube, the free end of which had a collar, on which was a leather washer, against which the collar of the pipette was firmly held by means of a detachable screw-clamp. Air was expelled from the pipette; the outer cock was then closed and the pipette two-thirds filled with the liquid ammonia, and the inner cock was then closed. The pipette was detached from the cylinder, and weighed, to ascertain the amount of ammonia taken. It was then placed upright, and the upper end connected to the first of a series of three weighed drying tubes packed with lumps of caustic potash. The outlet of the last drying tube dipped under mercury. By careful opening of the upper stop-cock of the pipette, the ammonia was allowed to slowly vaporise through the drying-tubes and mercury-seal. At the end of this evaporation, some drops of brown liquid remained in the pipette. The free end of the latter was attached to a caustic potash exsiccating apparatus, and when the coating of ice had gone from its exterior, the pipette was placed horizontally in an air-bath at 70°—80°, and a stream of dry air drawn through it for some time. Thus the volatile organic compounds and the water were collected in the weighed potash tubes, and a trace of brown organic substance, insoluble in ether and alcohol, but soluble in nitric acid and liquid ammonia, remained in the pipette. The impurities found in one analysis were:—

	Per Cent.
High-boiling organic substances left in the pipette...	0·08
Volatile substances resembling alcohol and water ...	0·41

—J. A. B.

Perchlorate, Estimation of, in Saltpetre. F. Winteler. Chem. Zeit. **21**, (1897), 75. (See also this Journal, 1897, 163.)

The methods for the estimation of perchlorate, which depend on the conversion of this, by heating, into chloride, are liable to error from three causes, and these errors may become serious when only small quantities of perchlorate are present. The errors which may be introduced are due, firstly, to volatilisation of unchanged perchlorate or of its resultant chloride; secondly, evolution of chlorine by the interaction of chloride and perchlorate; and thirdly, loss of chlorine due to the action of NO (arising when nitric acid is used to decompose accompanying chloride) on the perchlorate.

The author finds that the following method avoids these losses and leads to satisfactory results. The method, which depends on the observation that fuming nitric acid and its decomposition products quantitatively reduce perchlorates (and chlorates) by heating to a temperature of over 200° C. under pressure, is carried out by heating a given weight of the sample, together with an excess of silver nitrate and 10 c.c. of fuming nitric acid for 5 hours in a sealed tube at a temperature of 230° C., and weighing the resulting silver chloride. The author gives some analyses, made with pure potassium chlorate and perchlorate, to show the accuracy of the method, and also an analysis of a sample of potassium nitrate. 10 grms. of this sample yielded 0·0282 grm. of AgCl, equivalent to 0·27 per cent. of $KClO_4$. (The solution of this nitrate remained colourless after the addition of concentrated sulphuric acid and gave no precipitate with silver nitrate, *i.e.*, all chlorine was present as perchlorate). Chlorides, if present, are decomposed, according to Erck, by boiling with nitric acid (sp. gr. 1·14) and alcohol, whilst chlorates may be removed from a mixture of chlorate and perchlorate by evaporation with concentrated hydrochloric acid.—J. T. C.

Antimonio-Tungstates, and the Separation of Tungsten and Antimony. L. A. Hallopeau. Bull. Soc. Chim. 1897, **17**, 170—175.

Antimonic hydrate, prepared by precipitating potassium bimetantimoniate with hydrochloric acid, dissolves in a boiling solution of potassium paratungstate,

$$5K_2O.12WO_3.11H_2O;$$

the filtered solution deposits potassium antimoniotungstate

$$3K_2O.4WO_3.3Sb_2O_5 + 16H_2O \text{ or } 3KSbO_3.2WO_3 + 8H_2O.$$

The salt may be purified by recrystallisation, it is unaltered in air and loses $6H_2O$ at 100° C., and calcination decomposes it into potassium tungstate, acid antimoniate, and tungstic acid. The metallic salts give insoluble precipitates; from the silver salt by the action of hydrochloric acid and evaporation *in vacuo*, there is obtained a clear vitreous mass, $4WO_3.3Sb_2O_5.3H_2O + 8H_2O$, which does not lose water at 100° C.

The analysis of the compounds was effected by precipitating with mercurous nitrate, the mercury salt was filtered off, and the base determined as sulphate in the filtrate. The mixture of tungstic acid and antimony tetroxide formed by ignition of the mercury salt, was fused with a large excess of potassium cyanide (12 parts) in a porcelain crucible, according to the method given by Talbot (Chem. News, **22**, 229) for the separation of tin and tungsten. The fused mass was taken up with water, the metallic antimony collected on a tared filter, washed with water, finally with weak alcohol, and dried at 100°. The sodium or potassium sulphate, after weighing, should be fused with potassium cyanide; a small quantity of antimony is sometimes thus recovered. If, after the fusion, a small quantity of antimony adheres to the crucible and cannot be removed, the crucible is washed, dried, and weighed, a small quantity of ammonium chloride added, and, by heating to redness, the antimony is completely volatilised; the loss in weight represents the adherent metal. Results by this process are said by the author to agree exactly with determinations by precipitating as sulphide and converting into the tetroxide. The conversion of the metallic antimony into tetroxide is tedious; even by repeated treatment with fuming nitric acid the filter paper is not completely destroyed; in order, then, to prevent loss of antimony on ignition, ammonium bisulphate is added and the crucible slowly raised to a red heat.

It was not found possible to separate antimonic oxide and tungstic acid by fusion with caustic soda in a silver dish, on taking up with weak alcohol a certain quantity of sodium antimoniate always dissolving in the alkaline tungstate solution.—A. C. W.

Potassium, Estimation of: A Simplification of the Schweitzer-Lungwitz Method. Adolf Mayer. Zeits. anal. Chem. 1897, **36**, [3], 159—163.

In the Schweitzer-Lungwitz method of determining potassium in manures, &c., the sulphates are precipitated by means of a solution of barium oxalate in hydrochloric acid, any iron present oxidised with hydrogen peroxide, and ammonia added to alkaline reaction, so as to simultaneously precipitate the alkaline earths as oxalates, and iron and aluminium as hydrates. The author's objection to this is, that owing to the small solubility of barium oxalate in hydrochloric acid in the cold (1·7 per cent.), more than 800 c.c. of the solution must be added when, as in the case of superphosphates, 15 grms. of barium oxalate are required.

In the following modification (which was taken into consideration and rejected by Schweitzer and Lungwitz), the hydrochloric acid solution of barium oxalate is replaced by

barium chloride and oxalic acid. Twenty grms. of the substance under examination are boiled for 30 minutes with water, cooled, made up to 500 c.c. and filtered. Fifty c.c. of the filtrate are warmed and normal solution of barium chloride added from a burette so long as a precipitate results. An amount of normal oxalic acid equivalent to that of the barium chloride used is then added, the liquid boiled for 20 minutes, and made alkaline with ammonia. On cooling, it is made up to 100 c.c., allowed to settle, and filtered. Fifty c.c. of the filtrate are evaporated in a platinum basin and the residue ignited at as low a temperature as possible, in order to expel the ammoniacal salts. The ignited residue is dissolved in water, the solution filtered and the potassium precipitated by adding 10 c.c. of platinic chloride solution (containing 1 grm. of platinum and which is free from hydrochloric and nitric acids) and evaporating nearly to dryness; when cold, alcohol (80 per cent.) is added, and after standing for some time the precipitate is filtered off and dried at 120° C. The results obtained with this method agreed closely with those found by the ordinary method of estimating potassium. It is necessary, however, to boil the liquid for not less than 20 minutes after adding the oxalic acid, since with less boiling, the results are too low.

—C. A. M.

Aluminium and its Alloys, Analysis of. F. Jean. Rev. Chim. Industr. **8**, 5—8.

The influence on the physical properties of aluminium, exerted by the presence of various metals and metalloids renders the careful analysis of this metal and its alloys a matter of importance, and though Moissan (this Journal, 1896, 136) and Gouthière (this Journal, 1896, 230) have published complete methods, the author considers the subject as by no means exhausted. The method pursued by him consists in the first place in carefully attacking 10 grms. of aluminium borings by 10 per cent. hydrochloric acid in a flask and absorbing the evolved gases by bromine water in a Will and Varrentrapp bulb tube. When reaction has finally ceased, the bromine liquor—containing the sulphur, phosphorus, and arsenic of the substance,—is evaporated to drive off the bromine, and the sulphur is determined from the precipitate produced by barium nitrate. Arsenic is thrown down from the filtrate by H_2S, and weighed as arsenic acid after drying, treating with fuming nitric acid, and calcining. The residue is taken up with water and any insoluble antimonic acid is calcined, weighed, and deducted from the weight previously obtained. This allows the arsenic and antimony to be calculated.

The filtrate from the arsenical precipitate is freed from hydrogen sulphide by heat and treated with 5 c.c. of nitric acid and 10 c.c. of concentrated ammonium nitrate, for the determination of phosphoric acid by the Pemberton method.

The hydrochloric acid solution of the metal is diluted and passed warm through a tared filter, and the residue, after washing with boiling water, is weighed and dried, the loss in weight on calcination indicating carbon. The remaining residue is fused with potassium and sodium carbonates and potassium nitrate; taken up with water; the solution acidified with HCl, and evaporated and calcined to estimate silica. The insoluble portion is calcined, dissolved in aq. regia and united to the original solution, which is then saturated with hydrogen sulphide to throw down the insoluble sulphides. In the absence of tin and antimony, the precipitate is dried, calcined, treated with nitric acid and a few drops of sulphuric acid and diluted with water and alcohol, the lead being determined as sulphate, and the copper thrown down by sodium hydroxide. When tin or antimony is present, yellow ammonium sulphide is added to the mixed sulphides and the soluble sulphides are determined in the ordinary way.

A colourless filtrate from the hydrogen sulphide reaction indicates absence of nickel and chromium, in which case the liquid is concentrated to 100 c.c. and silica estimated from one-half this quantity, by evaporation in presence of potassium chlorate. It is always advisable to fuse the silica with alkali carbonates, and separate anew, to ensure the absence of alumina. Iron is determined from the same solution, after the removal of silica, by adding 25 to 30 c.c. of HCl, and titrating with stannous chloride.

The second 50 c.c. are reserved for estimating zinc and manganese, to which end the liquid is made strongly alkaline with soda and boiled to dissolve the alumina, the manganese being thrown down by bromine, and the zinc titrated with sodium sulphide.

When chromium is present it is determined, in company with iron, in a separate portion of substance, 1 grm. of which is attacked by caustic soda assisted by heat until the evolution of hydrogen ceases. The liquid is then evaporated and fused with potassium nitrate and sodium bicarbonate, taken up with water and filtered, the insoluble oxides being dissolved in dilute sulphuric acid; and, after reduction of the solution by zinc, the iron is determined by titration. Chromium is then estimated colorimetrically from the same solution made up to 50 c.c., standard solutions of potassium chromate being employed for comparison.

Nitrogen, which is said to be present in some samples of the metal, is estimated by acting on 10 grms. with caustic soda solution of 36° B., passing the evolved hydrogen through 10 c.c. of decinormal acid in a Schlœsing nitrogen apparatus, and determining the ammonia by titration with alkali of equal standard.

For sodium, Moissan's method is the most convenient, modified by converting the sodium nitrate formed into sulphate, digesting with concentrated ammonium carbonate, and, after calcination and treatment with 10 c.c. of barium hydrate solution, re-evaporating to dryness with more ammonium carbonate, and re-dissolving in boiling water—titrating the sodium as carbonate by the aid of decinormal acid. This modification prevents error arising from the incomplete insolubility of aluminium, &c. during the calcination of the nitrate. Any alkali contained in the barium hydroxide must be ascertained and deducted from the total obtained.—C. S.

Copper; Analysis, Quantitative, of Commercial. E. Murman. Monatsh. f. Chem. 1896, **17**, 697—731.

Estimation of Copper as Sub-Sulphide.—The introduction of a small proportion of hydrogen sulphide in the hydrogen used during the ignition of the copper sulphide was found to give more latitude both as to the time and temperature required. But in this case the crucible must be heated to a bright red throughout, or the results will be considerably too high; they will always be higher than those with pure hydrogen. The use of either pure hydrogen sulphide, carbon dioxide, or carbon monoxide, in place of hydrogen, also gives high results. It is therefore best to heat the sulphide in pure hydrogen for from 10 to 15 minutes in such a way that the crucible is surrounded by the flame, and to repeat the heating until the weight is constant, adding only a very small fragment of sulphur before re-igniting, and taking care that the mass is raised uniformly to a dull red-heat. Analyses made in this way gave numbers ranging from 99·996 to 100·023 per cent. The precipitated copper sulphide is apt to contain traces of alkali, lime, and silica, especially if it should have been treated with an alkaline sulphide to remove antimony and arsenic. 3 mgrms. of alkali have thus been separated from 2·5 grms. of sulphide. It may be completely removed by heating the sulphide precipitate in the funnel in an air-bath, and then washing it with hydrogen sulphide water, prior to drying and igniting as usual. The lime, which is derived from the containing vessel, is not so easily removed; but it causes only a very minute error. The silica must be separated, after weighing, by treating the precipitate with nitric acid. A little iron and nickel may also be present, and it is preferable to adopt Hampe's thiocyanate method for the separation of the bulk of the copper (see this Journal 1894, **13**, 421).

Estimation of Total Oxygen.—The author prepares the sample, if of copper plate, by cutting it into pieces 6—8 mm. square, or if in fragments, by shearing it into pieces 4—6 mm. in diameter, and 10—20 mm. long. The sample is next treated with benzene or chloroform to remove fat, and dried on a towel, which is then sprinkled with sea-sand, and rubbed over the copper until the surface of the metal is perfectly bright (about 2—3 minutes). The sand is then thrown away, and a fresh quantity is

applied in the same way; this is finally removed and the copper is weighed immediately. Larger fragments may be cleansed by filing before applying the solvent for the grease. The pieces are then heated in hydrogen in a bulb tube, or preferably in a boat enclosed within a straight glass tube. A single wash-bottle, containing caustic potash and a little potassium permanganate, and a calcium chloride tube suffice for the purification of the hydrogen. A flask containing alkaline lead solution is unnecessary, as this reagent was found to be incapable of retaining such traces of hydrogen sulphide as may be present in the gas. If a bulb tube be employed, it should be 25 cm. long by 10—12 mm. wide (internal measurement), with a bulb 5 cm. in diameter, and should be fairly thick in the walls. The boat is preferable because a higher temperature may be applied and the operation may therefore be shortened. The best results are obtained by determining the loss of weight undergone by the copper; but for this purpose the bulb-tube, if used, should not be weighed with the specimen, because it is certain to lose weight on heating: copper filings, however, cannot be weighed separately, for some of them will fuse into the glass. It is preferable not to attempt to weigh the water produced by the action of the hydrogen, as this necessitates the use of a very elaborate purifying apparatus for the hydrogen, to ensure that it is entirely free from air. It is found that thick pieces of copper are practically as rapidly attacked by the hydrogen as thin fragments are; and as the errors of the method are mainly due to surface oxidation, it is desirable to employ pieces of appreciable size rather than sheet or filings. Thin sheet must be heated in the bulb tube for at least an hour to a clear red heat, and thick pieces for two hours, followed by a second heating for half an hour by way of control. A portion of the total oxygen, probably that which is not combined with copper, is removed with greater difficulty than the remainder. Reduction in carbon monoxide was found to be slower, but it failed to throw any light upon the difference in the condition of the different portions of the oxygen.

Determination of the Cuprous Oxide.—The silver nitrate method is found to give results which are so high that the process should never be employed, except in the treatment of crude copper containing much suboxide, in which case the error is relatively small.

Estimation of Lead.—Precipitation of lead as sulphate by evaporation with sulphuric acid and dilution, gives high results, owing to the presence of silica and bismuth in the precipitate. It is best to treat the cold nitrate or sulphate solution with sufficient hydrogen sulphide solution to precipitate a few centigrams of copper; the finely-divided precipitate is filtered off and will be found to contain the whole of the lead, which is estimated electrolytically, after filtering off the precipitate and (without washing) dissolving it in nitric acid. As little as 0·0003 per cent. of lead has thus been detected in a sample of copper, which gave no indication by the sulphate precipitation method. At least a part of the antimony and bismuth is thus concentrated with the lead.

Estimation of Antimony and Arsenic.—A large quantity of the copper is introduced into a bulb-tube, and is heated to clear redness for at least two hours in pure hydrogen, the bulb being covered with a hood of asbestos-card, and being turned round from time to time to prevent the deposition of antimony upon any part of the bulb itself. A portion of the antimony is deposited as a mirror in the straight part of the tube, and the escaping gases are passed through a Peligot tube containing hydrochloric acid and a little bromine. If the antimony be weighed as oxide, it is necessary, after once heating the precipitate, to treat it with cold water, which dissolves out fixed alkalis, and then to dry and re-ignite it before weighing. The tetroxide is not volatile at a red heat in the absence of reducing gases, and it is therefore recommended to pass air into the crucible during ignition. The method of weighing the tri-sulphide, after heating in carbon dioxide in a Gooch crucible, may give incorrect results owing to oxidation from access of air through the bottom of the crucible. The precipitate should, therefore, be heated in hydrogen sulphide gas after the carbon dioxide treatment.

Determination of the Copper.—From 1·2 to 1·5 grm. of the copper is dissolved in warm dilute nitric acid, one or two drops of hydrochloric acid and a distinct excess of sulphuric acid are added, and the solution is evaporated until all nitric acid is expelled. After dilution, the silver chloride and lead sulphate are filtered off, and washed with water containing sulphuric acid, and the copper is precipitated by hydrogen sulphide and weighed as cuprous sulphide. With the precautions named in the first section of this abstract, the result should be correct to within 0·03 per cent. The sulphide may be rapidly filtered by employing a filter paper 3 cm. wider than the bottom of the funnel, pressed on to a porcelain funnel with perforated bottom, with the aid of a truncated wooden cone; this is used in conjunction with a filter pump. Alcohol may be used for the last washing to expedite the drying.

The electrolytic estimation, as commonly used, is apt to give slightly incorrect results, partly owing to irregularities in the successive weighings of the platinum dish, which may show variations of 3 or 4 mgrms. A little water may also be retained in the deposited copper. Better results are obtained if the dish with its deposit be heated in hydrogen to a red heat; but trouble is thus given in the subsequent cleansing of the dish, owing to the superficial alloying of the copper and platinum.—W. G. M.

Chromium, Determination of, in Ferrochrome and Chromium Steel. Spüller and Brenner. Chem. Zeit. 1897, **21**, [1], 3—4.

The method of Spüller and Kalman (this Journal, 1894, 867, 950) has been modified as follows:—

1. To lessen the amount of sodium hydroxide needed (and the consequent inconvenience from its contained water) the substance to be fused is mixed with sodium hydroxide and a layer of sodium peroxide placed on the top. On fusion, the peroxide mixes gradually, and the action does not become violent. The rest of the peroxide is then added to the fused mass.

2. At the end of the fusion a further quantity of peroxide is mixed in, which greatly facilitates and quickens the solution of the cooled mass in water.

3. Chromium steels are dissolved in hydrochloric acid, and the chlorides converted by evaporation with sulphuric acid into sulphates. This gets over the difficulty of dissolving tungsten steels in sulphuric acid.

4. With chromium steels containing little chromium, the final titration is based on Zulkowsky's method (iodine and thiosulphate), not on Schwarz' method (permanganate).
—J. T. D.

Phosphatic Slags, Mechanical Analysis of Basic. H. W. Wiley. J. Amer. Chem. Soc. 1897, **19**, 19—22.

Experience has shown that the ammonium citrate method is of varying value, a lower solvent action of the acid ammonium citrate being noticed when the sample contains, in addition to tetracalcium phosphate, tricalcium phosphate, and particles of silicious slags and of iron and steel. It therefore seemed desirable to effect a mechanical separation, using sieves for particles $\frac{1}{10}$ mm. in diameter and upwards, and separating smaller particles by levigation. As water cannot be used, from its solvent action on the free lime which the slag contains, 90 per cent. alcohol was used. Photographs are given showing the uniformity in size of the grains of the various separates. Analysis reveals a regular progression in the richness and availability of the sample from the coarsest to the finest parts. The data lead to the inference that it may be advantageous to the manufacturer of basic slags to secure a separation for the purpose of being able to place on the market a more concentrated and available fertilising material.—L. J. de W.

Ozone, Atmospheric; Estimation of, on Mont Blanc. M. de Thierry. Comptes Rend. 1897, **124**, [9], 460—463.

After having made qualitative tests with various ozone papers in 1894, 1895, and 1896, the author in 1896 made several quantitative analyses of the amount of ozone in the atmosphere about Mont Blanc. The method employed was that of Albert-Levy, in which the air is aspirated through three tubes containing 20 c.c. of a mixture of potassium

arsenite solution and potassium iodide solution free from iodate, the arseniate formed being determined by means of iodine, and the oxygen which has effected the change calculated from the result and multiplied by three.

The subjoined table gives the results obtained at Chamonix (altitude, 1,053 m.), and Grands-Mulets (altitude, 3,020 m.).

—	Temp.	Barom.	Volume of Air corrected.	Ozone in 100 c.c.
		Mm.		Mgrms.
Chamonix, August 23, 1896	20	680	386	8·5
" " 24, "	19	684	417	3·9
Grands-Mulets. September 1, 1896.	21	739	315	9·4

The corresponding amount of ozone, as determined in the Montsouris observatory in Paris, was 2·0 mgrms. on August 23 and 1·9 mgrms. on September 1. From these figures the conclusion is arrived at that the amount of ozone in the air increases with the altitude.—C. A. M.

Gold or Auriferous Mineral, Method for Estimation or Extraction of. E. Serrant. Comptes Rend. 1897, **124**, [9], 480.

See under X., page 334.

ORGANIC CHEMISTRY.—QUALITATIVE.

Magenta and Magenta S, Distinction between, by Schiff's Reaction. P. Cazeneuve. Bull. Soc. Chim. 1897, **17**, 196.

A REPLY to Lefèvre (this Journal, 1896, 922), who maintains that Magenta and Acid Magenta, when decolorised by sulphurous acid, give similar colorations with aldehydes; this is also stated in Lefèvre's "Traité des Matières Colorantes," pp. 1063 and 1065. The author made similar solutions of 200 c.c. of water, 30 c.c. of magenta solution (1 in 1,000), 20 c.c. of sodium bisulphite of 34° B., and 3 c.c. of sulphuric acid of 66° B. 10 c.c. of this solution made with ordinary magenta give, rapidly, an intense violet coloration with one drop of formal or 15 per cent. acetic aldehyde; and much more dilute solutions give the same reaction. In the case of the solution made with Magenta S, no coloration was seen; and the same result was obtained with one drop of 40 per cent. aldehyde. The colour only appears on adding a large excess of aldehyde, but with pure alcohol the original rose tint reappears, and not the violet given by ordinary magenta with aldehydes. Moreover, a solution of 49 c.c. of 50 per cent. alcohol and 1 c.c. of 40 per cent. formal gives a less intense coloration with the Acid Magenta than is given by pure alcohol.

Acid Magenta is not a suitable reagent for the detection of aldehydes (see Paul, this Journal, 1897, 265).—A. C. W.

Colour Reactions yielded by Tartaric and other Organic Acids. E. Piñerua. Comptes Rend. 1897, **124**, [6], 291—292.

THE reagent employed is a solution of β-naphthol in strong sulphuric acid (0·02 grm. in 1 c.c. of H_2SO_4). About 0·05 grm. of the substance (*dry*) to be examined is treated in a porcelain dish with 10—15 drops of the solution. The following reactions are obtained:—

Tartaric Acid.—Coloration, *blue*, changing to an intense *green* on application of heat. When the mass is diluted with water (15—20 volumes), a permanently *reddish-yellow* solution results.

Citric Acid.—Coloration, *blue*, *not* affected by heat. On addition of water, a *colourless*, or *slightly yellowish* liquid is obtained. In presence of an admixture of tartaric acid, the green tint readily appears; and with 10—12 per cent. of the impurity, the coloration is dull blue-green.

Malic Acid.—Coloration, *yellow-green*, which becomes *bright yellow* on heating, and *bright orange* on dilution. This reaction is very delicate. Other organic acids yield similar reactions, but not sufficiently distinctive to be of value. With alkaline *nitrites* (2—3 drops of a solution), the reagent yields a very intense *red* coloration, which remains unchanged on addition of water. A solution of resorcinol (0·1 grm. in 1 c.c. of H_2SO_4) may be similarly employed to distinguish *nitrates* from *chlorates*. The former yield a *red-brown* product, which gradually assumes an intense *violet* tint, forming with water, an *orange* solution. With the latter, an intense *green* coloration is obtained, passing to *brown* on dilution.—H. T. P.

Colza Oil, New Colour Reaction of. Palas. Ann. de Chim. Analyt. **1**, 431; Bull. Assoc. Belge des Chim. **10**, [9], 358.

A ROSE coloration, gradually increasing in intensity, is produced when colza oil is agitated in the cold with an equal volume of rosaniline bisulphite (prepared by mixing, without heat, 30 c.c. of a 1 per cent. solution of magenta, 20 c.c. of 34° B. sodium bisulphite, 200 c.c. of water, and 5 c.c. of H_2SO_4). The reaction is very delicate, sufficing for the detection of 2 per cent. of colza oil in olive oil. Linseed oil gives a bright yellow coloration, but no reaction occurs with olive, sesame, cotton, arachis, castor, rape, nut, or poppy oil, or with the fatty acids of colza oil.—C. S.

White Wines, Testing for Coal-Tar Colours in, and the Difference between these Colours and Caramel. A. d'Aguiar and W. da Silva. Comptes Rend. 1897, **124**, [8], 408—410.

THE authors do not agree with the results recently published by Magalhães (see this Journal, 1897, 156), and they find that caramel, when added to a wine, can be distinguished from the coal-tar colours. The wine is rendered alkaline by the addition of ammonia, and is then shaken up with amyl alcohol. The colour taken up by the latter is noted, and also a dye trial is made with a little silk. The amyl alcohol becomes but faintly coloured if caramel only is present, and the fixation of the latter by silk is also very feeble. In the case of wines to which coal-tar colours have been added, the silk generally becomes distinctly dyed, and where this is not the case, the colouring matter (*e.g.*, chrysoidine, amido-azobenzene) can be identified by other reactions.
—A. K. M.

Vanillin, Test for. Pharm. J. 1897, **58**, 167.

To the solution under examination, add a few drops of a 1 per cent. solution of ferrous sulphate, and then bromine water, drop by drop. Vanillin gives a bluish-green colour, turning yellow after a time.—A. S.

Legal's Reaction, The Generalisation of. G. Denigès. Bull. Soc. Chim. 1897, [6], 381.

THE author has shown in a former communication (Bull. Soc. Chim. **15**, 1058) that the coloration given by acetone with sodium nitroprusside on the subsequent addition of an alkali and acetic acid, is common to all compounds containing the acetyl group (substituted or not), if the carbonyl group be united to certain other groups. Von Bittó (Annalen, **267**, 372) had previously shown that the reaction does not affect the aldehyde or ketone groups, and he had attributed it to the presence of the groups $CH_n.CO$ and $CH_n.CO.H$. The author's attention has now been drawn to this statement, which he considers too sweeping, since he has found that if the carbonyl be united to OH, OR.OM, NH_2, or Cl, the reaction does not take place.—A. C. W.

Acetanilide, Exalgine, Phenacetine, and Methacetine; Separation of, by Micro-chemical Tests. Schoepp. Pharm. Zeit. 1897, **42**, 106.

THE mixture is dissolved in the least possible quantity of concentrated hydrochloric acid and the analysis conducted in the following systematic manner:—

A. A little saturated solution of sodium bromide is added to a drop of the solution, and one crystal of potassium chlorate. An amorphous precipitate results after about a minute: one drop of 20 per cent. alcohol is added. Numerous needles separate = Acetanilide.

B.—1. Iodine, dissolved in potassium iodide, is added to a drop of the solution. An amorphous precipitate results, which should be treated immediately with a drop of water. The insoluble portion forms brown crystalline scales = Exalgine.

2. A small quantity of the dry mixture is placed in a drop of concentrated hydriodic acid. Brownish-red scales form immediately = Exalgine.

3. A small quantity of the dry mixture is placed in a drop of a mixture of saturated sodium iodide solution and gold chloride in hydrochloric acid. Brown four-cornered scales result = Exalgine.

C.—1. A drop of 20 per cent. alcohol and one crystal of potassium chlorate are added to a drop of the hydrochloric acid solution. After some minutes the mixture becomes somewhat turbid. Crystalline rosettes or stars separate = Phenacetine.

2. A little saturated solution of sodium iodide is added to a drop of the solution. Fine light yellow needles result after a few minutes = Phenacetine.

3. A little of the dry mixture is added to a drop of hydriodic acid. Light yellow needles form on the crystals which have not dissolved = Phenacetine.

D.—1. A little saturated potassium bichromate solution, or chromic acid, is added to a drop of the hydrochloric acid solution. Numerous cruciform crystals separate after a few minutes, sometimes only after heating = Methacetine.

2. A little concentrated ferricyanide of potassium solution is added to a drop of the solution and covered immediately with the cover glass. Yellow cubes separate = Methacetine.

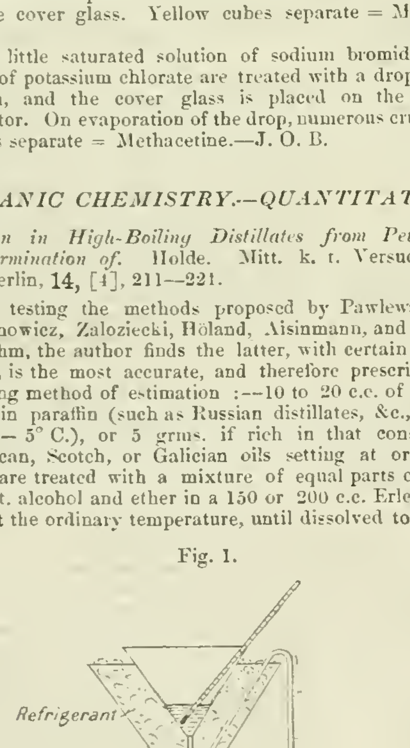
3. A little saturated solution of sodium bromide and a crystal of potassium chlorate are treated with a drop of the solution, and the cover glass is placed on the micro-exsiccator. On evaporation of the drop, numerous cruciform crystals separate = Methacetine.—J. O. B.

ORGANIC CHEMISTRY.—QUANTITATIVE.

Paraffin in High-Boiling Distillates from Petroleum, Determination of. Holde. Mitt. k. t. Versuchsanst. zu Berlin, **14**, [4], 211—221.

After testing the methods proposed by Pawlewski and Filemonowicz, Zaloziecki, Höland, Aisinmann, and Engler and Böhm, the author finds the latter, with certain modifications, is the most accurate, and therefore prescribes the following method of estimation :—10 to 20 c.c. of the oil, if poor in paraffin (such as Russian distillates, &c., setting below − 5° C.), or 5 grms. if rich in that constituent (American, Scotch, or Galician oils setting at or above 0° C.), are treated with a mixture of equal parts of 98·5 per cent. alcohol and ether in a 150 or 200 c.c. Erlenmeyer flask, at the ordinary temperature, until dissolved to a clear

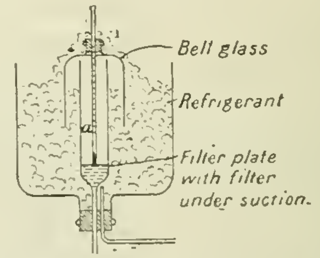
Fig. 1.



solution. The liquid is then cooled down to between − 18° and − 20° C., and a further addition of the alcohol-ether stirred in until no oil drops, but only solid paraffin flakes, remain in suspension, whereupon it is passed through a 9 cm. filter, previously cooled in the apparatus shown in Fig. 1, and the precipitate rinsed with a little cold (− 18° to − 20° C.) alcohol-ether (1 : 1, or, for soft paraffin 2 : 1), the temperature being kept throughout as far below − 15° C. as possible, especially in the case of soft paraffin, when it should average − 17° C. at the highest. The washing is discontinued when the solvent is found to leave on evaporation a trace of fat or paraffin-like (but not oily) residue. Should the process of freeing the paraffin from oil take too long, the filter is transferred to another funnel and the paraffin removed, by the aid of hot benzene, into a basin, and, after evaporating the benzene, rinsed with 4 to 5 c.c. of warm ether into a test glass, where it is precipitated by stirring with a double volume of absolute alcohol at about − 18° C. This reprecipitation is also necessary for oils containing much soft paraffin, since, in the ordinary process, the large quantity of washing liquid required, redissolves an appreciable proportion of the precipitate. The purified paraffin is finally rinsed into a glass basin with hot benzene and heated on the water-bath until the odour of benzene disappears; after drying for $\frac{1}{4}$ hour in the oven at 105° C. and cooling, it is weighed. Unnecessarily prolonged drying causes loss of paraffin by volatilisation.

The whole operation is complete in between an hour and an hour and a half, and the results—provided the prescribed temperatures, &c. be maintained—are regular to within 0·23 per cent. for hard paraffin and 0·33 per cent. for soft paraffin. For refrigerating the filter, the apparatus (Fig. 2)

Fig. 2.



devised by Normann, is considered suitable, since the paraffin can then be precipitated direct in the funnel *a*.—C. S.

Mineral Oils, Dissociation of Petroleum Acid Salts and the Estimation of Free Acids in. R. Zaloziecki. Chem. Rev. Fett- u. Harz Ind. **4**, [2] and [3], 25—27 and 36—38.

In view of the converse dissociative reactions exerted by petroleum on the compounds of petroleum acids with alkalis, in aqueous and alcoholic solution respectively, the author proceeded to ascertain whether the one would counteract the other when both solvents were simultaneously present, and thereby enable an accurate titration of the free acids in the oil to be effected.

To this end experiments were instituted with $\frac{1}{5}$ N, $\frac{1}{10}$ N, and $\frac{1}{20}$ N aqueous caustic soda, $\frac{1}{10}$ N alcoholic alkali, and mixtures of $\frac{1}{10}$ N aqueous alcoholic alkali in the proportions of 1 : 1 and 1 : 2; 50 c.c. of the alkali being agitated with 100 grms. of oil in a separating funnel for one minute (or three to five minutes in the case of viscous oils, unless warmed to increase fluidity), and, after separation of the layers of oil and water, titrating back the excess of alkali in 10 c.c. of the latter, with normal acid. Tested in this way petroleum distillates, allowed to acidify by spontaneous oxidation, gave the following results, expressed in c.c. of $\frac{1}{10}$N acid per 100 grms. of oil; $\frac{1}{10}$N NaHO, 15·25; $\frac{1}{20}$N NaHO, 15·12; $\frac{1}{10}$N NaHO (1 aq. : 1 alcoh.), 15·0; $\frac{1}{10}$N NaHO (1 aq. : 2 alcoh.), 15·75.

Parallel experiments showed the correctness of the above-named assumption respecting the use of 1 : 1 aqueous-alcoholic alkali.

With regard to the preparation of neutral petroleum for mixing with known quantities of free acid, it was found that freshly prepared distillate from oil previously refined with alkali rapidly became acid, owing to the formation of oxidisable bodies during distillation, and it was therefore found preferable to employ a sample a year old, neutralising this with an excess of 1 per cent. of alkali without redistillation.

In nine other instances with oils, either with or without added petroleum acids, the results showed that an indirect titration with $\frac{1}{10}$ N or $\frac{1}{20}$ N alkali, effected by the aid of an excess of lye titrated back, is a sufficiently concordant and reliable method. For lubricating oils of high viscosity, it is advisable to use the decinormal alcoholic-aqueous alkali, since alcoholic alkali gives results in excess of the truth, either on account of dissociation, the insufficiently accurate determination of the end point in the case of these highly coloured liquids, or by alterations in the amount of alkali, due to the solvent action of the alcohol on the oil.

Experiment showed that though hydrolysis occurs when the salts of petroleum acids are boiled, or when they are shaken up with oils, either free or nearly free from acid; the decomposition is comparatively insignificant in amount, not exceeding 0·5 per cent. of the total acid in $\frac{1}{10}$ N solutions.

Working on the same lines as Krafft (this Journal, 1894, 1207; 1896, 206 and 601), but substituting petroleum soaps for sodium palmitate and petroleum distillate for toluene, similar results were obtained; the dissociation ranging from 0·5 to 0·88 per cent. of the original acid, and being therefore negligeable in acidimetric determinations.—C. S.

Petroleum Oils, &c., Testing; Recent Experience in. Holde. Mitt. K. t. Versuchsanst. zu Berlin, **14**, [4], 229—237.

1. *Resinification of Mineral Oils.*—In continuation of the experiments on the behaviour of these oils when exposed in thin layers to the air (this Journal, 1895, 891), samples spread out thickly were heated to 100° C. for about 7 hours daily, with the following results:—Colourless, resin-free, "paraffin" oil (from the lowest lubricating oil fractions) gradually volatilised by the end of 15 months without leaving any trace of resinous residue; the thicker "motor" oil from the higher fractions left a small solid residue, consisting partly of original resin and partly of oxidised hydrocarbons; a very thick Russian oil volatilised only slightly and resinified but little, especially when previously extracted by 75 per cent. alcohol. On the other hand, lubricating oils (Russian and Oelheim) containing residuum, resinified considerably, and the first-named, which contained the larger proportion of low-boiling fractions, volatilised more rapidly and left a larger residue than the other Russian oil referred to above. In each case the residue was imperfectly soluble in petroleum spirit (although some of the original oils were completely so), but was almost totally dissolved by benzene. These results are parallel with those obtained in the previous series.

2. *Quantitative Estimation of Water in Fatty Oils.*—Experiments were made with various fatty oils, respecting the advantage of checking by a blank experiment, the loss of weight due to volatilisation of oil in determining the percentage of water by the evaporation test, though the figures obtained show that a fairly accurate estimate can be made without this check, still it is safer to make the parallel test in the case of all fatty oils and saponifiable fats. Where the amount of water is large, it is advisable to reduce the proportion by mixing with a known weight of oil perfectly free from water. To make the estimation, 10 to 20 grms. of oil are heated in a glass basin over a boiling water-bath, with continued stirring, until froth ceases to form on the surface, the ascending bubbles of steam being directed, by the aid of the stirrer, to the side of the basin and there dispersed. Another sample of the oil, previously freed from water, is heated concurrently, and the amount of water is determined by the difference between the losses of weight sustained by both.

3. *Vaselin Oil "Soluble in Water."*—A sample of this oil (an ammoniacal soap dissolved in mineral oil) was submitted to examination with regard to its property of protecting cast iron and steel against the action of water, and for comparison in this respect with the soft-soap solutions generally used for milling and cutting machinery. With this object small polished cast-iron and steel plates were immersed in water containing 2 and 5 per cent. of the oil, and also in 1 and 2 per cent. solutions of soft-soap, for three weeks. The effect of the oil was to preserve the steel from all corrosion, the surface being merely covered with a faint black deposit, and the loss in weight restricted to 0·2 per cent., whereas with soap solutions the loss amounted to between 9·9 and $14\frac{1}{2}$ per cent., and, on removal of the deposit, the surface was found to have become dulled. The cast iron was not so well preserved, having lost between 6·2 and 7·6 per cent. in weight, and parted with its lustre, in the oil and water mixtures, but was less corroded than in the soap solutions, where the loss in weight amounted to 13·8 and 17·5 per cent., and a copious brownish-yellow deposit was formed, especially at the points of contact between the plate and the vessel. The working tests on the machinery, were made with 2 and 4 per cent. emulsions of the oil in water and 1 to 2 per cent solutions of soft-soap. Here also the superior protection afforded by the oil was manifested by iron and steel, the latter being free from corrosion and the former merely coated with easily removable brown flakes, whilst with soap the steel was slightly, and the cast-iron considerably, rusted.

It was also found that the presence of from 2 to 10 per cent. of this oil lowered the freezing point of water by 3° to 5° C., but no further reduction could be confirmed on the addition of 1 per cent. of glycerin to the 2 and 5 per cent. solutions, although with 0·1 to 0·5 per cent. of glycerin, they more frequently remained liquid at −5° C. than without this addition.—C. S.

Lards, Determination of Solid Fats in Compound. G. F. Tennille. J. Amer. Chem. Soc. 1897, **19**, 51—54.

The mechanical process adopted in the United States laboratory for the determination of the constituents of compound lards as described by Wainwright (this Journal, 1896, 620), cannot be relied upon under all conditions.

From a large number of tests of lards of known composition, 10 are selected containing cotton-seed oil of varying body, and both hard and soft stearin, but all having 20 per cent. of oleo-stearin to 80 per cent. of cotton-seed oil. Although some of the results are within $1\frac{1}{2}$ units of the true percentage, others are 7 or 8 per cent. too high or low.—L. J. de W.

Cocoa Butter, The Iodine Number of. D. Holde. Zeits. anal Chem. 1896, **36**, [3], 163—164.

The higher limit for the iodine value of cocoa butter has been given as 51, and this led Strohl to examine a large number of specimens of cocoa butter (this Journal, 1896, 362), and Strohl's figures were in turn criticised by Filsinger (this Journal, 1896, 747). The author now finds that de Negri and Fabris made a mistake in the transcription, the iodine value given by Hübl being 34.—C. A. M.

Fatty Acids [Stearic Acid, &c.], Determination of Unsaponified Fat in Commercial. F. and J. Jean. Rev. de Chim. Ind. 1896, **7**, [83], 338—339.

The following method is given:—The sample is melted, thoroughly washed with hot water, dried at 100° C., and titrated directly (5 grms.) with $\frac{1}{2}$ N alcoholic potash and phenolphthalein. Another portion of the sample is completely saponified, the resulting soap decomposed by an acid, the fatty acids washed, dried, and a known weight titrated with standard alkali. The average equivalent of the mixed fatty acids is thus found, from which, and the results of the preceding titrations, the absolute composition of the sample may be deduced. For the examination of samples contaminated with tarry decomposition products, and containing fatty acids soluble in hot water, the following scheme is suggested:—10 grms. of the sample (dried at 100° C.) are washed with boiling water until the washings pass off perfectly neutral. From the wash-water the fatty acids are extracted by means of a mixture of ordinary and petroleum ethers, and weighed after evaporation of the solvents. The portion of sample insoluble in water is dissolved in alcohol, exactly neutralised with soda, and the solution evaporated to dryness. The residue is ground up with an excess of sand and fibrous asbestos, and extracted in a Soxhlet tube with ordinary and petroleum ether. The residue remaining when the ethereal solution is evaporated,

consists of tar and undecomposed fat. It is weighed, then saponified by soda, and again extracted (in aqueous solution) with the mixed ethers. The extract on evaporation leaves as residue the tar alone.—H. T. P.

Shellac, Behaviour of the Resin Acids of, in Separating Fatty and Resin Acids by Gladding's and Twitchell's Processes. F. Ulzer and R. Defris. Zeits. anal. Chem. **36**, [1], 24—27.

The resinous acids of shellac behave quite differently from colophony (resin) acids, in Gladding's process; thus a dark-coloured sample of shellac, containing 0·05 per cent. of unsaponifiable matter, gave only 13·76 per cent. of "resin acids" by this process. The silver salts of the acids differ from those of resin by being mainly insoluble in ether, resin silver salts being soluble. A mixture of 51 parts of colophony, containing 12·9 per cent. of unsaponifiable matter and 49 parts of the resin acids of the above-mentioned shellac, yielded 48·64 per cent. of "resin acids." The shellac used had an acid number of 65·43, and a saponification number of 204·78. A sample of commercial lac, composed of colophony and shellac, yielded only 63·7 per cent. of "resin acids" by Gladding's process.

Examined by Twitchell's process, the dark-coloured shellac gave 66·56 per cent. of "resin acids," the petroleum-ether layer containing an ester—a light yellow, transparent, resinous mass having the saponification number 199·5. A determination of its acid number gave a negative result. An orange-coloured sample of shellac, having an acid number of 53·05 and a saponification number of 201, gave by Twitchell's method, 72·89 per cent. of "resin acids." These results show that the shellac acids in part react as the fatty acids do, forming esters on treatment with HCl gas in alcoholic solution.

Samples of Angola-copal and Kauri-copal gave respectively 86·01 and 86·37 per cent. of "resin acids" by Twitchell's process. Gladding's process could not be used, as part of the soda-soap of the acids separated out from the alcoholic solution on adding sodium hydrate solution, and the addition of the ether caused a further precipitation.
—H. B.

Tannin, Estimation of. [*The Tannoform Reaction.*] E. Aweng. Apoth. Zeit. 1896, 831.

The author has examined, as to its value for quantitative purposes, the well-known reaction between tannin and formaldehyde, whereby insoluble condensation products (tannoforms) are produced. Experiments were made with solutions of oak-bark tannin (1—25 per cent.) in water. He shows by a series of experiments that the tannoform reaction is unsuitable for quantitative work.—H. T. P.

Gelatin, Volumetric Method for the Estimation of, in Glue and Glue Liquors. F. Jean. Rev. de Chim. Ind. 1896, **7**, [83], 339—340.

One grm. of the sample is soaked in cold water, then dissolved by gently heating the liquid in a water-bath, and diluted to 100 c.c. at 35° C. To 10 c.c. of this solution are added 10 c.c of a 1 per cent. solution of tannin, 5 grms. of salt, and 1 grm. of sodium bicarbonate, the mixture well shaken, rapidly filtered, and the precipitate washed with salt water (23° B.) until the filtrate measures 45 c.c. The excess of tannin is then determined by slowly adding to the filtrate a solution of iodine (4 grms. per litre) until a drop of the liquid yields with dry starch powder (thinly spread on filter paper) a blue stain. The liquid is now diluted to 60 c.c., and titration continued until a very faint end-reaction is obtained. The *titre* of the iodine solution being known (by titration against 0·01 grm. of tannin dissolved to 60 c.c.), the weight of tannin consumed in precipitating the gelatin is found by difference. According to the author, 100 parts of tannin = 88·5 of gelatin.

Since even the purest commercial tannin procurable, frequently contains impurities, the actual strength of the sample used must be found by the "hide" method; and the standard solution prepared so as to contain 1 grm. of *real* tannin per 100 c.c.—H. T. P.

Beetroot, Method of Analysis of the. L. Jesser. Bull. de l'Assoc. des Chim. de Sucr. 1897, **14**, 684—688.

It is a well-known fact that the polarisation of the beetroots received is always higher than that of the fresh slices, and factories which pay for the beets on their sugar content are obliged to take this factor into account. The reason of the discrepancy is not yet very clear; the author, however, can personally confirm this difference, which reaches, one year with another, 1 per cent., notwithstanding conscientious sampling and every care in analysis. Attention is called to one source of error: the division of the beetroot, and, consequently, the apparatus for the division. The taking of the sample and rasping it are made after the two following principles:—(1.) The sugar content of the beetroot depends on its size. Roots of medium size are richest in sugar, the richness diminishing towards the two extremes. For this reason, samples are taken of every size to represent the lot. (2.) The sugar is not regularly distributed in the beetroot. The heart is richer in sugar than the periphery. For conversion into pulp the beetroot is cut lengthwise in half, and then into quarters, and rasped. In this way an equal portion of each root passes into the sample, and the average sugar content results very fairly. But experience has shown that the polarisation of the juice thus obtained gives erroneous results, and gradually the method has been arrived at of testing the sugar directly. For this purpose, machines are necessary by which it is possible to obtain a suitable pulp or cream. These machines may be classed in two groups: those which subdivide a part of the root prepared by hand, and those which remove a portion—of cylindrical form—in the form of cream. The only good ones are those which cut out segments, as then, from each root, great and small, a portion is obtained proportional to its size. A good machine ought to furnish only a small quantity of pulp, by taking from each root only a small segment, in order that a large number may be included in the sample. The cream should be tested without delay; a loss of 1 per cent. has been observed in a cream kept in a closed vessel for three hours. The sugar is tested by one of the methods of digestion, or by alcoholic extraction, the testing by the juice should be absolutely condemned. Digestion always gives higher results than polarisation of the juice. If pulp already pressed be subjected to a second subdivision and pressure, a juice of a richer quality and of higher quotient is obtained than at the first pressing.—L. J. de W.

Beetroot, Determination of Sugar in the. K. Kaiser. Bull. de l'Assoc. des Chim. de Sucr. 1897, **14**, 688—691.

The difficulty of estimating the true sugar content of a lot of beetroots is attributed, not to the methods of analysis, but to the difficulty of sampling. Of 1,000 roots grown in proximity, the mean richness, 15·5 per cent., was shown by only 25, or one-fortieth of the lot; the others varying from 18·2 to 12 per cent.

In estimating the sugar in any sample of roots, it is important to take, as far as possible, a quantity of substance from each root proportional to its size. If the diameters of three beetroots are as 4 : 5 : 6, their volumes are as 64 : 125 : 216. To obtain equal proportions of pulp, the one of diameter 4 should be pierced once, the one of 5 twice, and the one of 6 three times. By a table the author has drawn up for beets of all sizes, and a scale placed near the perforator to show the number of perforations to be made, he obtains regularly 3 per cent. of pulp for analysis.—L. J. de W.

Sugar in Beetroot, Comparison of Direct Methods of Estimating. J. Graftiau. Bull. Assoc. Belge des Chim **10**, [9], 354—357.

On comparing the de Molinari, Masson, cold digestion and alcohol-extraction methods with that of cold digestion of an arbitrary volume, the author finds that as a rule the latter gives results inferior to the other direct methods, the deficit decreasing, however, if the digestion be prolonged. Instantaoeous digestion he characterises as chimerical, the results obtained in the cold depending for their approximate accuracy on the compensating effect of two errors: one an

excess, due to imprisoned air, and the other a deficit, caused by insufficient diffusion; but digestion in the warm tends to eradicate both. For cold digestion *in vacuo* a sufficient time for complete diffusion is necessary to ensure accuracy.—C. S.

Beetroot Juice, Estimation of Lime in, by Alcoholic Soap Solution. N. Rydlewski. Bull. de l'Assoc. des Chim. de Sucr. 1897, **14**, 696—699.

In view of the importance of the organic lime salts present in beetroot juice, and to obtain a quicker method of estimation than that of incinerating, dissolving the ash, and precipitating by ammonium oxalate, the author has prepared a soap solution on Müller's formula. It is standardised by barium chloride, of which 100 c.c. represent 0·0207 grm. of BaO or 0·0075 grm. of CaO. To carry out the test, 10 c.c. of juice or 5 c.c. of syrup are poured into a 300 c.c. flask, making up to the mark with distilled water. The soap solution is then run in drop by drop from a burette until a froth is obtained lasting five minutes. From the number of c.c. of soap solution used, the lime may be calculated. Tables are given of results obtained.—L. J. de W.

Starch, Estimation of, in Frozen Potatoes. D. Saare. Zeits. f. Spiritusind. 1897, **20**, 51—52.

The estimation of starch in hard frozen potatoes is conveniently carried out by taking rather more than 5 kilos. of the sample and thawing in lukewarm water, continually replenishing the water until all the earthy particles have been removed. 5 kilos. of the drained potatoes are weighed, and the tare under water determined by a Reimann's balance. A deduction of 1 per cent. is made from the starch found, as a correction for errors of manipulation.—J. L. B.

Liquorice Extract (Cakes, Pastilles, &c.), The Analysis and Composition of. G. Py. J. de Pharm. et de Chim. 1897, [6], 280—284.

Moisture.—10 grms. of the sample are dried for 7 hours at 100° C.

Ash.—Total, and that soluble in water, determined in the 10 grms. that have served for the estimation of water.

Alcoholic Extract.—2 grms. are dissolved in about 30 c.c. of warm water, and to the solution, when cool, absolute alcohol is added until the mixture contains 75 per cent. of alcohol by volume. After 12 hours the precipitate is filtered off and washed with alcohol of 75 per cent. The filtrate is evaporated in a tared flask, the residue dried at 100° C., and weighed.

Glycyrrhizin (Ammonium Glycyrrhizate).—The alcoholic extract (from previous determination) is dissolved in warm water and precipitated by dilute sulphuric acid (1 in 10). The precipitate is washed with acidulated water, redissolved in strong ammonia, and the solution evaporated in a tared dish, dried at 100° C., and the residue (*ammonium glycyrrhizate*) weighed.

Organic Matters Insoluble in Alcohol are found by deducting the sum of the moisture, ash, and alcoholic extract percentages from 100.

The above determinations, as a rule, suffice to indicate the quality of commercial liquorice extracts. A more complete examination would include the determination of *total nitrogen* by Kjeldahl's process (the N derived from glycyrrhizin is found by multiplying the latter by 0·0593), and of *reducing sugars*. For the latter purpose the acid filtrate from the glycyrrhizin precipitate may be employed. *Gelatin*, being often added, either fraudulently, or to impart suppleness to liquorice sticks, &c., should always be looked for. To this end the portion of sample insoluble in 75 per cent. alcohol may be dissolved in water and tested with tannin, phosphomolybdic acid, picric acid, &c. Pure liquorice gives negative results with these reagents. The above liquid likewise serves for the detection of *gum*, the presence of which is indicated (1) by a white precipitate obtained on addition of three drops of copper sulphate (1 in 10) and 10 c.c. of potash solution (45° B.) to a little of the liquid, or (2) by the usual ferric chloride and chalk test. If desired, gelatin may be detected in the original sample by dissolving 5 grms. in water and saturating the solution with ammonium sulphate. The precipitate of gelatin and glycyrrhizin is washed with ammonium sulphate solution; subsequently with 75 per cent. alcohol, which dissolves out the glycyrrhizin. The residue is dissolved in water and examined for gelatin. Admixtures of *starch* and *lamp-black* (frequently contained in liquorice pastilles) may be detected by microscopical examination.

The analyses of a number of extracts are given in a table. The author concludes that commercial liquorice extracts, whether containing gelatin or not, differ in a decided manner only in respect to the percentage of glycyrrhizin, and that the other constituents are subject to irregular variations. Samples containing less than 10 per cent. of glycyrrhizin, the author considers to have been produced, probably, from spent liquorice root.—H. T. P.

Potassium Bitartrate, Estimation of the, contained in Wine. H. Gautier. Comptes Rend. 1897, **124**, [6], 298—300.

100 c.c. of the wine is concentrated by evaporation in a conical flask to about 15 c.c., and then set aside in a cool place, subject to as small fluctuations of temperature as possible, and alongside of it is placed a second flask, containing a saturated aqueous solution of potassium bitartrate. At the end of 2—3 days, the excess of tartar will have separated from the wine. (If crystallisation does not commence after about 12 hours, a minute crystal of tartar may be dropped into the liquid, and will induce the desired separation.) The mother-liquor is then decanted through a small filter, and its volume accurately ascertained. Subsequently, the crystals are washed with the aqueous tartar solution referred to above, the filter is returned to the flask, a volume of tartar solution, equal to that of the wine after concentration, added (to correct for the tartar retained by the mother-liquor), and then water, to dilute the whole to about 100 c.c. Finally, the liquid is heated, to facilitate solution of the crystals, and titrated with standard potassium hydrate and phenolphthalein in the usual way. The end-reaction, although somewhat obscured by the presence of colouring matters, is still sufficiently sharp. From the result obtained, 0·0015 grm. must be subtracted for each 1 c.c. of mother-liquor (*vide supra*), to allow for the slightly greater solubility of tartar in water as compared with wine extract. According to the author, this correction is practically constant for wines of different origin.—H. T. P.

Tea, Analysis of. Aglot. Rev. Chim. Industr. **8**, 17—20.

For the estimation of tannin, the author boils 1 grm. of powdered tea for 20 minutes with 50 c.c. of water, and repeats the operation several times with fresh water, finally making the volume up to 100 c.c., and determining the amount of tannin by the (Aglot) optical method, disregarding the turbidity due to albumin tannate.

From the examination of a number of teas, it appears that there is no definite relation between percentage of tannin and commercial value, the highest priced tea (Long-soo) containing 9·90, and the next highest (Loong-Tsing) 17·0 per cent., whereas another tea which contained the lowest of all (8·9 per cent.) was worth only one-tenth as much as the first-named. The theine, determined by the Domergue method—treatment with mercuric acetate and extraction by a mixture of equal parts of benzene and chloroform—ranged from 2·20 to 3·65 per cent.

In considering the question of detection of the adulteration of tea, it should be borne in mind that the most common practice consists in adding spent tea, and furthermore, that this spent tea still contains about one-half of its original tannin and theine. Now, since the percentage of tannin in sound tea ranges from 9 to 18 per cent., and the theine from 2 to 4 per cent., it is easy to make up an adulterated tea containing more than the minimum usually prescribed for *either one* of these bodies, but more difficult to prepare one having more than the minimum of *both*. It is therefore advisable to insert in hospital and similar contracts, a stipulation for a certain minimum percentage of tannin also, instead of theine alone, as at present. By this means,

assisted by the physical tests of flavour and odour, and by a knowledge of the original value of the reputed brand in the country of production, the difficulty of restricting such methods of adulteration would be lessened.—C. S.

Fat in Milk, Estimation of. H. Fresenius. Zeits. Anal. Chem. **36**, [1], 31—32.

The results obtained by the Babcock, Gerber, and gravimetric processes are compared. The gravimetric process used, consisted in evaporating the milk with quartz sand, and extracting the dry residue with ether in an extraction apparatus. The fat, after evaporation of the ether, was dried 1 hour at 100° and weighed. The results quoted show that only insignificant differences arise in the results obtained by the processes contrasted.—H. B.

Cheese, Estimation of Nitrogen in. B. A. van Ketel and A. C. Antusch. Nederl. Tijdschr. Pharm. 1897, 82—96.

The authors, having analysed a number of cheeses, come to the conclusion that only about 80 per cent. of the nitrogen exists in the form of albuminoids, the remainder being due to ammonia and amido bodies. The nitrogen existing as ammonia is estimated by distilling the sample, previously powdered with the addition of sand, with water holding barium carbonate in suspension. The distillate is received into a measured quantity of standard sulphuric acid, and, after boiling, the excess of acid is neutralised with standard soda, using rosolic acid as indicator. The nitrogen existing as amido compound, is estimated by macerating the powdered cheese with water for 15 hours at the ordinary temperature. After adding a little dilute sulphuric acid (1—4), the albuminoids and peptones are precipitated by phospho-tungstic acid. The precipitate is filtered off, and then washed with water containing a little sulphuric acid. The filtrate is made up to a definite bulk, and the nitrogen is estimated in an aliquot part of the liquid by Kjeldahl's process, allowance being of course made for the nitrogen existing as ammonia. The peptones and albumoses are estimated jointly by boiling the powdered cheese (mixed with sand, as before) with water, and filtering off from the undissolved casein and albumin. In an aliquot part of the filtrate, the peptones and albumoses are precipitated by adding dilute sulphuric acid and phospho-tungstic acid. After washing with acid water, the precipitate is submitted to Kjeldahl's process. By the same process, the total nitrogen of the cheese is estimated, and, after allowing for the nitrogen existing in a different state, the balance is calculated as usual to casein. The amount of indigestible casein was found to be very trifling.—L. de K.

Alkaloids, Determination of. E. H. Farr and R. Wright. Pharm. J. [4], **4**, 202.

The present official process of the British Pharmacopœia was found, with slight modifications, to be well suited for the determination of morphine in opium. The authors confirm Dott's statement that morphine as ordinarily precipitated, does not become perfectly anhydrous at 100° C., but loses often 5 or 6 per cent. of water, on further drying at 110° C. The following details for the assay of tincture of opium were given :—80 c.c. of tincture is evaporated to about 20 c.c., mixed with 3 grms. of freshly slaked lime, and diluted with water to 85 c.c., stirring at intervals for 30 minutes. 50 c.c. of the filtrate are then filtered into a well-stoppered 4-oz. bottle, and 2 grms. of ammonium chloride, 30 c.c. of ether, and 5 c.c. of alcohol added. The whole is then shaken well at intervals for half an hour and set aside for 12 hours. The ethereal layer is removed with a pipette, filtered through counterpoised filters, and the contents of the bottle rotated with another 15 c.c. of ether, which is also passed through the filters. These are washed with a little ether, and the crystalline precipitate is collected on them, and washed with morphinated water till colourless. It is then dried at a gradually increased temperature, and finally at 110° for one hour. It is then weighed. 30 centigrms. of the crystals are now dissolved in a slight excess of $\frac{1}{10}$N. H_2SO_4 solution, and then titrated back with $\frac{1}{10}$N. NaHO, using litmus paper as indicator. To the amount of pure anhydrous morphine in the total amount of crystals, indicated by this titration, 0·05 grm. is added, as representing the average amount of loss in the process. This method gives results closely concordant with those obtained by Dott's and by Teschemacher and Smith's processes.

The authors found that quinine salts, and the cinchona alkaloids generally, may be obtained in a perfectly anhydrous condition in a water oven, provided time enough for complete drying be allowed. They recommend flat-bottom dishes to be used for drying alkaloidal residues, and quote figures proving that desiccation therein takes place much more rapidly than in the round-bottom capsules usually employed.—J. O. B.

Kola, The Caffeïne Compound in. J. W. T. Knox and A. B. Prescott. J. Amer. Chem. Soc. 1897, **19**, 63—90.

Strong evidence is given that kolanin, the so-called glucoside of kola, is in reality a mixture of tannates of caffeïne and theobromine, and that the glucose obtained on hydrolysis is obtained from the tannin. Both the free and combined tannins were separated by a method, taken in part from Allen, and were found to possess the appearance and properties of glucoside tannins. Artificial preparations of caffeïne kola-tannin resembled kolanin, and gave similar reactions. Further work on its composition is contemplated.

When fresh kola seed is cut or bruised, a chemical change immediately takes place, as shown by the rapid change of colour of the cut surface from pink or cream-colour to red-brown. Various solvents were tried with the object of removing the active constituents without change, but without success. The coloration having been attributed to a diastatic ferment, sterilisation by heat was tried. A temperature of 45° C. is sufficient, when alcohol is employed, to coagulate the ferment—at least, to prevent the coloration afterwards.

For the assay of kola the process used was that given by Gomberg (this Journal, 1896, 384), based on the very complete precipitation of caffeïne as periodide by Wagner's reagent from aqueous solutions acidulated with mineral acids, preferably hydrochloric, but avoiding excess. The precipitate formed, $C_8H_{10}N_4O_2HI.I_4$, is constant under varying conditions of formation. The excess of iodine is measured, after filtering through a dry asbestos filter, by taking an aliquot part of the filtrate and titrating back with decinormal thiosulphate. 1 c.c. of Wagner's solution = 0·00485 grm. of caffeïne, the proportion of theobromine (1·48 per cent. of the total alkaloids) not appreciably affecting the factor.

For the estimation of theobromine in presence of caffeïne, the method of Kunze (this Journal, 1894, 178) was found very satisfactory; the alkaloid being precipitated as silver theobromine from an ammoniacal aqueous solution by nitrate of silver, and the precipitate converted into silver chloride on the filter.

The free alkaloid is extracted by chloroform, the combined by 90 per cent. alcohol. Dried kola contains half the alkaloid combined, fresh kola 60 per cent. Sterilising the kola, which checks the formation of the coloured body called kola-red, does not at the same time check the liberation of the alkaloid.—L. J. de W.

Formaldehyde, Methods for the Estimation of. G. Romijn. Zeits. anal. Chem. **36**, [1], 18—24.

In comparing four methods for estimating formaldehyde, the following aqueous solutions were used :—(1.) 2·075 grms. of pure "formaline." (2.) 2·075 grms. formaline + 1·3 grms. concentrated acetaldehyde. (3.) 2·075 grms. formaline + 0·353 grm. pure acetone. (4.) 2·075 grms. formaline + 1 grm. benzaldehyde,—these quantities being made up to 500 c.c. in each case. The first two methods are new.

(1.) *Iodometric Method*, based on the oxidation of formaldehyde in alkaline solution. 10 c.c. of solution "1" were mixed with 25 c.c. of tenth-normal iodine solution, and sodium hydrate solution dropped in till the liquid became clear yellow. After 10 minutes the reaction was complete; the excess of iodine was liberated with HCl, and

titrated with thiosulphate. Two atoms of I are equivalent to one molecule of formaldehyde. The results gave 37·38 and 37·40 per cent. of formaldehyde in the formaline. With solution "2," containing acetaldehyde, some iodoform was produced, and irregular, erroneous results were obtained. With solution "3" both the formaldehyde and the acetone were fully oxidised, the latter producing iodoform (as in Krämer's process). Solution "4," containing benzaldehyde, gave erroneous results, the benzaldehyde being considerably oxidised.

(2.) *Potassium Cyanide Method*, based on the property of formaldehyde of combining with KCN when solutions containing them are mixed. The addition-product can reduce alkaline silver nitrate solution in the cold, but if the solution be kept acid with HNO_3, then, when the aldehyde is in excess, no precipitate of silver cyanide appears, and when the KCN is in excess, one molecule of formaldehyde combines with one molecule of KCN, the excess of KCN precipitating AgCN from the $AgNO_3$ present. The same formaline solutions as above were used for these tests. To 10 c.c. of tenth-normal silver nitrate, acidified with two drops of nitric acid, were added 10 c.c. of a 0·62 per cent. KCN solution, the whole diluted to 50 c.c., filtered, and an aliquot portion titrated by Volhard's process, 0·39 c.c. being consumed by the whole 50 c.c. When solution "1" (formaline alone) was mixed with the KCN solution before adding to the silver nitrate solution, good results—37·39 and 37·67 per cent. of formaldehyde—were obtained. Using solution "2," if the cyanide-aldehyde solution, *immediately* after mixing, was added to the silver nitrate, good results were obtained; but if the cyanide and aldehyde stood some time after mixing, the results were much too high. Solutions "3" and "4" gave correct results, even after the cyanide-aldehyde mixture stood for half an hour.

(3.) *Hydroxylamine Method* (Brochet and Cambier's, Comptes Rend. **120**, 449).—This process gave good results with pure formaline, but the acetaldehyde, acetone, and benzaldehyde all vitiated the results.

(4.) *Legler's Method* (Ber. **16**, 1335).—The author does not recommend this process.

In general, pure formaldehyde solutions are best analysed by the iodometric process, and where other aldehydes may be suspected, the KCN process should be used.—H. B.

Acetone, Estimation of. L. F. Kebler. Amer. J. Pharm. **69**, 65; and Pharm. J. 1897, **58**, 161.

Place 20 c.c. of Squibb's alkaline potassium iodide solution (this Journal, 1897, 168) in a flask, add 10 c.c. of 1 to 2 per cent. aqueous solution of acetone, and excess of sodium hypochlorite solution (about four-fifths normal), then close the flask and shake. Next acidify with hydrochloric acid, add excess of decinormal sodium thiosulphate solution, and, after allowing to stand for a few minutes, add starch indicator, and re-titrate the excess of sodium thiosulphate. The relation of the sodium hypochlorite solution to the sodium thiosulphate solution being known, the amount of iodine set free by the hypochlorite used, can readily be calculated, and this multiplied by 0·07612 gives the amount of acetone. The method is said to yield satisfactory results for ordinary work.—A. S.

Guaiacol, Determination of, by Demethylation. M. Adrian. Nouveaux Rem. **13**, 97.

The process is a modification of that of Behal and Choay, compared with which the author claims that it gives more accurate results. 100 grms. of either commercial guaiacol or of creosote are placed in a flask of 250 c.c. capacity, connected on the one hand with another flask which serves as a generator of gaseous HBr, and on the other with a reflux condenser. The exit tube from the condenser leads the gas through two wash-bottles containing water; HBr is generated by the action of water upon PBr_3. The delivery tube between the generator and the guaiacol is furnished with a large bulb to prevent aspiration of the liquid when cooling. The gas is allowed to pass slowly through the guaiacol, which is gently warmed after the process has been in operation for half an hour, and is continued for 90 minutes. The liquid is then cooled, transferred to a large flask, diluted with 5 or 6 vols. of water, and all volatile bodies removed by steam distillation. When no more oily liquid comes over, the distillation is stopped, the pyrocatechol in the residual liquor is extracted, together with homopyrocatechol, by washing out with ether, the ether is cautiously distilled off, and the residue dried. This is then treated with benzene, which extracts the whole of the pyrocatechol, leaving the homopyrocatechol insoluble. From this last solvent pyrocatechol is crystallised out and weighed. Care must be taken at the end of the process to avoid the least trace of moisture either in the residue or in the solvent, or crystallisation will not take place.—J. O. B.

Iodoform Gauze, Examination of. G. Schacherl. Apoth. Zeit. 1897, **12**, 95.

From 1 to $1\frac{1}{2}$ grms. of the gauze are heated under pressure in a strong flask on the water-bath for half an hour with 30 grms. of a solution of sodium ethylate, containing about 2 per cent. of sodium. After cooling, the solution is decanted, the residue thoroughly washed with water, and the alcohol driven off from the united liquids by evaporation. The liquid, after cooling, is then acidulated with dilute nitric acid, 30 c.c. of $\frac{1}{10}$ N solution of silver nitrate added, and the whole made up to exactly 200 c.c. with distilled water. 100c.c. are then filtered off, and in this liquid the uncombined silver is determined by means of $\frac{1}{10}$ N solution of ammonium sulphocyanide with the customary iron alum indicator. Each c.c. of $\frac{1}{10}$ N silver solution = 0·01309 grm. of iodoform. It is found that many commercial specimens of iodoform gauze fall far below their reputed strength, 14·9 and 6·4 per cent. of the antiseptic being found in samples which were said to contain 50 and 30 per cent. respectively.—J. O. B.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Gallium, Occurrence of, in the Clay Ironstone of the Cleveland District of Yorkshire. W. N. Hartley and H. Ramage. Proc. Roy. Soc. 1897, **60**, 393.

The authors found that the blast-furnace metal obtained from the Yorkshire ironstone, smelted at Middlesbrough-on-Tees, contained 0·00304 per cent. of gallium. It was found that the gallium is contained in the ore and is concentrated in the metal. The gallium was estimated by a method of fractional precipitations and spectrographic analysis of the precipitates, supplemented by gravimetric determinations of the purified gallium sesquioxide.—A. S.

Crystalline Alumina, Synthesis by the Action of Chlorine on an Alkaline Aluminate. H. Loyer. Bull. Soc. Chim. 1897, **17**, [6], 345.

Debray has prepared corundum by the action of hydrochloric acid on sodium aluminate at a red heat. The author finds that a similar result is obtained by the use of chlorine, a slow current of which gas was passed for 4—5 hours through a porcelain tube containing the aluminate mixed with 2 per cent. of potassium chromate. Oxygen is evolved, the reaction appearing to be—

$$Al_2O_3.Na_2O + Cl_2 = Al_2O_3 + 2NaCl + O.$$

The tube contains a rose-red crystalline mass, which, under the microscope, is seen to consist of small hexagonal tables, these are occasionally green and more rarely blue. The crystals are insoluble in hydrochloric and nitric acids and have a greater density than methylene iodide.

—A. C. W.

Zircons in Tasmania, Discovery of. U.S. Cons. Reps., March 1897, 364.

The United States Consul-General at Melbourne reports that among the many valuable discoveries of late in the colony of Tasmania, one which has created much interest in mining circles has recently been partially developed. It consists of a deposit of zircons, allied with other gems and

rare earths. The gems found in this deposit are chiefly zircons, sapphires, and cinnamon rubies. The first-named are cound in large quantities and in every variety of colour. Many of them have been cut and polished, with very gratifying results, being hard and of good lustre. The specific gravity of the zircon is 4·7, thus being heavier than the diamond, which is 3·75; its hardness, compared with the diamond, is 7, the latter being 10. It is of high commercial value for use in the manufacture of mantles for incandescent lights. The composition of the pure zircon is 64 per cent. of zirconia and 36 per cent. of silica. Analyses by Dr. W. H. Gaze, of Melbourne, of two samples of zircons from the above-mentioned deposit give 63 per cent. and 64 per cent. of zirconia, thereby testifying as to the purity of the gem. The rarer earths, *viz.*, lanthanum, thorium, didymium, niobium, erbium, yttrium, cerium, and chromium, are all more or less present.

The property which contains this deposit has been purchased by a Melbourne syndicate. It comprises an area of 105 acres, situated on the north-west coast of Tasmania, about midway between Emu Bay and Circular Head, and is easily accessible by road from either port. The property is heavily timbered, among which are trees of unusual height and girth. There is also permanent water on the ground. A large amount of work has been done in cutting races and forming dams for sluicing purposes, much labour being expended in removing the heavy timber from the creeks.

Associated with the zircon samples is a very rare mineral known as pyrochlore, of which there are several distinct varieties. They are, however, all composed of two rare metals—niobium and uranium—with some of the cerium metals. It may be described as niobate of uranium, with cerium, thorium, yttrium, didymium, and titanium. The percentage of each metal varies, and often some of them are absent. Thorium and yttrium are often present to the extent of 10 per cent. each. At present Dr. Gaze has only determined niobium, uranium, and titanium, and the residue contains the other rare earths. Uranium is contained in very considerable quantities.

Dr. Gaze reports as follows :—

"In the first place, the mineral varies much in composition. It is, therefore, impossible to get two results alike, especially when one has to pick out hundreds of little grains to obtain enough to operate on, and there is no certainty that all the grains are identical. The mineral is of very complex nature, as the analysis below demonstrates. In some instances, chromium has to a great extent replaced uranium; in others, lanthanum and didymium replace thorium and yttrium. With more compact and larger specimens better results should be obtained. In some instances only traces of the rare earths were obtained.

"Assay of Shekleton minerals, nitrate of uranium and chromium, a variety of pyrochlore:—Uranium, 5 to 0·5 per cent.; chromium, 10·5 to 12·5 per cent.; titanium, 12 to 13 per cent.; niobium, 4·5 to 2·5 per cent.; iron, 25·5 to 27·7 per cent.; magnesium, 2·2 to 0·5 per cent.; alumina, 7·3 to 6·2 per cent.; lime, 2·6 to 1·5 per cent.; silica, 15 to 12 per cent.; didymium, 7·5 to 0·5 per cent.; lanthanum, 6·2 to 2·2 per cent.; thorium, 1 per cent. to traces; yttrium, 1·5 per cent. to traces."

Clevite. Chem. and Druggist, April 17, 1897, 623.

From a mine recently discovered in Ryfylke, Norway, various minerals containing rare earths have been obtained. Clevite is amongst them, and it is now offered at 2*l.* per kilo., or, say, 1*s.* 4*d.* an ounce. It yields 1·5 to 2 c.c. of helium per gramme; therefore, an ounce bottleful of helium is worth at least 8*d.*, and this does not include the cost of isolating it.

Sugar Group, A New Synthesis in the. H. J. H. Fenton. Proc. Chem. Soc. 1897, [176], 63.

In previous communications, it has been shown that the acid (dihydroxymaleic acid) obtained by oxidation of tartaric acid in presence of iron, decomposes, on heating with water, almost quantitatively into glycollic aldehyde and carbon dioxide. Also, that this aldehyde, when heated in a vacuum, undergoes condensation, yielding a sweet-tasting, solid gum which has the formula $C_6H_{12}O_6$.

The present paper describes an investigation which has been made upon the properties of this condensation product.

It is easily soluble in water, and its solution quickly reduces Fehling's solution and ammoniacal silver nitrate. It gives various colour reactions characteristic of "sugars," and, after purification with alcohol, yields, with phenylhydrazine, a normal hexosazone, $C_{18}H_{22}N_4O_4$, melting at 168°—170°. Heated with water to 140°, it yields furfural. It is optically inactive, and appears to be incapable of fermentation by yeast.

The purified "sugar," when further heated in a vacuum to 100°—106°, loses water and becomes hard and brittle. After 2—4 hours' heating it has the composition $C_{12}H_{22}O_{11}$ and after 24 hours' heating the composition nearly approximates to $C_6H_{10}O_5$.

The conditions under which tartaric acid may be converted into dihydroxymaleic acid by atmospheric oxygen exhibit close analogies with some of the essential conditions of vegetable growth; and it is suggested that the direct production of a "sugar" in the manner above indicated may possibly help to throw light upon the natural formation of carbohydrates.

PATENT.

Type, and of Typographic Plates and Matrices, A New Paste [Lithographic] adaptable for the Manufacture of; designed as a Substitute for Lithographic Stones; and Process for the Preparation and Use of this New Paste. Communicated by C. Delaze, Paris. Eng. Pat. 28,460, Dec. 12, 1896.

The basis of this paste is a limestone found in France near Vigan, in Hungary near Fünfkirchen, and in Bavaria near Munich. This is mixed with oleic acid, vaseline, or paraffin oil and with hydrochloric acid. For the preparation of typographic plates or matrices, silicate of potash is added. If required for lithographic purposes, carbonate of soda is also added. The composition, in the form of a paste, is then blown or cast through a pulveriser on to the surface destined to receive an impression.—V. C.

New Books.

Engineering Chemistry, A Manual of Quantitative Chemical Analysis for the use of Students, Chemists, and Engineers. Thomas B. Stillman, M.Sc., Ph.D. Professor of Analytical Chemistry in the Stevens Institute of Technology. Chemical Publishing Co., Easton, Pa., U.S. America. 1897. Price, 4·50 dols.

8vo volume containing preface, table of contents, and subject-matter, 505 pages, followed by an alphabetical index. The latter 18 pages of the work are devoted to tables. The text is illustrated with 154 engravings. The plan and scope of the work are given in the following summary of the contents :—Determination of Iron in Iron Wire; of Alumina in Potash Alum; of Copper in Copper Sulphate, &c., &c. Analysis of Limestone. Coal and Coke Analysis. Scheme for Analysis of Hematite, Limonite, and Spathic Iron Ores. For Analysis of Blast-Furnace Slag. Analysis of Water for Scale-forming Ingredients. Sanitary Analysis of Water. Composition of Boiler Scale. Water for Locomotive Use. Feed-Water Heaters, &c. Determination of Heating Power of Coal and Coke. Calorimetry. Determination of Sulphur in Steel and Cast Iron; of Silicon in Iron and Steel; Aluminium in Iron and Steel, &c., &c. Determination of Sulphuric Acid and SO_3 in fuming Nordhausen Acid; of Zinc in Zinc Ores, &c. Sodium Cyanide as a Component of Potassium Cyanide. The Chemical and Physical Examination of Portland Cement. Determination

of Nickel in Nickel Steel. Analysis of Chimney Gases for O, CO_2, CO and N · of Flue Gases with the Orsat-Moencke Apparatus: of Coal Gas, Water Gas, Producer Gas, &c. by the Hempel Apparatus, &c. Manufacture of Water Gas and Calculation of Heating Power of Various Illuminating Gases. Practical Photometry, Calorimetry, Analysis of Clay, Kaolin, Fire Sand, Building Stones, &c. Physical Tests of Building Stones. Alloys. Chemical and Physical Examination of Paper. Soap Analysis. Technical Examination of Petroleum and Lubricating Oils. Oils used for Illumination. Analysis of Lubricating Oils, of Cylinder Deposits. Paint Analysis. Pyrometry. The Electrical Units. Energy Equivalents.

Principles and Practice of Agricultural Analysis. A Manual for the Examination of Soils, Fertilisers, and Agricultural Products; for the use of Analysts, Teachers, and Students of Agricultural Chemistry. Vol. III. Agricultural Products. By Harvey Wiley, Chemist of the United States Department of Agriculture. Chemical Publishing Co., Easton, Pa. 1897. Price 3·75 dols.

Volume I. of this work has received notice (this Journal, 1895, 401). Vol. III. contains 644 pages of subject-matter, illustrated with 124 engravings, and followed by an alphabetical index. The subjects treated of are classified as follows:—Part I. Sampling, Drying, Incineration, and Extractions. II. Sugars and Starches. III. Separation and Determination of Carbohydrates in Crude and Manufactured Agricultural Products. IV. Fats and Oils. V. Separation and Estimation of Bodies containing Nitrogen. VI. Dairy Products. VII. Miscellaneous Agricultural Products.

The Printing of Textile Fabrics. A Practical Manual on the Printing of Cotton, Woollen, Silk, and Half-Silk Fabrics. By C. F. Seymour Rothwell, Lecturer on Calico and Delaine Printing at the Municipal Technical School, Manchester. Chas. Griffin and Co., Ltd., Exeter Street, Strand. 1897. Price 21s.

This work, it is stated, is intended to form a companion volume to Knecht Rawson, and Loewenthal's Manual of Dyeing (this Journal, 1897, 473). The volume is of similar size and appearance to the volume of the last-named work, and contains 302 pages of subject-matter and an alphabetical index, followed by 20 pages filled with printed and dyed patterns. The text is illustrated with 50 wood engravings, and is sub-divided into chapters treating of the following branches of the subject: — Introduction. Methods of Textile Printing. Styles. I. Machinery used in Textile Printing. II. Thickeners and Mordants. III. Printing of Cotton Goods. *a*. Bleaching of Cotton Piece Goods for Printing. *b*. The Steam Style. *c*. Colours produced directly on the fibre. *d*. Dyed Styles. *e*. Padding Style. *f*. Resist and Discharge Styles. *g*. Printing of Compound Colourings. IV. Printing of Woollen Goods. V. Printing of Silk Goods. VI. Practical Recipes for Printing. Appendix of Useful Tables.

Notes on Assaying. By P. de Peyster Ricketts, E.M., Ph.D., Professor of Analytical Chemistry and Assaying, School of Mines, Columbia University, and Ed. H. Miller, A.M., Ph.D. John Wiley and Sons, New York, U.S.A. Chapman and Hall, Ltd., London. 1897. Price 12s. 6*d*.

8vo volume containing preface, introduction, text filling 289 pages, and alphabetical index. The wood-cut illustrations are 39 in number, and at the close of the volume are given a collection of examples and problems, a list of suitable works of reference, &c. on assaying, and a list of articles for a laboratory outfit for ordinary fire assays, with the costs of these. The work is sub-divided as follows:—Part I. Apparatus. Reagents and Chemicals. Operations. II. Dry or Fire Assays. III. Wet Assays or Analyses. IV. Laboratory Tests for Ores. Assay, Blowpipe, and Qualitative Schemes. V. Tables, Examples, and References.

The Elements of Electro-Chemistry, treated Experimentally. By Dr. Robert Lüpke, Lecturer in the Imperial School of Posts and Telegraphs, Berlin. Translated from the 2nd revised and enlarged Edition by M. M. Pattison Muir, M.A., Fellow and Lecturer of Gonville and Caius College, Cambridge. H. Grevel and Co., 33, King Street, Covent Garden, London, W.C. 1897. Price 7*s*. 6*d*.

"Although the main purpose of this book," as stated in the preface, "is to set forth the scientific aspects of Electro-Chemistry, the practical sides of the subject have not been left unnoticed." The work contains 218 pages of subject-matter, with 54 wood-cuts, and an alphabetical index. The matter is classified as follows:—Part I. Recent Theories of Electrolysis. (i.) Phenomena of Electrolysis. (ii.) Faraday's Law. (iii.) Hittorf's Transport Numbers. (iv.) Law of Kohlrausch. (v.) Dissociation Theory of Arrhenius. Part II. The Theory of Solutions of Van' T. Hoff. (i.) Osmotic Pressure. (ii.) The Vapour pressures of Solutions. (iii.) Boiling Points and Freezing-Points of Solutions. (iv.) Aqueous Solutions of Electrolytes. Part III. The Osmotic Theory of the Current of Galvanic Cells. (i.) Liquid Cells. (ii.) Concentration Cells. (iii.) Daniell Cells. (iv.) Reduction Cells and Oxidation Cells. (v.) Solution-pressures of the Metals. (vi.) Intensity of Fixation and Polarisation. (vii.) Irreversible Cells. (viii.) Accumulators. (ix.) The Energetics of Galvanic Elements.

Entwickelung, Bau und Betrieb der Elektrischen Öfen zur Gewinnung von Metallen, Carbiden, und anderen Metallurgisch-wichtigen Produkten. Von Dr. W. Borchers. Wilhelm Knapp, Halle a/S., Germany. H. Grevel and Co., 33, King Street, Covent Garden, London, W.C. 1897. Price M. 2.

Pamphlet containing 64 pages of subject-matter, illustrated with 42 illustrative engravings, and followed by an alphabetical index of names of authors, discoverers, or inventors. The contents of the little work are classified as follows:—Die Erhitzungsarten. I. Widerstandserhitzung. II. Lichtbogenerhitzung.

Le Four Électrique. Par M. Henri Moissan. G. Steinheil, 2, Rue Casimir-Delavigne, Paris. 1897. Price Fr. 15. H. Grevel and Co., 33, King Street, Covent Garden, London, W.C.

Large 8vo volume containing preface, 373 pages of subject-matter illustrated with 41 engravings, 3 pages of a classified Bibliographic index, and a table of contents. The matter is arranged as follows:—I. Description of various models of Electric Furnace. II. Researches on the different Varieties of Carbon. III. Preparation in the Electric Furnace of some Simple Substances. IV. Study of Carbides, Silicides, and Borides. A valuable feature of the work is the brief chapter devoted to "Conclusions" following each section of the subject.

Trade Report.

TARIFF CHANGES AND CUSTOMS REGULATIONS.

(*From the Board of Trade Journal.*)

Italy.

Importation of Medicines.

The *Bollettino di Notizie Commerciali* publishes a decree of the Director-General of Taxes, according to which medicinal specialities which are not sold as patent medicines (*rimedi o specifici segreti*) must have a ticket printed with the formula of composition attached, either to the receptacle itself (*i.e.*, the box or bottle in which the medicine is directly contained), or else on the outer wrapper in which the article is exposed for sale when the receptacle is wrapped in another wrapper or contained in a sealed packet or something similar.

United States.

Tariff Changes.

Name of Article.	*McKinley Bill, 1890.	Wilson Bill, 1894.	Proposed Dingley Bill, 1897.
Acetate of lead, white	5¼ c. per lb.	2¾ c. per lb.	3½ c. per lb.
,, ,, brown	3½ c. ,,	1¾ c. ,,	2½ c. ,,
Acid, acetic or pyroligneous	1½ c. ,,	20 per cent.	3 c. ,,
,, benzoic	Free	Free	25 per cent.
,, boracic	5 c. per lb.	3 c. per lb.	3 c. per lb.
,, carbolic	Free	Free	25 per cent.
,, chromic	6 c. per lb.	4 c. per lb.	4 c. per lb.
,, citric	10 c. ,,	25 per cent.	8 c. ,,
,, gallic	Free	Free	10 c. ,,
,, lactic	,,	,,	4 c. ,,
,, oxalic	,,	,,	25 per cent.
,, salicylic	,,	,,	10 c. per lb.
,, sulphuric	¼ c. per lb.	,,	¼ c. ,,
,, tannic	75 c. ,,	60 c. per lb.	50 c. ,,
,, tartaric	10 c. ,,	20 per cent.	7 c. ,,
Alcoholic perfumery	$2·00 per gallon and 50 per cent.	$2·00 per gallon and 50 per cent.	60 c. per lb. and 45 per cent.
,, preparations, medicinal	50 c. per lb.	50 c. per lb.	55 c. per lb.
Alum, alum cake, patent alum, sulphate of alumina and alum, crystals or ground.	6/10 c. ,,	4/10 c. ,,	½ c. ,,
Alumina	6/10 c. ,,	4/10 c. ,,	6/10 c. ,,
Ammonia, carbonate	1¼ c. ,,	20 per cent.	1¼ c. ,,
,, muriate or sal-ammoniac	¾ c. ,,	10 ,,	¾ c. ,,
,, sulphate	½ c. ,,	20 ,,	½ c. ,,
Antimony, regulus or metal	¾ c. ,,	Free	¾ c. ,,
Arzols, or crude tartar	Free.	,,	1½ c. ,,
Baryta, sulphate of, manufactured	$6·72 per ton	$3·00 per ton.	$5·25 per ton
Bay rum	$1·50 per proof gallon	$1·00 per proof gallon	$1·50 per proof gallon
Benzol naphtha, &c.	Free	Free	25 per cent.
Blue vitriol or sulphate copper	2 c. per lb.	,,	1 c. per lb.
Borax, crude, or borate of soda or lime	3 c. ,,	1½ c. per lb.	2 c. ,,
,, refined	5 c. ,,	2 c. ,,	3 c. ,,
Bromine	Free	Free	25 per cent.
Castor oil	80 c. per gallon	35 c. per gallon	35 c. per gallon
Calomel	35 per cent.	25 per cent.	35 per cent.
Camphor, refined	4 c. per lb.	10 ,,	4 c. per lb.
Caustic potash, in sticks or rolls	1 c. ,,	Free	1 c. ,,
Chalk, prepared, precipitated, French, and red	1 c. ,,	20 per cent.	1 c. ,,
Chloral hydrate	50 c. ,,	25 ,,	55 c. ,,
Chloroform	25 c. ,,	25 c. per lb.	20 c. ,,
Chlorate potash	Free	Free	3 c. ,,
Chromate and bichromate of potash	3 c. per lb.	25 per cent.	3 c. ,,
Coal tar colours or dyes. (Aniline colours)	35 per cent.	25 ,,	35 per cent.
Coal tar products and preparations, not colours or dyes	20 ,,	Free	25 ,,
Cobalt, oxide of	30 c. per lb.	25 c. per lb.	25 c. per lb.
Cocoa butter	3½ c. ,,	3½ c. ,,	6 c. ,,
Cod liver oil	15 c. per gallon	20 per cent.	15 c. per gallon
,, oil	8 c. ,,	Free	Free
Copperas or sulphate of iron	3/10 c. per lb.	,,	¼ c. per lb.
Cotton seed oil	10 c. per gallon	,,	7 c. per gallon
Cream of tartar	6 c. per lb.	20 per cent.	6 c. per lb.
Croton oil	30 c. ,,	Free.	20 c. ,,
Cyanide of potassium	25 per cent.	25 per cent.	6 c. ,,
Dextrine of British gum	1½ c. per lb.	1½ c. per lb.	1½ c. ,,
Drugs which have been advanced in value by refining, grinding, or other process of manufacturing.	10 per cent.	10 per cent.	10 per cent.
Ether, fruit, oils, or essences	$2·50 per lb.	$2·00 per lb.	$2·00 per lb.
,, nitrous	25 c. ,,	25 c. ,,	25 c. ,,
,, sulphuric	40 c. ,,	40 c. ,,	25 c. ,,
Extracts of logwood, sumac, &c.	⅜ c. ,,	10 per cent.	⅜ c. ,,
,, hemlock bark	1 c. ,,	,,	½ c. ,,
Fuller's earth, manufactured	$3·00 per ton	$1·00 per ton	$2·00 per ton
Fusel oil or amylic alcohol	10 per cent.	10 per cent.	½ c. per lb.
Glauber salts (sulphate of soda)	$1·25 per ton	Free	$1·25 per ton
Glycerine, crude, not purified	1¾ c. per lb.	1 c. per lb.	1 c. per lb.
,, refined	4½ c. ,,	3 c. ,,	3 c. ,,
Gold leaf, packages of 500 leaves	$2·00 per package	30 per cent.	$2·00 per package
Indigo, extract or paste	¾ c. per lb.	Free	¾ c. per lb.
Indigo, carmined	10 c. ,,	,,	10 c. ,,
Ink, printing ink and ink powders	30 per cent.	25 per cent.	25 per cent.
Iodide of potassium	50 c. per lb.	25 c. per lb.	25 c. per lb.
Iodine, resublimed	30 c. ,,	Free	20 c. ,,
Iodoform	$1·50 ,,	$1·00 per lb.	$1·00 per lb.
Linseed oil, raw or boiled	32 c. per gallon	20 c. per gallon	32 c. per gallon
Litharge	3 c. per lb.	1½ c. per lb.	2¾ c. per lb.
Magnesia, carbonate	4 c. ,,	3 c. ,,	3 c. ,,
,, calcined	8 c. ,,	7 c. ,,	7 c. ,,
,, sulphate	3/10 c. ,,	⅕ c. ,,	⅕ c. ,,
Mica	35 per cent.	20 per cent.	3 c. per lb. and 15 per cent.
Morphine and its salts and codeine	50 c. per oz.	50 c. per oz.	$1·00 per oz.
Naphthalene	20 per cent.	Free	25 per cent.
Oils, lemon, bergamot, citronella	Free	,,	,,
,, myrbane	20 per cent.	,,	,,
Olive oil, fit for salad purposes	35 c. per gallon	35 c. per gallon	50 c. per gallon.
Opium, aqueous extract, tincture and other liquid preparations	40 per cent.	20 per cent.	40 per cent.
,, containing less than 9 per cent. of morphine	$12·00 per lb.	$6·00 per lb.	$6·00 per lb.
,, prepared for smoking	$12·00 ,,	$6·00 ,,	$6·00 ,,
,, crude, containing 9 per cent. and over of morphine	Free	Free	$1·00 ,,
Paraffin	,,	,,	40 per cent.
Paris green and London purple	25 per cent.	12½ per cent.	25 ,,
Peppermint oil	80 c. per lb.	25 ,,	65 c. per lb.
Phenacetin	25 per cent.	25 ,,	8 c. per oz.

Tariff Changes—continued.

Name of Article.	McKinley Bill, 1890.	Wilson Bill, 1894.	Proposed Dingley Bill. 1897.
Phosphorus	20 c. per lb.	15 c. per lb.	20 c. per lb.
Prussiate of potash, red	10 c. „	25 per cent.	8 c. „
„ „ yellow	5 c. „	25 „	4 c. „
Prussian blue	6 c. „	6 c. per lb.	8 c. „
Quicksilver	10 c. „	7 c. „	10 c. „
Red lead	3 c. „	1½ c. „	2½ c. „
Rochelle salts	3 c. „	2 c. „	4 c. „
Saccharin	25 per cent.	25 per cent.	$2·00 per lb. and 15 per cent.
Saltpetre, refined	1 c. per lb.	½ c. per lb.	¾ c. per lb.
Santonin	$2·50 „	$1·00 „	$1·00 „
Silver leaf, per package of 500 leaves	75 c. per package.	30 per cent.	75 c. per package
Soap, castile	1¼ c. per lb.	20 „	1½ c. per lb.
„ common	20 per cent.	10 „	20 per cent.
„ fancy and toilet	15 c. per lb.	35 „	15 c. per lb.
Soda, ash	¼ c. „	¼ c. per lb.	⅜ c. „
„ bicarbonate	1 c. „	½ c. „	¾ c. „
„ bichromate and chromate	3 c. „	25 per cent.	2 c. „
„ caustic	1 c. „	½ c. per lb.	½ c. „
„ crystals or sal soda	¼ c. „	⅛ c. „	⅛ c. „
„ nitrite	25 per cent.	25 per cent.	2½ c. „
„ silicate	½ c. per lb.	⅜ c. per lb.	⅜ c. „
Strychnine, and its salts	40 c. per oz.	30 c. per oz.	30 c. per oz
Sugar of milk	8 c. per lb.	5 c. per lb.	5 c. per lb
Sulphur, refined	$8·00 per ton	20 per cent.	$8·00 per ton
„ sublimed, or flowers of	$10·00 „	20 „	$8·00 „
Sumac, ground	3/10 c. per lb.	10 „	3/10 c. per lb.
Thymol	Free	Free	25 per cent.
Toilet preparations: Cosmetics, dentifrices, pomades, &c.	50 per cent.	40 per cent.	50 „
Turkey-red oil (for 50 per cent.)	40 c. per gallon	30 „	40 c. per gallon
Ultramarine blue, dry, pulp, or mixed with water	4½ c. per lb.	3 c. per lb.	4 c. per lb.
Vanillin	25 per cent.	25 per cent.	70 c. per oz.
Varnishes, including gold size and japan	35 „	„	35 per cent.
Vermilion red, and colours containing quicksilver	12 c. per lb.	20 per cent.	12 c. per lb.
White lead, dry or ground, in oil	3 c. „	1½ c. per lb.	2⅞ c. „
Whiting and Paris white, dry	½ c. „	¼ c. „	¼ c. „
Zinc, oxide, dry or ground, in oil	1¼ and 1¾ c. per lb.	1 c. „	Dry, 1 c. per lb. Ground, 1¾ c. per lb.
„ chloride	25 per cent.	25 per cent.	1 c. per lb.
„ sulphate	„	„	1 c. „

March 18, 1897.

SCHOELLKOPF, HARTFORD, AND MACLAGAN, LTD.

GENERAL TRADE NOTES.

THE TECHNICAL MUSEUM AT HONG KONG.

Bd. of Trade J., April 1897, 474.

The *Manufacturer* (Philadelphia) states that attention has been called to the great success attending the "Technical Museum" in Hong Kong, which, about a year ago, was established by Tschingtschaufu missionaries. Beginning in a small way, it has been gradually extended, and has become very popular with the Chinese, 82,000 of whom visited it during the last year. The Chinese cannot be convinced or enlightened by circulars or pictures, and hold fast to the belief that the Celestials are vastly superior to the Western barbarians. But when they see, as in this Hong Kong museum, the wonderful achievements of electrical machines, little models of railroads in full operation, and other technical appliances, then they become interested, susceptible to conviction, and willing to have the thing introduced in their country or their houses.

FRENCH SUGAR PRODUCTION.

Bd. of Trade J., April 1897, 469.

The French *Bulletin* of the Minister of Agriculture for March last, states that the sugar season of 1894–95 is characterised by two principal features, *viz.*, the marked increase in the output (a larger amount than has ever been produced in France), and the diminution in prices.

Sugars of 88° have fallen from 33·86 frs., the average price (per 100 kilos.) of 1893–94, to 26·50 frs., and No. 3 sugars from 35·65 frs. to 27·65 frs. This is a result of the general over-production, to which France, long the most important of producing countries, has only contributed in a slight degree.

Twenty years ago, in 1874–75, Germany produced 250,708 tons of raw sugar, Austria 106,312 tons, and France 450,711 tons. In 1894–95 the output of raw sugar has risen in Germany to 1,831,600 tons, in Austria to 1,044,500 tons; but in France to only 745,100 tons.

THE INDIA-RUBBER TRADE OF LAGOS.

Bd. of Trade J., April 1897, 481.

The *Lagos Echo* states that, according to the returns to hand for the period July—December 1896, the total value of imports into the colony was 442,075*l.*, as against 459,302*l.* for the same period in 1895—a decrease of 17,227*l.* The total amount of exports for the same period in 1896 was 456,698*l.*, as against 568,423*l.* in 1895, showing the enormous decrease of 117,726*l.*

The decrease, particularly in the exports, is very marked and unusual, and speaks volumes for the retrogression of the trade of the colony. The item of export in which there is the greatest decrease is rubber, of which commodity there were exported, from July to December 1896, 3,541,287 lb., valued at 198,167*l.*, as against 4,480,871 lb., valued at 240,127*l.*, in 1895, a decrease of 939,584 lb., valued at 41,959*l.* A large portion of this decrease may be set down to the fact that the yield in the near districts has been about exhausted, the trees being nearly all destroyed by the reckless manner in which the bark is stripped; so that collectors have now to seek their supply further from home, where the difficulties of transportation and the additional expense have been such as to discourage many from pursuing the industry. Palm kernels also, however, and palm oil show—especially the former—if not such a large decrease, at any rate a sufficiently marked one.

THE MINERAL PRODUCTION OF SPAIN IN 1896.

Eng. and Mining J., April 17, 1897, 379.

According to his usual custom, Señor Roman Oriol has prepared and published in the *Revista Minera* an interesting summary of the mineral production of Spain for the past year, from which the figures given below are obtained.

Silver.—The total production of silver in 1896 is estimated at 222,900 kilos., showing an increase over the preceding year. Of this total, 192,900 kilos. were obtained

from argentiferous lead. The only mines in which silver ores other than lead ores are found are in the province of Guadalajara; these mines produced last year 30,000 kilos. of silver.

Lead.—This is the most valuable mineral product of Spain, on account of the large quantity produced and the number of mines. A large part of the ores produced are exported as ores, but the amount of lead made in Spain is considerable. The total quantity of lead ores mined in 1896 was 320,000 metric tons, of which 162,600 tons were exported. The production of metallic lead, including the lead exported in ores, is estimated at 170,790 tons, an increase of 10,000 tons over the preceding year.

Quicksilver.—In the quicksilver mining industry, the most notable event in 1896 was the installation of compressed air rock drills in the twelfth level of the Almaden mines. These mines produced 22,100 tons of ore during the year under review. In Asturias, El Porvenir Mining Company raised 7,065 tons of ore; the Union Asturiana, 4,471 tons: and La Soterraña, 670 tons. In Granada, 800 tons were obtained. At Almaden, 16,076 tons were smelted, yielding 41,330 flasks of quicksilver. The total quicksilver production of Spain amounted to 43,884 flasks.

Copper.—The copper industry continued its prosperous career in Huelva in 1896. The Rio Tinto Company extracted 1,440,000 tons of cupriferous pyrites. Adding to this the output of other mines, the ore product of the province of Huelva amounted to 2,800,000 tons. The province of Seville produced from the mines of Aznalcollar some 25,000 tons of similar ore, and the mine Profunda, of Villamanin, in Leon, produced 1,050 tons of low-grade ore. The metallurgical treatment of the Spanish ores yielded 35,000 tons of precipitates with 75 per cent. of metal, and 23,000 tons of matte with 45 per cent. of metal. Besides these, 4,174 tons of sulphate of copper were produced by the Rio Tinto Company.

Zinc.—The centre of the zinc industry is the province of Santander, where the Royal Asturian Company raised 24,000 tons of crude calamine from the mines at Reocin, and 7,200 tons from the Udias and La Florida mines. From Reocin, also, 810 tons of blende were obtained. Zinc smelting is confined to the Arnao Works, in Asturias, where the out-turn amounted to 6,000 tons of spelter.

Iron Ore and Iron.—The production of iron ore in Spain for the year 1896 was 6,808,000 metric tons, an increase of 1,293,671 tons, or 23·5 per cent. over 1895. The exports were 6,253,473 tons, a gain of 1,005,281 tons, or 19·2 per cent. The production of pig-iron in Spain was 246,326 metric tons in 1896, of which 23,805 tons were exported. The production of steel ingots was 104,577 tons, 62,511 tons being made by the Bessemer and 42,066 by the open-hearth process. The total production of wrought iron and steel in bars, plates, and other finished forms was 137,809 tons. There were seven steel and iron works in operation during 1896.

Coal.—The production of coal in Spain for the year is given as follows, in metric tons:—

—	1895.	1896.	Changes.
Coal	1,739,075	1,830,771	Increase, 91,696
Lignite	44,008	44,000	Decrease, 8
Total........	1,783,083	1,874,771	Increase, 91,688

Of the coal in 1896, the Asturias produced 1,122,700 tons; Cordoba, 304,000 tons; Palencia, 132,000 tons; and Ciudad Real, 100,000 tons. Of lignite, Baleares produced 18,000 tons and Barcelona 15,000 tons.

Other Minerals.—Iron pyrites were mined and shipped by the Aguas Tenidas Company to the extent of 200,000 tons, a decrease of 40,000 tons from 1895. In manganese mining there was a remarkable gain, the production of manganese ores increasing from 10,162 metric tons in 1895 to 92,000 tons in 1896.

The production of salt amounted to 350,000 tons. The province of Gerona reports an output of 105 tons of barytes and 592 tons of soapstone. The production of fluorspar in Barcelona has almost ceased, only three tons being reported last year.

Amendments to the United States Patent Statutes.

Scientific American, March 20, 1897, 179.

The Old Statutes.	*The Amended Statutes.*
Sect. 4886.—Any person who has invented or discovered any new and useful art, machine, manufacture, or composition of matter, or any new and useful improvement thereof, not known or used by others in this country, and not patented or described in any printed publication in this or any foreign country, before his invention or discovery thereof, and not in public use or on sale for more than two years prior to his application, unless the same is proved to have been abandoned, may, upon payment of the fees required by law, and other due proceedings had, obtain a patent therefor.	Sect. 4886.—Any person who has invented or discovered any new and useful art, machine, manufacture, or composition of matter, or any new and useful improvements thereof, not known or used by others in this country before his invention or discovery thereof, *and not patented or described in any printed publication in this or any foreign country, before his invention or discovery thereof, or more than two years prior to his application,* and not in public use or on sale in this country for more than two years prior to his application, unless the same is proved to have been abandoned, may, upon payment of the fees required by law, and other due proceeding had, obtain a patent therefor.
Sect. 4920.—In any action for infringement the defendant may plead the general issue, and having given notice in writing to the plaintiff or his attorney 30 days before, may prove, on trial, any one or more of the special matters:	Sect. 4920.—In any action for infringement the defendant may plead the general issue. and having given notice in writing to the plaintiff or his attorney 30 days before, may prove, on trial, any one or more of the following special matters:
. . . . . .	. . . . . .
Third. That it had been patented or described in some printed publication prior to his supposed invention or discovery thereof; or,	*Third. That it has been patented or described in some printed publication prior to his supposed invention or discovery thereof, or more than two years prior to his application for a patent therefor; or,*
Sect. 4887.—No person shall be debarred from receiving a patent for his invention or discovery, nor shall any patent be declared invalid, by reason of its having been first patented or caused to be patented in a foreign country, unless the same has been introduced into public use in the United States for more than two years prior to the application. But every patent granted for an invention which has been previously patented in a foreign country shall be so limited as to expire at the same time with the foreign patent, or, if there be more than one, at the same time with the one having the shortest term, and in no case shall it be in force more than 17 years.	Sect. 4887.—No person *otherwise entitled thereto* shall be debarred from receiving a patent for his invention or discovery, nor shall any patent be declared invalid, by reason of its having been first patented or caused to be patented by the inventor or his legal representatives or assigns in a foreign country, *unless the application for said foreign patent was filed more than seven months prior to the filing of the application in this country, in which case no patent shall be granted in this country.*

Amendments to the U.S. Patent Statutes—*cont.*

The Old Statutes.	*The Amended Statutes.*
Sect. 4894.—All applications for patents shall be completed and prepared for examination within two years after the filing of the application, and in default thereof, or upon failure of the applicant to prosecute the same within two years after any action therein, of which notice shall have been given to the applicant, they shall be regarded as abandoned by the parties thereto, unless it be shown to the satisfaction of the Commissioner of Patents that such delay was unavoidable.	Sect. 4894.—All applications for patents shall be completed and prepared for examination within *one year* after the filing of the application, and in default thereof or upon failure of the applicant to prosecute the same within *one year* after any action therein, of which notice shall have been given to the applicant, they shall be regarded as abandoned by the parties thereto, unless it be shown to the satisfaction of the Commissioner of Patents that such delay was unavoidable.
Sect. 4898.—Every patent or any interest therein shall be assignable in law by an instrument in writing; and the patentee or his assigns or legal representatives may, in like manner, grant and convey an exclusive right under his patent to the whole or any specified part of the United States. An assignment, grant, or conveyance shall be void as against any subsequent purchaser or mortgagee for a valuable consideration, without notice, unless it is recorded in the Patent Office within three months from the date thereof.	Sect. 4898.—Every patent or any interest therein shall be assignable in law by an instrument in writing; and the patentee or his assigns or legal representatives may in like manner grant and convey an exclusive right under his patent to the whole or any specified part of the United States. An assignment, grant, or conveyance shall be void as against any subsequent purchaser or mortgagee for a valuable consideration, without notice, unless it is recorded in the Patent Office within three months from the date thereof. *If any such assignment, grant, or conveyance of any patent shall be acknowledged before any notary public of the several States or Territories or the district of Columbia, or any commissioner of the United States Circuit Court, or before any secretary of legation or consular officer authorised to administer oaths, or perform notarial acts under section* 1750 *of the Revised Statutes, the certificate of such acknowledgment, under the hand and official seal of such notary or other officer, shall be primâ facie evidence of the execution of such assignment, grant, or conveyance.*
Sect. 4921.—The several courts vested with jurisdiction of cases arising under the patent laws shall have power to grant injunctions according to the course and principles of courts of equity, to prevent the violation of any right secured by patent, on such terms as the court may deem reasonable; and upon a decree being rendered in any such case for an infringement, the complainant shall be entitled to recover, in addition to the profits to be accounted for by the defendant, the damages the	Sect. 4921.—The several courts vested with jurisdiction of cases arising under the patent laws shall have power to grant injunctions according to the course and principles of courts of equity, to prevent the violation of any right secured by patent, on such terms as the court may deem reasonable; and upon a decree being rendered in any such case for an infringement, the complainant shall be entitled to recover, in addition to the profits to be accounted for by the defendant, the damages the

Amendments to the U.S. Patent Statutes—*cont.*

The Old Statutes.	*The Amended Statutes.*
complainant has sustained thereby; and the court shall assess the same or cause the same to be assessed under its direction. And the court shall have the same power to increase the such damages, in its discretion, as is given to increase the damages found by verdicts in actions in the nature of actions of trespass upon the case.	complainant has sustained thereby; and the court shall assess the same or cause the same to be assessed under its direction. And the court shall have the same power to increase such damages, in its discretion, as is given to increase the damages found by verdicts in actions in the nature of actions of trespass upon the case. *But in any suit or action brought for the infringement of any patent there shall be no recovery of profits or damages for any infringement committed more than six years before the filing of the bill of complaint or the issuing of the writ in such suit or action, and this provision shall apply to existing causes of action.*
	Sect. 7.—*That in every case where the head of any Department of the Government shall request the Commissioner of Patents to expedite the consideration of an application for a patent, it shall be the duty of such head of a Department to be represented before the Commissioner in order to prevent the improper issue of a patent.*
	Sect. 8.—That this Act shall take effect January 1st, 1898, and sections 1, 2, 3, and 4, amending sections 4886, 4920, 4887, and 4894, of the Revised Statutes, shall not apply to any patent granted prior to said date, nor to any application filed prior to said date, nor to any patent granted on such an application.
	Approved, March 3, 1897.

This new law comes into operation on January 1st, 1898.

The Patent Law.

Chem. and Druggist, April 17, 1897, 642.

Sir Courtenay Boyle, Permanent Secretary to the Board of Trade, received, on April 8, a deputation of prominent British manufacturers, mostly from Lancashire, who had come to represent to him that great injury is inflicted upon British commerce by the ineffective character of the Patent and Trade Marks Act of 1883, especially that part of it (section 22) which enables foreigners to take out a patent in the United Kingdom for an invention for which patent rights have been refused them in their own country, or for one which is never worked in Britain at all. In almost every State, except Britain, the granting of patent rights is subject to the condition that the object patented shall be manufactured in the country within a certain time. But in this country the Board of Trade have, nominally, the power to grant licences to work a patent if the patentee "unreasonably" refuses such a licence. During the 14 years' existence of our patents law, however, only four applications for such licences have been made, and not one has been granted, the fact being that the Board of Trade considers itself prevented from exercising its power unless

the patentee absolutely refuses a licence. So long as he stops just short of a direct refusal, he may go on without hindrance making his patented article abroad, and dumping it down here in the finished state. This the deputation want remedied, either by a short Act of Parliament or by action by the Board of Trade. Mr. Ivan Levinstein, who was a member of the deputation, said that between 1891 and 1895 no fewer than 600 patents were granted in this country to foreigners, and in not a single instance of all the number were the goods patented made in this country. Sir Courtenay Boyle could not hold out much hope of alteration in this state of things. There would be no time this Session to pass such an Act as the deputation asked for, he said.

Proposed Tax on Calcium Acetate and other Distillation Products of Wood in Germany.

Chem. Ind. 1897, **20**, 133.

A group of forest proprietors in Germany is about to agitate for the imposition of a tax on the products of the dry distillation of wood, with the idea of increasing the market value of the raw material. In this connection it is pointed out that, so far as methyl alcohol is concerned, the only object effected would be to cripple the export colour and other industries employing this product (a good deal of which is imported). So, too, with acetic acid, which Germany is in a position to export in a manufactured condition as lead compounds, dyestuffs, &c.; and the present business done in the purchase of calcium acetate, and its conversion into more valuable products, would also suffer, owing to the difficulties in the way of arranging rebate of duty on re-export.

The case of Austria-Hungary is cited, where the import duty is prohibitive, without, however, increasing the price of wood.—C. S.

Production of Camphor in China.

A. Henry. Pharm. J., **4**, 201.

The value of the secretion of *cinnamomum camphora* has only been known to the Chinese for 300 or 400 years, although the tree was valued for its timber from ancient times. The camphor first used in medicine was doubtless Malay camphor. Until recent years no camphor was produced on the mainland of China, but the industry has now been started, and promises to become important. This is the more noteworthy as Formosa has become Japanese territory, and it seemed likely at one time that the Japanese Government would establish a monopoly of the camphor trade. The increase of the camphor industry in China is shown from the fact that in 1885 only 25 piculs were exported from Ningpo, while in 1895 five ports of the Empire exported a total of 1,756 piculs. In Fukien the forests of indigenous camphor trees are considerable, but as yet this district has not furnished any material quantity to the above output, which is chiefly derived from Kwangsi, where it promises to develop in importance.—J. O. B.

BOARD OF TRADE RETURNS.

Summary of Imports.

Articles.	Month ending 31st March.	
	1896.	1897.
	£	£
Metals	1,602,956	2,060,013
Chemicals and dyestuffs	953,968	1,012,437
Oils	581,593	476,778
Raw materials for non-textile industries.	3,330,205	3,381,380
Total value of all imports	38,366,750	40,655,114

Summary of Exports.

Articles.	Month ending 31st March.	
	1896.	1897.
	£	£
Metals (other than machinery)	2,684,456	3,169,955
Chemicals and medicines	867,574	930,096
Miscellaneous articles	2,931,006	3,073,757
Total value of all exports	20,449,623	21,647,269

Imports of Metals for Month ending 31st March.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
Couper:—			£	£
Ore .... Tons	4,317	5,661	40,198	61,601
Regulus .... „	6,053	12,184	147,541	296,590
Unwrought..... „	4,548	4,830	206,197	240,580
Iron:—				
Ore............ „	487,538	563,010	346,422	417,113
Bolt, bar, &c. ... „	3,955	2,670	34,109	21,411
Steel, unwrought.. „	754	2,410	6,149	14,390
Lead, pig and sheet „	17,044	13,800	190,414	160,519
Pyrites .... „	56,959	63,740	94,261	108,241
Quicksilver .... Lb.	24,580	391,460	2,389	37,850
Silver ore....... Value £	..	..	76,892	129,358
Tin .... Cwt.	73,175	90,767	221,678	271,951
Zinc .... Tons	4,225	6,177	78,418	107,456
Other articles ...Value £	..	..	158,198	192,953
Total value of metals	..	..	1,602,956	2,060,013

Imports of Chemicals and Dyestuffs for Month ending 31st March.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Alkali............ Cwt.	17,100	17,514	8,269	7,504
Bark (tanners', &c.) „	21,269	15,456	7,751	5,035
Brimstone .... „	15,036	47,320	3,506	10,673
Chemicals...... Value £	..	..	112,694	126,219
Cochineal .... Cwt.	94	78	608	483
Cutch and gambier Tons	2,508	2,634	62,818	49,786
Dyes:—				
Alizarin ....Value£	..	..	22,628	23,430
Anilin and other „	..	..	39,495	40,022
Indigo .... Cwt.	25,801	32,616	453,038	593,852
Nitrate of potash . „	30,490	45,470	26,705	34,025
Valonia .... Tons	3,302	1,625	36,337	16,507
Other articles... Value £	..	..	180,119	104,901
Total value of chemicals	..	..	953,968	1,012,437

Imports of Oils for Month ending 31st March.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Cocoa-nut......... Cwt.	22,230	7,115	24,740	7,652
Olive.............. Tuns	2,809	1,195	87,661	40,084
Palm .... Cwt.	94,456	69,597	97,878	72,209
Petroleum .... Gall.	10,554,270	11,054,302	228,227	210,282
Seed .... Tons	2,536	2,669	54,544	58,625
Train, &c. .... Tuns	774	3[illegible]0	12,997	7,564
Turpentine .... Cwt.	858	8,306	609	8,309
Other articles .. Value £	..	..	74,937	72,053
Total value of oils...	..	..	581,593	476,778

Imports of Raw Material for Non-Textile Industries for Month ending 31st March.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Bark, Peruvian .. Cwt.	4,514	1,950	6,999	3,813
Bristles.......... Lb.	295,152	221,189	37,063	35,735
Caoutchouc ...... Cwt.	55,314	50,023	686,558	627,198
Gum:—				
Arabic......... "	9,268	8,587	23,483	16,293
Lac, &c. .... "	23,861	20,468	116,394	100,225
Gutta-percha .... "	4,132	2,734	37,655	25,480
Hides, raw:—				
Dry............ "	34,841	49,888	89,676	127,537
Wet .... "	27,904	51,751	59,143	103,905
Ivory .... "	553	567	24,614	24,882
Manure:—				
Guano .... Tons	1,993	983	11,977	5,736
Bones .... "	7,876	6,789	30,456	29,008
Nitrate of soda... "	26,057	12,817	206,766	102,136
Phosphate of lime "	28,124	23,929	50,583	38,027
Paraffin.......... Cwt.	51,393	67,320	51,388	61,429
Linen rags....... Tons	2,630	2,069	22,543	21,135
Esparto.......... "	20,419	25,056	85,667	99,097
Pulp of wood .... "	19,575	25,855	96,092	130,091
Rosin............ Cwt.	116,723	69,387	30,269	18,239
Tallow and stearin ..	239,934	182,533	269,638	175,747
Tar ....Barrels	739	752	274	157
Wood:—				
Hewn .... Loads	137,874	136,051	250,696	262,682
Sawn .... "	101,958	114,237	237,662	294,903
Staves......... "	5,886	7,816	29,346	43,818
Mahogany ..... Tons	3,768	6,692	35,937	60,963
Other articles....Value £	..	..	839,316	973,144
Total value	..	..	3,330,295	3,381,380

Besides the above, drugs to the value of 73,983*l.* were imported, as against 84,899*l.* in March 1896.

Exports of Metals (other than Machinery) for Month ending 31st March.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Brass............ Cwt.	10,894	11,823	42,337	49,743
Copper:—				
Unwrought.... "	52,669	26,603	129,296	70,204
Wrought ...... "	32,562	27,599	88,145	86,730
Mixed metal... "	24,197	17,649	51,918	44,261
Hardware...... Value £	..	..	180,256	198,265
Implements ..... "	..	..	117,615	119,450
Iron and steel ... Tons	263,341	351,144	1,862,020	2,290,506
Lead .... "	2,632	2,521	35,061	34,703
Plated wares ... Value £	..	..	30,901	32,926
Telegraph wires . "	..	..	21,238	139,642
Tin .... Cwt.	9,992	5,526	32,357	17,859
Zinc .... "	19,268	10,247	13,258	8,290
Other articles .. Value £	..	..	80,014	77,376
Total value	..	..	2,684,456	3,169,955

Exports of Drugs and Chemicals for Month ending 31st March.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Alkali............ Cwt.	449,030	474,790	109,772	117,538
Bleaching materials ..	97,878	87,479	34,872	28,532
Chemical manures Tons	40,893	45,561	235,875	240,321
Medicines....... Value £	..	..	98,865	106,137
Other articles... "	..	..	388,190	437,568
Total value	..	..	867,574	930,096

Exports of Miscellaneous Articles for Month ending 31st March.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Gunpowder...... Lb.	680,400	566,700	18,673	13,589
Military stores.. Value £	..	..	126,553	91,913
Candles.......... Lb.	1,756,000	2,284,500	28,527	31,548
Caoutchouc ..... Value £	..	..	107,961	109,458
Cement.......... Tons	30,552	40,733	50,553	67,113
Products of coal Value £	..	..	194,135	162,083
Earthenware ... "	..	..	150,115	167,914
Stoneware ...... "	..	..	11,417	17,017
Glass:—				
Plate........... Sq. Ft.	188,940	82,964	8,282	5,749
Flint........... Cwt.	8,926	8,383	19,121	19,965
Bottles......... "	61,518	72,530	28,905	33,472
Other kinds.... "	11,230	18,006	11,534	14,039
Leather:—				
Unwrought .... "	12,030	13,593	113,716	128,927
Wrought ..... Value £	..	..	34,224	32,690
Seed oil.......... Tons	4,869	6,192	93,803	104,162
Floorcloth ..... Sq. Yds.	2,166,300	2,623,500	82,189	104,869
Painters' materials Val. £	..	..	139,996	150,261
Paper .... Cwt.	95,266	93,639	146,478	140,417
Rags............ Tons	5,147	4,697	27,192	25,961
Soap .... Cwt.	65,634	65,947	68,081	67,682
Total value	..	..	2,931,006	3,073,757

Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—PLANT, APPARATUS, and MACHINERY.

Applications.

6916. P. Jensen.—From G. Lunge and L. Rohrmann. Improvements in Glover towers, Gay-Lussac towers, and the like, used, for instance, in the manufacture of sulphuric, muriatic, and nitric acids. March 16.

7071. C. W. Ingraham and P. B. McSwords. Carbureter. March 18.

7108. W. P. Thompson.—From A. Testelin. Improved process and apparatus for spraying, distilling, evaporating, or concentrating liquids. Complete Specification. March 18.

7127. P. Jensen.—From L. Rohrmann and H. H. Niedenfuhr. Improvements in Glover towers and the like. Complete Specification. March 18.

7133. J. Gwynne. Improved method and machinery or apparatus for cooling liquids. March 18.

7331. J. A. Smith.—From B. L. Ryder. Improvements in apparatus for drying fruit and other organic matter. March 20.

7534. H. A. Wheeler. A smokeless furnace. Complete Specification. March 23.

7547. H. H. Schou. Improvements in refrigerating apparatus. March 23.

7664. H. Higgins. Improvements in or relating to drying and sterilising. March 24.

7667. H. Higgins. Improvements in and relating to sterilising apparatus. March 24.

7668. H. Higgins. Improvements in or relating to sterilising apparatus. March 24.

7888. E. Fournier. Improvements in or relating to apparatus for use in sterilising, disinfecting, and similar purposes. Complete Specification. March 26.

8018. J. Grossmann. *See* Class IV.

8234. T. S. C. Lowe and J. Haug. Improvements in coke, gas, and metallurgical furnaces, and processes connected therewith. Complete Specification. March 30.

8247. J. Laidlaw and J. W. Macfarlane. Improvements in centrifugal machines. March 31.

8393. W. McG. Greaves. Improvements in water-heaters and superheaters. April 1.

8414. A. H. Crockford and A. S. Vose. Improvements in pressure and vacuum gauges. April 1.

8456. J. Muir, E. A. Morse, and F. O. Pierson. Refrigerating apparatus. Complete Specification. April 2.

8482. W. Tattersall. Improvements in heating or cooling apparatus. Complete Specification. April 2.

8506. F. Wippermann. Improvements in drying apparatus. April 2.

8596. T. C. Palmer. Improvements in and connected with condensers. April 3.

8775. N. S. Jenkins. Improvements in melting apparatus. April 6.

8976. L. Couher. Improved means applicable for use in storing sulphuric acid and facilitating its transport. April 8.

9042. T. Fletcher and Fletcher, Russell, and Co., Ltd. Improvements in furnaces for use with light hydrocarbons as fuel. Complete Specification. April 9.

9212. M. Graham. Improvements in or relating to apparatus for charging gas and other retorts. April 10.

Complete Specifications Accepted.*

1896.

6377. D. Adorjan. Apparatus for producing highly overheated steam and vapour. March 31.

7868. T. C. Sanderson. Apparatus for drying white lead, colours, and other powdery, granular, and nodular substances. March 31.

7997. W. Shedlock. Evaporating apparatus, chiefly designed for obtaining salt from brine. March 24.

12,557. S. J. Norton and H. A. Stenning. Apparatus for compressing air and other gases. April 14.

16,196. J. G. Chamberlain and J. Weddell. Evaporative surface condensers. April 7.

19,290. A. J. Boult.—From O. Patin. Electrical furnaces. April 14.

29,587. E. Gauhe and Gockel. Mixing drums. April 7.

1897.

189. W. L. Gilder. Furnaces. March 31.

1580. J. Thomas and L. P. Thomas. Presses suitable for the treatment of oil-cake and other materials. March 31.

4249. J. Bryson. *See* Class III.

II.—FUEL, GAS, AND LIGHT.

Applications.

6779. M. A. A. E. Boulaine. Process of manufacturing a material suitable for production of acetylene. March 15.

6797. A. Ehrenwerth. Process for the manufacture of incandescent light. March 15.

6940. H. A. Kent. Improvements in inverted incandescent gas lights, and attachment of incandescing material to same. March 17.

6997. G. Pereire, E. Sorel, and B. Cruvellier. Improvements in the manufacture of acetylene gas, and in apparatus therefor. Complete Specification. Filed March 17. Date applied for Nov. 5, 1896, being date of application in France.

7013. L. L. Merrifield. Improvements in apparatus for the manufacture of carburetted water-gas. March 17.

7043. T. Lyons, J. Lyons, and J. Pearson. Improvements in acetylene gas generators. March 18.

7168. F. S. Thorn and C. Hoddle. Improvements in acetylene gas generators. March 19.

* *See* Note (*) on previous page.

7172. Comte Albert Dillon de Micheroux. New process to desulphurate coals, to destroy their smoke and bad smell, to increase the number of their calories, &c. March 19.

7177. E. Bohm. Improvements in and in means for the production of ozone. March 19.

7295. P. L. Caron. Improved apparatus for producing acetylene gas. March 20.

7296. P. Guerard. Apparatus for the automatic production of acetylene gas. March 20.

7330. R. Schoder. A process for stiffening incandescence mantles. March 20.

7435. C. Billington, jun. An improved method of and means for removing "scurf" or retort carbon from gas retorts. March 23.

7651. A. Arter. Improvements in apparatus for the treatment of gas for illuminating and other purposes. March 24.

7744. E. G. Brewer.—From The Gesellschaft für Acetylen-Gaslicht Basel. Improvements in apparatus for generating acetylene gas. Complete Specification. March 25.

7782. H. H. Lake.—From F. Ferracciu. Improvements in apparatus for automatically generating acetylene gas. Complete Specification. March 25.

7858. E. S. Higgins, H. A. Bessemer, and W. B. Nicholson. Improvements relating to the production of gas and its utilisation for the generation of steam and for other purposes, and to apparatus therefor. March 26.

7918. J. G. A. Kitchen. Improvements in or relating to acetylene generators. March 27.

7920. J. B. de Lery. Improvements in the method of and apparatus for effecting illumination partly by flame and partly by incandescence. Complete Specification. March 27.

7921. J. B. de Lery. Improvements in and connected with burners for incandescence gas lighting. Complete Specification. March 27.

7929. F. H. Smith. An acetylene gas lamp or generator. March 27.

7956. W. B. Hartridge. Improvements in the manufacture of artificial fuel blocks, and apparatus for that purpose. March 27.

7991. A. A. Stephenson. An improved composition and apparatus for enriching gas. March 29.

8007. R. Dixon. Improvements in or relating to the manufacture of coke. March 29.

8108. J. G. A. Kitchen. Improvements in or relating to acetylene generators. March 30.

8270. J. G. A. Kitchen. Improvements in and relating to acetylene generators. March 31.

8332. A. E. Yeadon and S. N. Yeadon. Improvements in the manufacture of briquettes or compressed blocks for fuel smelting or other analogous purposes. April 1.

8359. C. W. Kemp. Improvements in or relating to incandescent gas lighting. April 1.

8363. W. C. Perrins. Improvements in gas lighting. April 1.

8485. E. Bouchaud-Praceiq. Improvements in apparatus for carburetting air. April 2.

8513. J. A. Bellon-Lencauchez. Improvements relating to the production and purification of gas, and apparatus therefor. April 2.

8551. J. H. Exley. Improvements in apparatus for manufacturing acetylene gas. April 3.

8552. H. Cousin. Improvements in apparatus for manufacturing acetylene. Complete Specification. April 3.

8583. W. Blackie. Improvements in the manufacture of oxygen gas, and apparatus therefor. April 3.

8721. V. Menne. Improved acetylene gas generator. April 6.

8759. D. Meyer. Improvements in incandescent gas burners. Complete Specification. April 6.

8793. W. P. Barltrop. An improved apparatus for the production of acetylene gas. April 6.

8989. J. G. Kerr and C. Fry. Improved apparatus for the production and storage of acetylene gas. Complete Specification. April 8.

9019. D. A. Casalonga. Improved method and plant or apparatus for the treatment of wood for the manufacture of charcoal. April 8.

9045. C. O. Rusling. Improvements in protecting and sustaining the mantles of "Welsbach" and similar lights. April 9.

9126. C. H. Stearn. Improvements in the manufacture of filaments for incandescence lamps. April 9.

9175. F. Wilkins. Improved apparatus and fittings for impregnating atmospheric air with pure volatile hydrocarbon in substitution of gas for producing light, heat, or power. April 10.

Complete Specifications Accepted.

1896.

6349. H. Hart. Illuminating apparatus. March 31.

6852. H. C. B. Forester. Apparatus for pressing artificial or patent fuel or the like into solid form. March 31.

6853. H. C. B. Forester. Manufacturing artificial or patent fuel. March 31.

6922. V. B. Lewes. Manufacture or production of illuminating gas. March 31.

9241. W. Nicholls. Manufacture of incandescence bodies for illuminating purposes. April 14.

9316. C. E. Botley and C. F. Botley, jun. Treatment of gas for illuminating and other purposes. March 31.

11,737. E. Clausolles. Apparatus for the production and storage of acetylene gas for illuminating purposes. March 31.

12,788. R. R. Gibbs. Generation and storage of acetylene. March 31.

15,654. D. Whalley and J. Hacking. Acetylene gas store generators. March 24.

16,277. L. V. Pratis and P. Marengo. Production of pure hydrogen gas and the application of hydrogen to the obtainment of light, heat, and power. March 31.

16,277A. L. V. Pratis and P. Marengo. The utilisation of hydrogen gas for the obtainment of light, heat, and power. April 14.

20,529. G. S. T. Gastine. Apparatus for the production of acetylene. March 24.

23,592. G. Trouve. An acetylene lamp. March 24.

24,558. H. H. Lake.—From V. Hanotier and G. Hostelet. Lamps adapted for producing and burning acetylene gas. March 31.

26,261. J. C. Mewburn.—From F. G. Bates. Manufacture of combustible gas, including illuminating gas in motor engines driven by such gas and in steam generators. March 24.

29,202. D. Nagy. Process for making brown coal and lignite coke. March 24.

29,863. C. Dellwik. Production of water-gas and apparatus therefor. March 24.

1897.

911. V. Daix. Production of acetylene gas. April 14.

1153. E. H. F. des Essards. An improved generator for acetylene and certain other gases. April 14.

1440. A. J. Boult.—From H. F. Fuller. Apparatus for the generation of gas. March 31.

2284. R. F. Carter. Apparatus for the production and storage of acetylene gas. April 7.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Applications.

7233. G. W. Johnson.—From Kalle and Co. Improvements in the manufacture of guaiacol. March 19.

9019. D. A. Casalonga. *See* Class II.

Complete Specification Accepted.

1897.

4219. J. Bryson. Retorts for the distillation of shale and other bituminous substances, or for the calcining of ironstone, lime, and other substances. March 31.

IV.—COLOURING MATTERS and DYES.

Applications.

6913. H. R. Vidal. An improved manufacture of colouring matters. Filed March 16. Date applied for March 11, 1897, being date of application in France.

7213. H. H. Lake.—From The Farbwerk Muhlheim vormals A. Leonhardt and Co. Improvements in the manufacture of sulpho acids of the naphthalene series and of colouring matters derived therefrom. March 19.

7329. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of new safranine derivatives. March 20.

7337. S. Pitt.—From L. Cassella and Co. Improvements in the production of azosafranine derivatives. March 20.

7341. J. Y. Johnson.—From C. F. Boehringer and Soehne. Improvements in the preparation of alkylated uric acids. March 20.

7704. W. H. Claus and A. Ree. Manufacture of brown colouring matters containing sulphur. March 25.

8018. J. Grossmann. Apparatus for testing indigos and other dyes and colours. March 29.

8175. G. B. Ellis.—From La Société Chimique des Usines du Rhone, anciennement G. P. Monnet et Cartier. Improvements in the manufacture of colouring matters. March 30.

8188. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Improvements in the manufacture or production of colouring matters. March 30.

8235. G. W. Johnson.—From Kalle and Co. Improvements in the manufacture and production of azo dyestuffs or colouring matters suitable for dyeing cotton. March 30.

8383. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of new safranine derivatives. April 1.

8569. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Improvements in the manufacture or production of colouring matters. April 3.

8994. A. M. Clark.—From Kullmann and Rapp. A process for the production of new tetrazo-azo compounds. April 8.

9092. M. N. J. Rigby. An instantly drying ink. April 9.

9213. H. R. Vidal. Improvements in the preparation of diphenyl, dinaphthyl, and their derivatives. April 10.

Complete Specifications Accepted.

1896.

7596. Levinstein, Ltd., and I. Levinstein. Manufacture or production of new colouring matters. April 14.

8860. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Process for discharging para-nitroaniline red fabrics, white and coloured, and manufacture of a discharging colour therefor. March 31.

9343. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of basic disazo dyestuffs. March 24.

11,309. Read, Holliday, and Sons, Ltd., and H. Dean. Manufacture of basic colouring matters. April 14.

18,489. H. R. Vidal. Manufacture of sulphuretted colouring matters. March 24.

20,250. B. Willcox.—From The Badische Anilin and Soda Fabrik. The manufacture and production of colouring matters from dinitronaphthalenes. April 14.

1897.

1145. H. Imray.—From F. Peterson and Co. Manufacture of new colouring matters from benzidin sulpho acids. March 31.

V.—TEXTILES, COTTON, WOOL, SILK, Etc.

Applications.

6768. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. A method of improving cotton fibre. March 15.

7093. C. D. Abel. — From J. Kleinewefers Sohne. Process and apparatus for mercerising cotton in the form of hanks. March 18.

8460. K. T. Sutherland. Improvements connected with the removal of gum and such like from fibres. April 2.

9056. E. Crepy. Improvements in the method of and means for "mercerising" vegetable fibres, fabrics, and the like. April 9.

Complete Specification Accepted.

1896.

12,897. L. Frankenstein. Finishing or ornamenting the surface of waterproof fabrics. April 7.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

Applications.

7622. J. Tait. *See* Class XIX.

7821. S. Schwabe and Co., Ltd., A. Binz, and R. Boral. Improvements in printing textile fabrics which may be subsequently dyed. March 26.

8033. W. Watson and E. Bentz. Improvements in the production of coloured discharge patterns on indigo-dyed cotton fabrics in calico-printing. March 29.

8114. O. H. Stone. A new or improved process of colouring papers. March 30.

9011. L. Koenig, jun. Improved appliance for dyeing and bleaching textile staples, filaments, or fibres, on tubes and bobbins. April 8.

Complete Specifications Accepted.

1896.

7977. H. W. V. Wilson and W. Brown. Apparatus for varnishing or otherwise coating paper, cardboard, or other material. April 7.

8311. L. C. G. Lesage. Process and apparatus for use in colour printing. April 7.

VII.—ACIDS, ALKALIS, and SALTS.

Applications.

6916. P. Jensen.—From G. Lunge and L. Rohrmann. *See* Class I.

7066. W. Garroway. Improvements in the manufacture of ammonium nitrate and other salts. March 18.

7192. A. Schmidt. Process and apparatus for the production of the acetates of lead, copper, and the like. Complete Specification. March 19.

7715. J. McGougan and A. McGougan. Improved process for the production of nitrites of the alkali or alkaline earth metals. March 25.

8094. B. C. Hinman. A new manufacture of a salt or compound containing bromine. March 29.

8220. S. Bourgois, A. Lencauchez, L. Hoche, and E. Isidor. Improvements in or relating to apparatus for the recovery of ammonia and other products from gases. March 30.

8335. W. Wyld and J. H. Cox. Improvements in the means or method of manufacturing ferric sulphate. April 1.

8671. B. C. Hinman. A new manufacture of a salt or compound containing bromine. April 5.

8976. L. Coulier. *See* Class 1.

9099. T. Savage. Improvements in the production of nickel sulphate. April 9.

9161. A. J. Bambridge and G. Weddell. Improvements in the manufacture of bicarbonate of soda and of carbonate and bicarbonate of ammonia. April 10.

9215. H. R. Vidal. An improved process for manufacturing alkaline cyanides and other cyanated products. April 10.

Complete Specifications Accepted.

1896.

10,252. D. A. Peniakoff. A process for obtaining and extracting caustic alkalis from alkaline aluminates, and hydrates of barium and alumina from barium aluminate. April 14.

15,834. A. J. Outhenin-Chalandre. Electrolysis of soluble salts, especially applicable for the production of chlorine. April 7.

VIII.—GLASS, POTTERY, and ENAMELS.

Applications.

6840. A. D. Brogan, A. M. Malloch, and J. Craig. Improvements in and relating to leers or kilns for annealing glass. March 16.

8494. L. Mondron. Improvements in the manufacture of plates of glass designed for covering walls, ceilings, and other surfaces. Complete Specification. April 2.

Complete Specifications Accepted.

1896.

7899. R. Stanley. Ovens for drying and burning tiles, pottery, and other plastic or semi-dry ware. April 14.

8148. The Coalport China Co., Ltd., and T. J. Bott. Ornamentation or decoration of china, earthenware, glass, and like ware. March 31.

10,471. W. West. Apparatus for the manufacture of moulded clay, earthenware, and ceramic articles. April 14.

15,804. J. Davis and A. J. Davis. Method of producing coloured glass lights for lenses, also applicable for other suitable purposes. April 7.

17,391. P. T. Sievert. Manufacture of sheets, cylinders, and other forms of glass. April 14.

1897.

334. G. G. M. Hardingham.—From G. W. Blair. Manufacture of glass articles. March 24.

1432. A. J. Boult.—From P. A. J. Gasse. Decoration of earthenware, china, glass tiles, bricks, and the like. April 7.

3376. E. W. Brock. New or improved process for transferring printed matter from paper to glass, earthenware, and other enamelled surfaces. April 7.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, and CEMENTS.

Applications.

7798. T. Parker.—From G. W. Parker. Improvements in the art, method, or process of treating gypsum rock for the production of artificial marble and the like. March 25.

8261. J. Howden. Improvements in fireproof floors. Complete Specification. March 31.

8268. J. Sayers. Improvements in the manufacture of artificial stone and the like. March 31.

9030. T. C. Hutchinson. An improved treatment of blast-furnace slag or scoria for the manufacture therefrom of hydraulic or similar cement, or for other purposes requiring use of dry granulated slag. April 9.

9072. D. Wilson. An improved cement or concrete. April 9.

9073. D. Wilson. Improvements in the manufacture of slabs for the beds of billiard tables and other purposes, steps and blocks for building and other purposes, pillars, columns, posts, poles, chimney pots, pipes, or tubes, baths, sinks, tanks, buckets, casks, drums, or barrels, and other hollow articles and vessels. April 9.

Complete Specifications Accepted.

1896.

9100. J. C. Nichol. Material to be used as a substitute for wood, and to be known as "fabricoid." April 14.

11,182. W. P. Thompson. Method of preparing or treating wood. March 31.

11,682. J. Dixon and A. Reynolds. Manufacture of silica bricks or other highly refractory bricks. April 14.

26,847. J. J. Moffat. Fireproof floors, ceilings, walls, and partitions. Nov. 26.

29,654. O. Kraner. Process for the production of a material for making floors, plasterings, ceilings, walls, roofs, and the like without joints or chinks. April 7.

1897.

2085. C. A. Allison.—From F. P. Stone. Process of solidifying wood. April 7.

2402. W. D. Cliff. Glazed bricks, and manufacture of the same. April 7.

2791. F. S. Winser. Method of manufacturing stoneware and the like. April 7.

3345. C. Lucke. Presses for forming artificial stones, slabs, tiles, and the like. April 7.

4048. W. S. Robinson and S. M. Brookfield. Cement and concrete. March 31.

X.—METALLURGY, MINING, Etc.

Applications.

6881. H. R. Cassel and B. C. Hinman. An improved process of and apparatus for extracting gold from its ores and compounds. March 16.

6890. W. H. Staliard.—From J. H. R. Whinfield. Improved method for treating iron and steel and other metals. March 16.

6991. H. Griffith, jun.—From V. Coppee and A. E. Kemplen. Improvements relating to aluminium. March 17.

6992. H. Griffith, jun.—From V. Coppee and A. E. Kemplen. Improvements relating to the casting of aluminium and other metals. March 17.

6993. H. Griffith, jun.—From V. Coppee and A. E. Kemplen. Improvements relating to the brazing of aluminium. March 17.

7021. La Société des Cuivres de France. Process and apparatus for obtaining pure copper in a single operation. Filed March 17. Date applied for Aug. 26, 1896, being date of application in France.

7198. J. Cope. Improvements in and relating to nickel or nickel-alloy castings. March 19.

7218. B. Becker. Improvements in extracting precious metals from minerals containing them, and apparatus therefor. March 19.

7234. S. O. Cower-Coles. Improvements in or connected with means for separating metals from cyanide solutions. March 19.

7532. E. A. Ashcroft. Improvements in the combined electrolytic and leaching treatment of zinc-bearing ores and zinc-bearing products. March 23.

7555. H. R. Cassel and B. C. Hinman. An improved process of and apparatus for extracting gold from its ores and compounds. March 23.

7756. W. J. May. Improvements in potassium fluxes for use in the manufacture and casting of iron or steel. March 25.

7757. W. J. May. Improvements in fluxes for use in the manufacture and casting of steel. March 25.

7758. W. J. May. Improvements in metallic sodium fluxes for use in the melting or working of metals. March 25.

7988. H. M. Martin. Improvements in the production of castings. March 29.

8040. A. Gilbertson and D. Jenkins. A new or improved method or process for the manufacture of sheets of iron, steel, or other material. March 29.

8273. J. Nicholas. Improvements in the recovery of metals contained in ores or other compounds. March 31.

8376. G. Kentler and F. Steinert. Process and apparatus for recovering iron from slag-heaps. Complete Specification. April 1.

8577. B. K. Jamison. Improvements in the manufacture or treatment of armour plates and in apparatus employed therein. April 3.

8578. B. K. Jamison. Improvements in the manufacture or treatment of steel or nickel steel, and of articles made from steel, and in apparatus employed therein. April 3.

8626. F. Moss and S. J. Davis. A new process for staining and colouring metals and for other suitable purposes. April 5.

8763. C. H. Foote, W. R. Walker, and E. A. S. Clarke. Improvements in the manufacture of steel by the Bessemer or open-hearth process. Complete Specification. April 6.

8873. W. Noad and W. Agate. An improvement in the obtainment of copper from ores and other copper-bearing substances. Complete Specification. April 7.

8887. O. Imray.—From The Pennsylvania Salt Manufacturing Co. Improvements in apparatus and appliances for casting metals. April 7.

8990. G. H. Blenkinsop. Improvements in or relating to the smelting of mixed minerals, ores, and the like containing lead and zinc. April 8.

Complete Specifications Accepted.

1896.

7315. J. E. Preston. Amalgamating apparatus. April 6.

7422. R. A. Hadfield. Manufacture of steel or alloy of iron. April 7.

7436. W. Gibbings. Blast furnaces. April 14.

8967. S. Vivian. Recovery of gold, silver, and other precious metals. March 24.

10,494. J. R. Wylde, J. W. Kynaston, and J. Brock. Extraction of zinc from ferruginous liquors, specially applicable to the treatment of the spent liquors of copper works. April 7.

10,864. B. Politzer. A process for protecting metallic surfaces against chemical and atmospherical action. March 24.

11,555. D. A. Peniakoff. A process of manufacture of sulphite of aluminium, which may or may not be combined with other sulphides. April 7.

12,782. A. Wilson and F. Stubbs. Carbonising iron and steel. April 14.

23,817. B. Meyer. Treatment of weldable iron. March 31.

30,144. A. F. Cothias. Process of casting for reducing the density of cast articles. March 31.

1897.

2789. H. S. Lloyd. Storage batteries. March 31.

4136. W. B. Johnson.—From The Pennsylvania Salt Manufacturing Co. Apparatus for casting metals. March 31.

4309. S. Neffgen. Process of treating granular or pulverulent iron ores and slag, or other residues containing iron, to facilitate the extraction of the same. March 24.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

Applications.

6818. J. Entwisle.—From C. T. Barret. An improved storage battery. Complete Specification. March 16.

6929. F. Taylor, A. Cooke, and B. W. D. Montgomery. An improved diaphragm for separating the gases in electrolysis of sodium chloride and other salts, and other improvements. March 17.

7024. G. W. de Tunzelmann and C. W. Baker. Improvements in electric welding. March 17.

7027. A. J. Marquand. Improvements in and relating to secondary batteries or accumulators, and the manufacture thereof. March 17.

7113. F. H. Perry. Improvements in primary batteries. March 18.

7314. F. E. Everard. Improvements in or relating to accumulator plates or electrodes for secondary batteries. March 20.

7775. W. J. S. Barber-Starkey. Improvements in or relating to electrodes for secondary batteries. March 25.

7958. E. A. Jahncke. Improvements in and connected with electric batteries. March 27.

8059. D. G. Fitz-Gerald. Improvements in elements for secondary batteries and electrodes for electrolysis. March 29.

8131. S. V. B. Essick. Improvements in primary electric batteries or generators. Complete Specification. March 30.

8231. G. F. Redfern.—From C. Riordon. Improvements in storage batteries. Complete Specification. March 30.

8537. H. Stanhope. Improvements in or connected with electric batteries. April 3.

8696. A. P. Laurie. Improvements in secondary batteries or storage cells. April 6.

9118. L. Epstein. Improvements in secondary voltaic batteries. April 9.

Complete Specifications Accepted.

1896.

8080. E. Commelin and R. Viau. An improved electrical accumulator under pressure. April 14.

10,472. G. A. Grindle. An improvement in active material for secondary voltaic batteries. March 31.

10,718. H. O. Tudor. Manufacture of positive lead electrodes for electrical accumulators. April 7.

11,212. T. Clark. Dry cells for galvanic batteries. April 7.

11,730. W. P. Thompson.—From The Elektra Galvanoplastische Anstalt, H. Teith, and A. Floeck. Apparatus for producing galvanic or electrolytic depositions. April 14.

11,732. E. Grunwald. Plate for storage batteries. April 14.

19,218. E. J. Constam and A. von Hansen. Manufacture or production of chemical products by electrolysis. April 14.

25,360. F. H. Snyder. Improvements relating to the coating, covering, or amalgamating of metals with metals, and of other substances with metals and other materials, by the aid of electricity, and to apparatus therefor. March 24.

27,262. O. Rothmund, E. von Burgwall, and L. Ofenschussl. An improved primary element with interchangeable electrodes and regenerable positive electrodes. March 31.

29,306. A. Heil. Secondary batteries. March 24.

1897.

859. Aschermann. Process for the extraction of metallic alloys and pure metals by electric heat. March 31.

995. W. S. Doe. Galvanic batteries. March 31.

XII.—FATS, OILS, and SOAP.

Applications.

7228. P. Villain. Improvements in the manufacture of scented antiseptic, medicinal, or multi-coloured or variegated soap, and in machinery or apparatus therefor. March 19.

8560. T. McKinnon. Improvements in or relating to soaps or other like preparations. April 3.

8796. E. G. Scott. Improvements in and connected with the saponification of fatty matters and in the manufacture of glycerine. April 6.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

7014. N. W. Meadows. A process for reclaiming india-rubber from fabrics coated on both surfaces. March 17.

7399. E. O. S. Gentele and H. R. Gentele. Improvements in and in the manufacture of pigments, printing inks, or paint mixtures. Complete Specification. March 22.

8479. J. F. Spong. Improvements in compounds of india-rubber. April 2.

8510. C. A. Hakanson, J. H. Hakanson, and A. R. Hakanson. Paint or composition to be used as a coating, more especially in the manufacture of artificial slates and for coating tiles, plates, and other articles. April 2.

8811. R. Hingston and J. C. Rankin. An improved anti-fouling and anti-corrosive paint. Complete Specification. April 6.

Complete Specifications Accepted.

1896.

12,451. E. Cadoret. Plastic and incombustible material—textiloid—to be used as a substitute for india-rubber, celluloid, leather, oil-cloth, linoleum, mother-of-pearl, tortoise-shell, amber, ivory, and the like, and capable of being moulded, drawn or made into threads, and in the form of plates, tubes, cylinders, or soft or silky threads resembling silk in appearance. April 7.

26,548. F. Fenton. Production of artificial gutta-percha and caoutchouc-like substances. March 31.

XIV.—TANNING, LEATHER, GLUE, and SIZE.

Applications.

6769. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. *See* Class XXI.

7270. E. White. Improvements in apparatus for tanning hides and skins. March 19.

8042. A. Mitscherlich. Process for the manufacture of glutinous substances. March 29.

8391. A. Zimmermann. Improvements in the treatment of gelatinous substances, skins, and the like. April 1.

8632. G. Honeyman, S. Honeyman, and R. Arneil. An improved method for the manufacture of chrome-tanned leather. April 5.

9106. E. O. Daniel. Improvements in the preparation for and tanning of hides and skins. April 9.

Complete Specifications Accepted.

1896.

13,686. H. Schmidt and J. Landini. Arrangement for performing a continuous work of tanning. April 14.

1897.

5762. A. J. Boult.—From G. L. Lippold. Improved substitute for leather or like material, and the method of manufacturing the same. April 14.

XV.—AGRICULTURE AND MANURES, Etc.

Complete Specification Accepted.

1895.

9789. F. M. Mooney and A. Dickson. Manufacture and storing of superphosphates and the like. April 14.

XVI.—SUGARS, STARCHES, GUMS, Etc.

Applications.

7861. M. Sachs. Processes for separating impure sugar at one operation into crystallised sugar and molasses. March 26.

9108. A. J. Boult.—From C. M. Higgins. Improvements in or relating to adhesive compounds. Complete Specification. April 9.

Complete Specification Accepted.

1897.

1144. A. Classen. Manufacture of chemical compounds of starch and dextrine, or equivalent bodies, with formaldehyde or its equivalents. March 24.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

8080. B. J. B. Mills.—From A. Dalmote. An improved diastasic preparation to be employed in the manufacture of bread and other foods and beverages, and to be used as an aid to digestion. March 29.

8223. J. G. Hathaway. An improved method of and apparatus for purifying and maturing alcoholic liquors. March 30.

9018. O. Trachmann. Process and product for the preparation of certain kinds of beer. April 8.

Complete Specifications Accepted.

1896.

13,262. B. W. Valentin. Manufacture of material for use in brewing or priming ales and beers. April 7.

29,965. J. Y. Johnson.—From The Electric Rectifying and Refining Co. Apparatus for treating or purifying, rectifying, ageing, deodorising, and decolorising alcoholic and other liquids or liquefied matter. March 31.

1897.

2434. J. Sinclair. Improved valve fittings for use in fermenting vats or stone squares. April 14.

XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

Applications.

A.—Foods.

6915. K. I. O'Doherty. An improved method of preventing the decomposition of animal, vegetable, or other alimentary substances or products. Complete Specification. Filed March 16. Date applied for Aug. 27, 1896, being date of application in Queensland.

7220. A. S. Peyser. A method of treating milk and obtaining therefrom a gelatinous proteid substance and thus forming a nourishing food for invalids and others. March 19.

7649. K. Franz. A new or improved food preparation and process for the manufacture thereof. Complete Specification. March 24.

8052. W. P. Thompson.—From E. O. Taflin. Improved substitute for coffee and process of making the same. March 29.

8080. B. J. B. Mills.—From A. Dalmote. See Class XVII.

8586. T. Hyatt. Improvements in the treatment and utilisation of chicory. Complete Specification. April 3.

B.—Sanitation.

7436. E. Phillips. Improvements in the disposal of sewage, and in the collection and storage of gas given off in such process. March 23.

8281. G. A. de Katow.—From Riensch and Co. A new or improved process and apparatus for purifying and utilising town sewerage. March 31.

8375. P. J. Frub. Process and apparatus for removing all dirtying ingredients in the chimney smoke. April 1.

8500. W. M. Ducat. An improvement in or connected with the purification of water sewage and other foul liquids. April 2.

C.—Disinfectants.

6823. J. L. Mortimer and J. H. Williams. A new and improved disinfecting and antiseptic block. March 16.

Complete Specifications Accepted.

A.—Foods.

1896.

7042. H. Higgins. Preservation of foods and other perishable goods. March 31.

7880. R. Haddan.—From Kathreiners Malzkaffee Fab. mit beschränkter Haftung. Process for producing coffee substitutes for grain. April 7.

11,060. J. Davidson. Manufacture of products from tea. March 24.

23,400. H. H. Lake.—From A. Astolfi and E. Brugnatelli. Improved alimentary substance. April 7.

25,295. H. D. Perky. Method and means for the reduction of grain to thread-like or shredded form, and for the disposition of the same in the manufacture of biscuit and other forms of bread. March 24.

27,114. S. P. Sorenson and A. P. Heyman. Manufacture of artificial food for swine, cattle, and the like. March 24.

B.—Sanitation.

1896.

11,243. E. W. T. Jones and J. Garfield. Purification of sewage and other liquids. April 7.

15,208. H. W. Kearns. Apparatus or means for treating the sludge or deposit from foul waters and the like. March 24.

23,676. S. de Petroff. A new or improved method for the treatment of town sewage, refuse, and the like, for the destruction of animalculæ and the production of a fertilising compound. April 7.

C.—Disinfectants.

1896.

1978. S. Wohle and A. C. Irwin. Manufacture of detergent. April 7.

XIX.—PAPER, PASTEBOARD, Etc.

Application.

7622. J. Tait. Improvements in bleaching and purifying paper pulp. March 24.

COMPLETE SPECIFICATIONS ACCEPTED.

1896.

210. H. H. Lake.—From E. Berland and J. Girard. Manufacture of paper pulp from the broom plant. April 7.

9100. J. C. Nichol. *See* Class IX. April 14.

12,682. W. A. Kershaw and The Medway Paper Mills Co., Ltd. Manufacture of multiple-pulp papers. April 14.

1897.

1550. P. H. Hansen and J. K. Westengaard. Manufacture of artificial horn and the like. March 24.

4099. A. A. Hunting and E. A. Leigh. Method and apparatus for drying paper and similar materials. April 7.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

APPLICATIONS.

6790. E. H. C. Durkopf. Improvements in the manufacture of products of condensation from formaldehyde and tannin. March 15.

7341. J. Y. Johnson.—From C. F. Boehringer and Soehne. *See* Class IV.

8026. C. Castel. Improvements in the manufacturing of acid ethyl-sulphuric and the production of alcohol. March 29.

8308. F. Richard. Improvements in vaporising apparatus suitable for converting methyl alcohol into formic aldehyde. March 31.

8387. H. Mack. Method of producing highly concentrated fat solutions of fragrant substances. Complete Specification. April 1.

8671. B. C. Hinman. *See* Class VII.

8915. J. Y. Johnson.—From C. F. Boehringer and Soehne. Method of preparing alkylated dichloroxypurines. April 7.

COMPLETE SPECIFICATIONS ACCEPTED.

1896.

7712. A. Classen. Production of formaldehyde compounds of the albumen and protein bodies. March 3.

9576. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Manufacture of a pharmaceutical substance. March 31.

10,003. J. Y. Johnson. — From C. Boehringer and Soehne. Manufacture or production of cinnamic aldehyde. March 31.

10,005. E. Fischer. A method of preparing alkylated uric acids. April 14.

11,061. J. Turner and Co., Ltd., and P. Schidrowitz and O. Rosenheim. Manufacture of derivatives from piperdine, tetra-hydro-quinoline, and their homologues. April 7.

24,568. A. Blaile. Process for the manufacture of artificial musk. April 14.

1897.

1164. W. L. Wise.—From The Chemische Fabrik von Heyden Gesellschaft mit beschränkter Haftung. Manufacture of *o*-sulphamin benzoic acid alkylesters and of anhydro-*o*-sulphamin benzoic acid. March 31.

XXI.—PHOTOGRAPHY.

APPLICATIONS.

6704. J. Joly. Improvement in or relating to photography in colours. March 15.

6769. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of prepared sensitised leather for photographic purposes. March 15.

COMPLETE SPECIFICATIONS ACCEPTED.

1896.

18,131. J. A. Michel-Dansac and L. Chassagne. Means for reproducing the colours in photography, suitable also for dyeing and printing. March 31.

1897.

5026. A. Rateau. Chromo-photographic apparatus. April 7.

XXII.—EXPLOSIVES, MATCHES, ETC.

APPLICATIONS.

6772. F. Müller, S. Oberlander, V. H. Fuchs, and S. Gomperz. Improved blasting compound, and process for its manufacture. March 15.

6995. B. E. Dickinson and Kynoch, Ltd. Improvements in fog signals. March 17.

8555. J. Nicholas. Improvement in explosive compounds. April 3.

9111. W. Greaves and E. M. Hann. Improvements in or relating to explosives. April 9.

COMPLETE SPECIFICATIONS ACCEPTED.

1896.

14,007. F. M. Hale and J. B. Reavil. Percussion fuses for explosive projectiles or shells. April 14.

1897.

1154. F. W. Jones. Manufacture of explosives. March 24.

XXIII.—ANALYTICAL CHEMISTRY.

COMPLETE SPECIFICATION ACCEPTED.

1896.

10,627. R. Wells. Apparatus for testing butter, margarine, and the like, and method of employing such apparatus. March 31.

PATENT UNCLASSIFIABLE.

APPLICATION.

8227. G. L. Schaefer. Improvements in chemical compounds. Complete Specification. March 30.

Printed and Published by EYRE and SPOTTISWOODE, East Harding Street, London, E.C., for the Society of Chemical Industry.

THE JOURNAL

OF THE

Society of Chemical Industry.

A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

No. 5.—Vol. XVI.] **MAY 31, 1897.** [Non-Members 30/- per annum; Members 21/- per Set of extra or back numbers; Single Copies (Members only) 2/6.

NOTICES.

Annual General Meeting.

The Annual General Meeting will be held in Manchester on the 14th, 15th, and 16th July next. The programme and application form for tickets will be found in this number. Tickets of Membership will be issued in time for the Meeting, and will form, as heretofore, vouchers for visits to works and excursions.

In accordance with the provisions of Rule 18 of the Bye-laws, notice is hereby given that those Members whose names are placed in italics in the list of Council will retire from their respective offices at the forthcoming Annual General Meeting.

Prof. F. Clowes, D.Sc., has been nominated to the office of President; Dr. Edward Schunck, F.R.S., has been nominated Vice-President under Rule 11; Mr. A. Gordon Salamon and Dr. Wm. Jay Schieffelin have been nominated Vice-Presidents under Rule 24; and Mr. D. Howard, Mr.

Ivan Levinstein, and Mr. Wm. Thorp have been nominated Vice-Presidents under Rule 8.

The Hon. Treasurer and Hon. Foreign Secretary have been nominated for re-election to their respective offices.

Members are hereby invited to nominate fit and proper persons to fill four vacancies among the Ordinary Members of Council under Rule 18. Nomination forms for this purpose can be obtained from the General Secretary upon application.

Extract from Rule 18:—"No such nomination shall be valid unless it be signed by at least ten Members of the Society who are not in arrear with their subscriptions, nor unless it be received by the General Secretary, at the Society's Office, at least one month before the date of the Annual General Meeting, to the election to take place at which it refers. Nor shall any such nomination be valid if the person nominated be ineligible for election under Rules 12 or 15. No member shall sign more than one nomination form."

An international congress on technical education will be held in London during the month of June. The congress will be opened on June 15th, and will close on Friday, June 18th. The congress has been invited by the Society of Arts to meet in this country, and the meetings will be held in the Society's large hall. The Companies of the Drapers, Fishmongers, Goldsmiths, Merchant Taylors, and Clothworkers are associated with the Society of Arts in the invitation issued to members of the Congress, and the Right Hon. the Lord Mayor has invited members to an evening reception at the Mansion House on June 17th. The congress will be opened at 11 a.m. on Tuesday, June 15th, by an address from the President, the Duke of Devonshire, K.G., and from the President of the last congress, M. le Professeur Leo Saignat. The subjects for discussion at the congress will include—

(1.) Advanced technical education.
(2.) Secondary technical education.
(3.) Advanced commercial education.
(4.) Secondary commercial education.

The subscription to the congress will be 5*s*. Applications for membership should be accompanied by the amount of the subscription, and be addressed to the secretary, Society of Arts, John Street, Adelphi, W.C.

Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, part of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows:—

To Members (see Rules 25 and 27) and Past Members (see Rule 30); Libraries, Corporations, and Exchanges on the Society's List	Each copy	10*s*.
To Subscribers	,,	12*s*. 6*d*.
To others........................	,,	15*s*.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. Eyre and Spottiswoode, the Society's printers and publishers, to whom all communications respecting them should be addressed. The circulation of the Journal is now more than 3,000 per month.

LIST OF MEMBERS ELECTED 24th MAY 1897.

Badock, Stanley H., 9, Buckingham Vale, Clifton, Bristol, Smelter.

Bird, Fred. C. J., 52, Cambridge Gardens, Notting Hill, W., Chemist.

Burrell, Loomis, Little Falls, Herkimer Co., N.Y., U.S.A., Manufacturer.

Butler, David B., 41, Old Queen Street, Westminster, S.W., Cement Expert.

Cairns, Wm., 5, Carlton Place, Glasgow, Plumber.

Caldwell, Jas., c/o Mexican Gold and Silver Recovery Co., Hermosillo, Sonora, Mexico, Chemist and Assayer.

Crawford, Walter W., Imperial Chambers, 91, Pitt Street, Sydney, N.S.W., Mechanical Engineer.

Durkee, Frank W., Tufts College, Mass., U.S.A., Assistant Professor of Chemistry.

Fitzgerald, Francis A. J., c/o The Carborundum Co., Niagara Falls, N.Y., U.S.A., Chemical Engineer.

Goldschmiedt, Dr. Guido, Laboratory, Salmgasse 1, Prag, Austria, Professor in Imperial German University.

Hartmann, Ernest E., c/o Onomea Sugar Co., Papaikou, Hawaii, Chemist.

Harvey, Jas., Love Clough, near Rawtenstall, Lancashire, Technical Chemist.

Hunter, Prof. A. G. Kidston, (subs.) c/o John Hunter, 81, Great Clyde Street, Glasgow; and (Journals) P.O. Box 164, Dunedin, N.Z., Professor of Chemistry.

Huntington, Dr. Harwood, 159, Front Street, New York, U.S.A., Consulting Chemist.

Jones, Fred. W., Barwick, near Ware, Herts, Explosives Works Manager and Chemist.

Kerr, Wm. M, 131, South 3rd Street, Philadelphia, Pa., U.S.A., Manufacturing Chemist.

McFarland, B. W., 420, Temple Street, New Haven, Conn., U.S.A., Chemist.

Nelson, Walter, Messrs. Geo. Nelson, Dale, and Co., Ltd., Emscote Mills, Warwick, Gelatine Manufacturer.

Patchett, James, Oakworth, Hadley, Wellington, Salop, Ironmaster.

Prochazka, John, 15, East 12th Street, New York, U.S.A., Coal-Tar Colour Chemist.

Schoder, Dr. Robt., The Incandescent Gas Light Co., 14, Palmer Street, Westminster, S.W., Chemist.

Smith, Theophilus R., c/o Brotherton and Co., Haigh Park Chemical Works, Stourton, near Leeds, Chemist.

Van Gelder, Arthur P., Columbia University, New York City, U.S.A., Instructor in Chemistry.

Wallace, Robt. A., Dorset Hall, Merton, Surrey, Chemical Manufacturer.

CHANGES OF ADDRESS.

Akitt, Thos., l/o Hermosillo; Mina Cerro Colorado, Altar, Sonora, Mexico; Journals as before.

Beckett, G. H., l/o San Remo; Bofors, Vermland, Sweden.

Bradburn, J. A., l/o 1816; 1709, West Genesee Street, Syracuse, N.Y., U.S.A.

Burrough, H., jun., l/o Lafayette Avenue; c/o Burrough Bros. Manufacturing Co., Baltimore, Md., U.S.A.

De Lessing, G. C., l/o Lexington Avenue; 145, East 29th Street, New York, U.S.A.

Dickinson, F. M.; Journals to c/o Broken Hill Proprietary Co., 3, Great Winchester Street, E.C.

Fanta, F., l/o Arundel Street; 97, Queen Victoria Street, E.C.

Graham, C. C., l/o Hull; Highmoor, Benrhydding Road, Ilkley, Yorks.

Heal, Carlton, l/o Finchley; Chapel Road, Penketh, near Warrington.

Heriot, T. H. P., l/o Trinidad; Pln. Albion, Berbice, British Guiana.

Holden, G. H., l/o Victoria Park; Mendip Mount, Heaton Mersey, near Manchester.

Hopkins, Erastus, l/o Worcester: 207, State Street, Boston, Mass., U.S.A., Port Examiner.

Howard, A. G., l/o Woodford; Burnt House, Chigwell, Essex.

Kawakita, M., l/o London; Imperial Engineering College, Tokio, Japan.

Langer, Dr. C., l/o London; Hampton House, Milverton Terrace, Leamington.

Lucas, B. R., l/o Middlesbro'; Moss Villa, Elworth Road, Sandbach, Cheshire.

McLellan, J. Y., l/o Bernburg; Fabriche Unite di Biacchi e Colori, Cogoletto, near Genoa, Italy.

Matsui, G., l/o Gower Street; 31, Nicholas Street, St. Peter's Road, Mile End, E.

Meggitt, H. A., l/o Mansfield; Laboratory, Sutton-in-Ashfield, Notts.

Meggitt, Loxley, l/o Mansfield; Laboratory, Sutton-in-Ashfield, Notts.

Messel, Dr. R., l/o 53; 30, Ebury Street, S.W.

Miles, G. Wellington, jun., l/o Milk Street; 7, Exchange Place, Boston, Mass., U.S.A.

Patterson, T. L., l/o Sugar Refinery; Maybank, Finnart Street, Greenock, N.B.

Preston, Alf., l/o Market Street; Park Hill View, Bury, Lancashire.

Rademacher, H. A., l/o Lee Street; P.O. Box 243, Lawrence, Mass., U.S.A.

Redwood, I. I., l/o Bayonne; Bantry House, Picardy Hill, Belvedere, Kent.

Royal-Dawson, H., l/o Cockle Creek; Retain Journals until further notice.

Schüpphaus, Dr. R. C., l/o Brooklyn; c/o G. H. Graham, 66, Broadway, New York, U.S.A.

Scrymgeour, Wm., l/o South Africa; Australian Gold Recovery Co., Kalgoorlie, West Australia.

Segner, P., l/o Princess Street; 8, Dickinson Street, Manchester.

Smith, Allan, l/o Bury Road; Bolton Road, Edenfield, near Bury, Lancashire.

Thorp, Wm., l/o Crouch End; 22, Sinclair Gardens, West Kensington, W.

Tipler, F. C., l/o Richard Street; 48, Brooklyn Street, Crewe.

Wilson, Ellwood, l/o East Orange; 10, Belsize Avenue, Hampstead, N.W.

Wyatt, Dr. Francis, l/o Old Slip; 39, William Street, and 29, Stone Street, New York, U.S.A.

Death.

Cunliffe, Jos., Kern Mill, Whittle-le-Woods.

London Section.

THE CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE, W.

Chairman: R. Messel.

Vice-Chairman: Boverton Redwood.

Committee:

B. Blount.	H. de Mosenthal.
C. F. Cross.	B. E. R. Newlands.
W. J. Dibdin.	*S. Rideal.*
R. J. Friswell.	*F. G. Adair Roberts.*
S. Hall.	F. Napier Sutton.
D. Howard.	Wm. Thorp.
E. Grant Hooper.	T. Tyrer.

Hon. Local Secretary:

John Heron, 110, Fenchurch Street, E.C.

The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Committee:* E. J. Bevan, Otto Hehner, J. B. Knight, A. R. Ling, and Lewis T. Wright.

SESSION 1896–97.

June 14, 1897:—

Dr. A. Dupré, F.R.S. "Note on a possible Danger from Fire involved in the Transport of Barium Peroxide in Wooden Barrels."

Dr. Pauli (Farbwerke, Hoechst a Main). "On the Valuation of Commercial Nitrate of Soda."

Mr. Hudson Maxim. "Recent Improvements in Smokeless Powder Compounds and in Processes of Manufacture."

Mr. Alex. Cameron. "Comparative Experiments on the Estimation of Phosphoric Acid."

Mr. William A. Davis. "The Strength of Commercial Formaldehyde Solutions."

Meeting held on Monday, May 3rd, 1897.

DR. R. MESSEL IN THE CHAIR.

ON A SLOW COMBUSTION ARC LAMP SUITABLE FOR FACTORIES AND STREET LIGHTING.

BY W. S. SQUIRE, PH.D., F.C.S.

According to Mr. W. H. Preece, one-half of the cost of arc lighting is due to the consumption and replacement of the carbons. If in respect of this expense an economy of 50 per cent. could be realized an important step in advance would be made, still more so, if an economy of 75 per cent. could be obtained. The lamp I am about to introduce to your notice goes much further than that, as you will see presently.

In an ordinary arc lamp the consumption of carbons is usually at the rate of about $1\frac{1}{2}$ ins. per hour—1 in. for the positive or upper carbon, and $\frac{1}{2}$ in. for the negative or lower carbon, or, say, 40 mm. per hour, both carbons included. This, of course, will vary somewhat according to the amount of the current. The consumption of carbons in the new lamp (varying also with the strength of the current) and consuming the same amount of electrical energy as the ordinary arc lamp will be from 1·20 to 2 mm. That is to say, only 5 per cent. of the consumption of the ordinary lamp—an economy of 95 per cent. As this result has been attained by means—partly of a mechanical, partly of a chemical character, I thought it a fit subject to bring before a society occupied to a great extent with questions of chemical engineering.

The lamp was invented by an American named Jandus, born at Wisconsin, of Hungarian parents.

I propose in the first place to describe the mechanical construction of the lamp, giving you an opportunity of handling and examining the various parts. I will then have the parts put together so that you may see the same lamp in action. While this is being done we can consider the chemical side of the question.

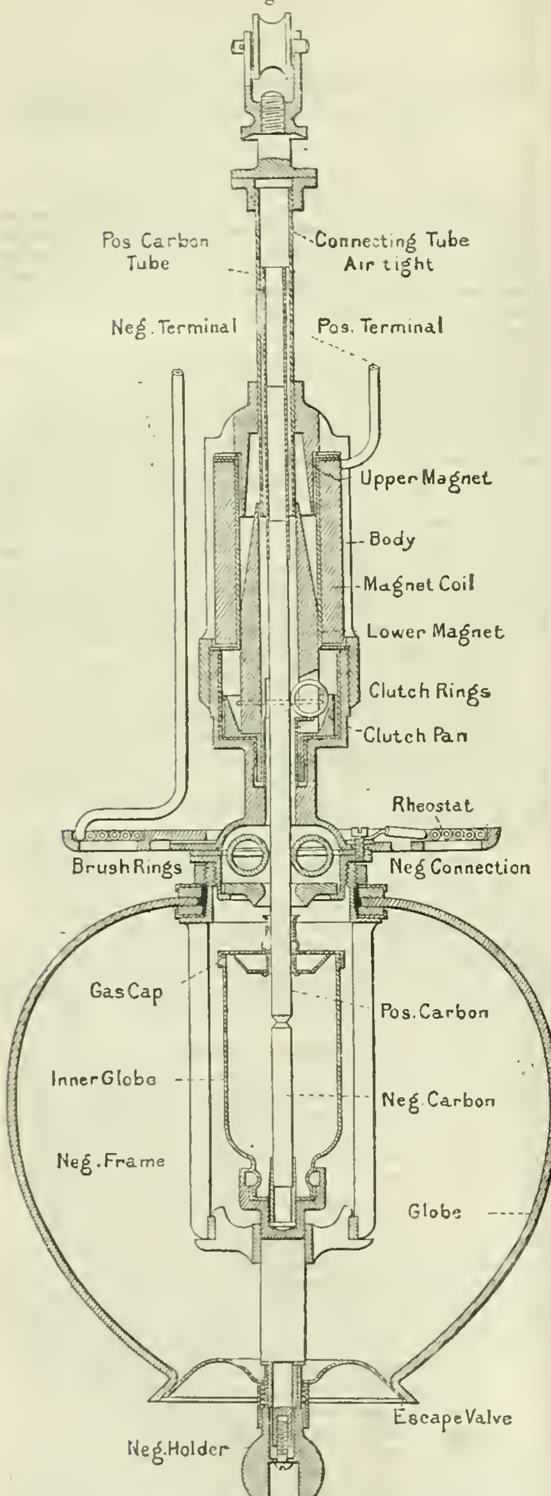
Fig. 1 represents the lamp in section. The current enters the lamp by the negative terminal, and then passes through a resistance which has the effect of reducing the voltage (which is in this room 108) down to 81. This is about double the voltage usual for arc lamps. It then passes by the insulated screw shown on the right hand to the lower carbon holder, and so to the lower carbon. It crosses the arc to the upper carbon. It leaves the carbon by the brush rings, and then goes through the magnet coil to the positive terminal.

The magnet coil encloses two cast-iron magnets, which I may call the male and female magnets. The female magnet is fixed, but the male is free to move up and down. These will have a tendency to attract one another, and when the current in the coil is strong enough to enable the induced magnetism to overcome the weight of the lower magnet this will rise carrying with it the upper carbon. The effect of this will be to lengthen the arc, and by increasing the resistance, to diminish the current through the coil, and so arrest the further rise of the magnet. Matters thus remain in equilibrium until, by consumption of the carbon, the arc becomes longer, and the current thereby diminished. The lower magnet then begins to descend. It will be seen that both the fixed and the movable magnet are hollow, and that the upper carbon is not attached to either of them. How then does the movable magnet carry the carbon with it? This is one of the most ingenious contrivances in the lamp. There are three equidistant slots in the lower magnet. In each slot a brass ring is inserted, the rings being prevented from falling out by a wire passing round the magnet. The tendency of these rings is to fall together and clip anything passing in a downward direction, but they will allow anything to pass in an upward direction. By this means the carbon is firmly attached to the lower magnet.

If, however, the magnet drops so far that the rings come in contact with a brass plate cut out so as to fit into the

slots in the magnet, they are lifted and rolled back so as to allow the carbon to slide down by its own weight between the rings; the moment the normal current is re-established

Fig. 1.



the magnet rises, the rings clutch the carbon, and its further movement is arrested. It is in this way that the upper carbon is gradually fed forward.

A somewhat similar device is employed to make an electrical contact with the upper carbon by means of the brush rings. These do not clutch the carbon at all, but rest gently against it, and being hung in such a way that they are free to turn, they make electrical contact without opposing any sensible resistance to the movements of the carbon. Moreover, they are so placed that the electrical contact is at no great distance from the arc, so that the carbon offers a minimum of resistance, and it is also an unvarying resistance—a point of some importance.

As shown in the drawing, the arc is produced inside a small glass cup, which is closed at the top by the so-called "gas cap." This cap has a double duty to perform. In the first place it prevents to a considerable extent the escape of the gases resulting from the combustion of the carbons inside the cup. In the second place it provides for automatically extinguishing the lamp when the upper carbon is nearly exhausted.

The carbon passes freely through the aperture in the centre of the cap, but the brass carbon holder cannot pass. As soon as this arrives at the cap the movement is arrested, the feed is stopped, the lamp is extinguished, and no harm can happen to the metal work.

Besides the small glass cup in which the arc is produced there is also an outer globe, which is so fastened by packing to the body of the lamp as to be quite air-tight. In this way a layer of highly heated gases is caused to float in the upper part of the outer globe upon the colder gases beneath. This tends to assist the action of the gas cap in keeping the air from the arc. Lastly, the outer globe is closed at the bottom by an escape valve, which opens to let out the gases when expanded by the heat of the arc. Of course this valve is removed when the carbons are renewed.

It is evident that the arc is produced in a small chamber, to which the possible access of air is reduced to a minimum. Consequently, very little combustion of the carbons takes place. After the lamp has been at work for a short time, almost all the oxygen present in the glass cup has been consumed, and the gases in the cup are found to have the following composition:—

Carbon dioxide	1·4
Carbon monoxide	23·6
Oxygen	2·7
Nitrogen	72·3
	100·0

The interior chamber is not, however, perfectly air-tight, and diffusion will occur between the gases in the interior chamber and those in the globe outside. Consequently, a very slow combustion of the carbons will go on, while, on the other hand, the gases produced in the interior chamber will diffuse into the outer globe. After some hours burning the gases in the outer globe are found to have the following composition:—

Carbon dioxide	0·8
Carbon monoxide	12·2
Oxygen	12·9
Nitrogen	74·1
	100·0

It is obvious that, in order to produce this effect, the tube in which the upper carbon, with its holder, slides must be perfectly air-tight, and the joint between the glass globe and the lamp must also be air-tight.

The result of this arrangement is that the carbons burn away very slowly, the consumption never exceeding 2 mm. an hour.

Using carbons of 13 mm. diameter, the upper one having a length of 270 mm., or, say, $10\frac{1}{2}$ ins., and the lower one a length of 150 mm., or, say, 6 ins., the lamp is found to burn—

	Hours.
With a current of 4 ampères	220
" " 6 "	160
" " 7 "	120

Consider what this means! When the ordinary arc lamps are used, a man must go up the post every morning

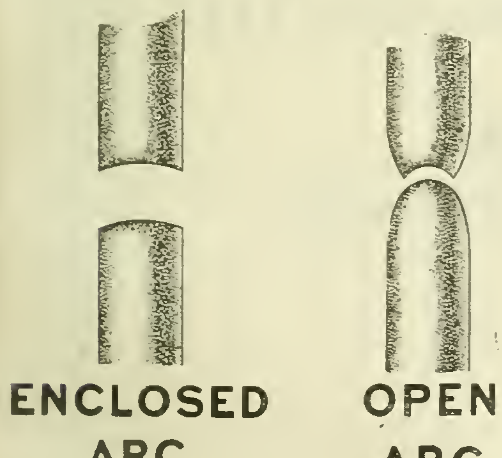
and put in fresh carbons. In the case of a lamp burning 220 hours it will be necessary to renew the carbons, on an average, only once in three weeks, say, once a fortnight in winter, and once a month in summer. This is not only a great saving in carbons but in wages as well.

The lamp which we have put together has now been burning for about 10 minutes, and the gases in the interior chamber or cup should have the composition I have indicated. I will quickly remove the lower carbon and cup. At the moment that I do so, the highly heated upper carbon as it passes out should set fire to the gases in the cup (*experiment performed*). You see the carbon monoxide burning with the characteristic blue flame.

I wish to direct your attention to the way in which these carbons burn. In the case of the ordinary arc lamp burning in air, the consumption of the positive carbon, as compared with the negative carbon, is as two to one. In the case of the Jandus lamp it is as four to one, so that the consumption of the lower carbon is practically a negligible quantity. Owing to the lower carbon, in the case of an arc produced in air, burning away at about half the rate of the upper carbon, which is in all lamps fed up to the arc, the point of light is gradually lowered in the globe, so that any devices, such as reflectors, or holophane globes, soon cease to act properly unless there is a special compensating mechanism, usually both complicated and costly. Owing to the very small consumption of the lower carbon in this lamp, the luminous point is practically fixed without any compensating mechanism whatever.

There is also a considerable difference between the shape of the ends of the carbons burning in air and those burning in the Jandus lamp (*see* Fig. 2). In the Jandus lamp they

Fig. 2.



remain very nearly flat, slightly convex on the negative, slightly concave on the positive. When the carbons burn in air the negative is very distinctly pointed, while there is a sort of crater formed on the positive. This is generally attributed to a transfer of the particles of carbon from one pole to another, but, of course, the forms produced in this way must be perpetually broken down and obliterated by the burning away of the carbons. When there is little or no burning away, as is the case with the Jandus lamp, we should expect to find this production of a point and a crater greatly accentuated, and in an exaggerated form. Nothing of the kind however happens; both the carbons remain practically flat without point or crater. Perhaps we shall have to modify our previous conceptions in regard to this matter.

We have still to consider the distribution of the light over a considerable space of ground. You will say perhaps that the light diminishes according to the square of the distance. Yes, that is quite true of a white hot ball, like the sun, but it is very far from true of the light of the electric arc. The light emitted in one direction is very much greater than that in another direction, and it depends very much what that direction is. Mr. Hesketh, of Blackpool, has been experimenting, both with the Jandus lamp and with an arc lamp burning in air. Figs. 3 and 4 give his results in a graphic form. You will notice that an arc lamp, burning in air, shoots down its rays in such a way that the maximum is attained at an angle of 45° from the perpendicular, while the Jandus lamp attains its maximum at about 75°, that is to say, only 15° from the horizontal. Consequently, while the ordinary arc lamp brilliantly illuminates objects almost immediately underneath it, and objects at a distance only slightly, the Jandus rays are better distributed over distant objects. Fig. 5 shows the amount of illumination on the ground at different distances. Assuming the lamps to be placed at a height of 20 ft., the ordinary arc lamp produces a prodigious illumination up to 20 ft. or thereabouts, but then it rapidly falls off. At 34 ft. it is less than the light of the Jandus, and then it dies away almost to nothing. The Jandus, on the contrary, starts low, owing to the partial shadow of the lower carbon. As soon as we get out of the shadow of the corner of the lower carbon the light increases, and then, owing to the nearly horizontal direction of its maximum, it diffuses its light very equally, and as you see it illuminates objects at 140 ft. quite as much as the ordinary arc does at half that distance.

The advantages presented by the Jandus lamp may be summed up as follows:—

It will burn on the public mains which have usually a voltage of about 110 without any accessories whatever.

The ordinary arc lamps must either be placed in series or must be furnished with a comparatively high resistance so as to reduce the voltage to about 40. This means conversion of a good deal of electricity into heat which does nothing but keep the wire of the resistance warm.

The lamp is very economical, the consumption of carbon being insignificant.

The distribution of light is very equable, far more so than in the case of the ordinary arc lamp.

The construction of the lamp is extremely robust. There is no clockwork or delicate mechanism. Probably it might fall from a considerable height without sustaining any other injury than the smashing of the glass.

It is eminently suited for illumination of chemical and other works. It is an essential part of the construction of the lamp that it should be practically air-tight, the working parts therefore would not be liable to be injured by gases or dust flying about in the factory.

I may mention that any further particulars about the lamp may be obtained from Messrs. Drake and Gorham, 66, Victoria Street, Westminster.

Discussion.

Mr. J. A. Hicks inquired what was the ampère consumption as compared with ordinary arc lamps.

Mr. H. Ballantyne was in a position to corroborate most of Dr. Squire's statements as to the advantages of the Jandus lamp. He would like further information, however, as to the consumption of the carbons and the gases produced in the outer envelope. Bearing in mind the materials of which the lamp was composed it was practically impossible to obtain absolutely air-tight joints, and one would expect, therefore, that when the lamp was switched off a certain amount of air would find its way in, and that after a few hours a diminution of the carbonic oxide would have taken place. He gathered that that was the case, and would like to know if the rate of consumption quoted by the author referred to a continuous run without switching off or to an intermittent run as in ordinary circumstances. He would also like to know whether the composition of the gases in the outer envelope also referred to their state after a continuous run or after intermittent switching off.

Dr. Ludwig Mond remarked that Dr. Squire had laid some stress on the remarkable saving in carbon effected by this lamp. He did not profess to be an expert on the subject, but he believed it was generally considered that in the ordinary arc lamp the rapid consumption of carbon

Fig. 3.

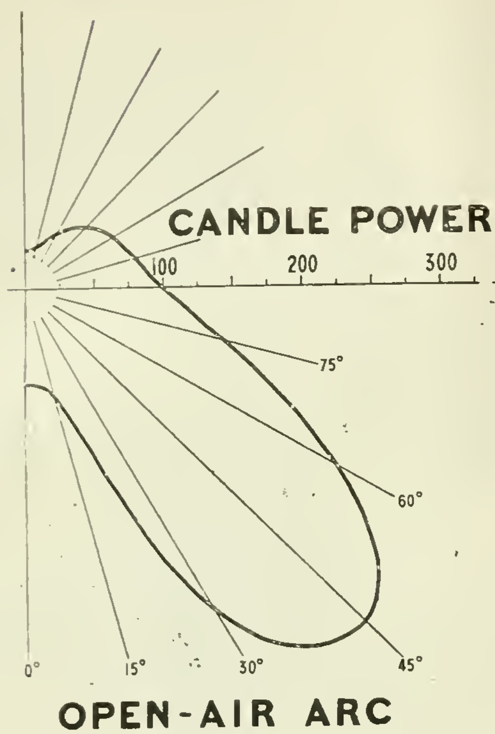


Fig. 4.

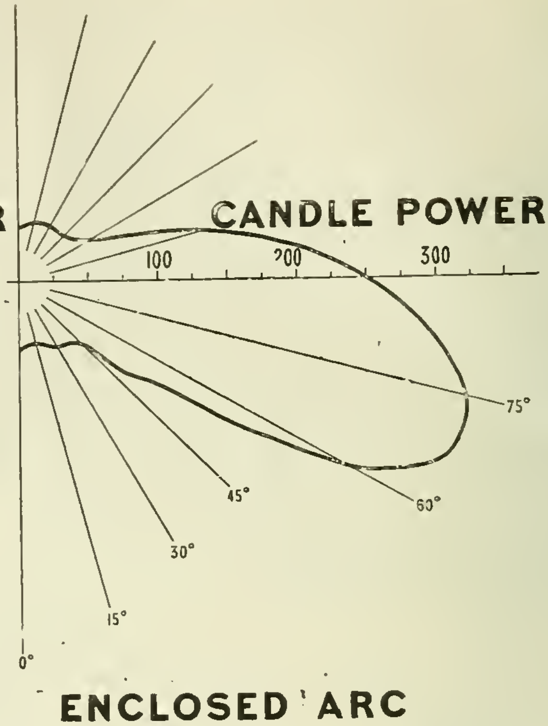
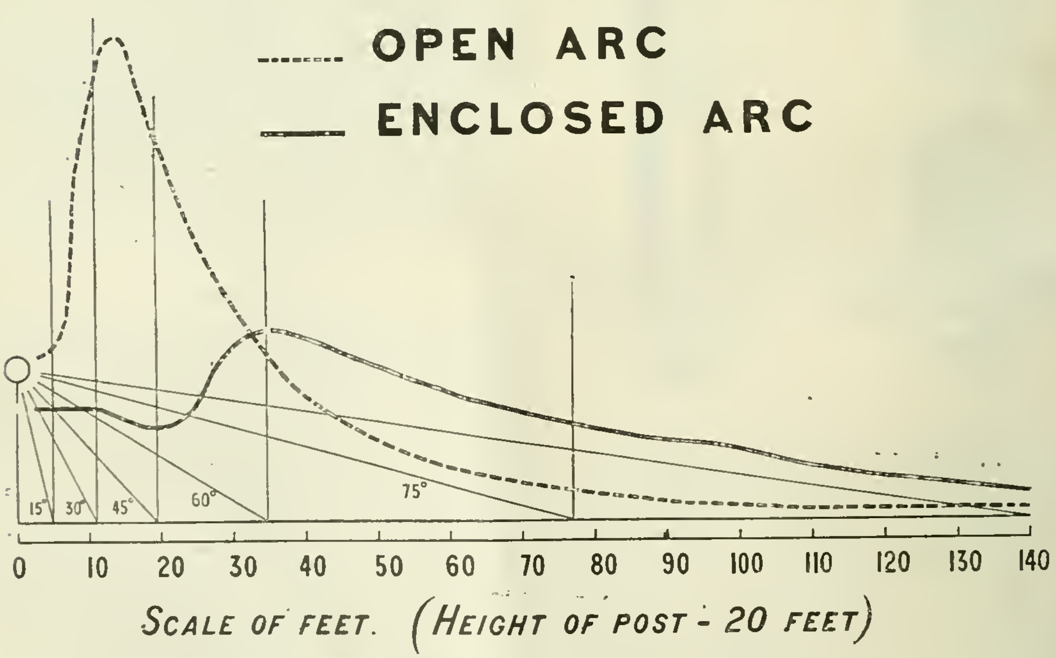


Fig. 5.



was not altogether wasteful, since such lamps gave more powerful illumination. He would therefore like to know what was the *efficiency* of the Jandus lamp as compared with the ordinary arc lamp, and to what extent the economy in carbon was counterbalanced by a loss of lighting power.

Mr. R. J. Friswell enquired whether there was anything like a plus pressure in the envelopes around the carbons, as, if so, the lamp might possibly be used in explosive atmospheres, since a continuous small current of carbonic oxide from the inner envelope would prevent the access of explosive gases to it. The lamp might thus be available for use in factories where the ordinary arc lamp was inadmissable on account of the explosive nature of the atmosphere.

Dr. S. Rideal pointed out that from the analyses given an explosive combination of carbon monoxide and oxygen

was found between the outer and inner envelopes, seeing that in the inner globe the monoxide was in excess whilst oxygen was in excess in the outer globe.

Mr. Leon Gaster asked for further information as to the light efficiency of the lamp as compared with ordinary arc lights. Prof. S. Thompson, in a recent course of lectures, had pointed out that in lamps with enclosed arc a brown deposit was found inside the globe, which obscured the green rays. That was an undesirable result, since green and yellow rays were what one wished to have as being the essential ones for light. Moreover, the accumulation of products of combustion, using impure carbons, must in time result in a loss of light. The author had stated that the lamp required 81 volts and 6·5 ampères, which meant an energy of over 500 watts to consume per hour. In a circuit of 110 volts an energy of 30 volts × 6·5 ampères, = 195 watts, would be wasted in heating the resistance put in series with the arc lamp. Would not the large amount of energy used in this lamp be in the long run dearer than the saving in the carbons? Experiments made by Prof. Ayrton showed how greatly the results of arc lamps varied according to the current employed, and that only certain current intensities and lengths of arc yielded good results. As the arc used in the Jandus lamp was a rather long one, he would like to know whether the arc could be kept steady and work satisfactorily with alternate currents, especially those of slow frequency.

Dr. W. S. Squire, in reply to Mr. Mond's remarks, said that the economy claimed for the Jandus lamp was not only in respect of the saving in the carbons, but also in the attention required to replace the carbons, which were so quickly consumed in the ordinary lamps. There was not any greater waste of current in this lamp than in the ordinary lamps, indeed not nearly so much if the ordinary lamps were used singly or in parallel with the current as usually supplied by the street mains, for in that case the ordinary lamp required a resistance involving a considerable loss of heating. The Jandus lamp being run with a high voltage and a low ampèreage, required therefore a long arc, while with the ordinary lamps the reverse was the case, and the arc was therefore a short one. In both cases the watts, *i.e.*, the amount of electric energy consumed in the lamp, would be practically the same. The proportion of gases produced would vary according to the time the lamp was burnt. The table before the meeting represented the composition of the gases in the outer envelope after the lamp had been burning for three or four hours. He did not make the analysis himself, and therefore could not vouch for its correctness, it being obvious that the exact proportion of the gases must depend to some extent on the particular part of the envelope from which they were taken. At any rate, the analysis proved that diffusion had taken place from the inner to the outer envelope. He did not claim that the escape valve was absolutely air-tight in the chemist's sense of the term. Unless it were very carefully made and tested, he would not consider it suitable for use in an explosive atmosphere, though as a general rule it might be fairly safe. Why, he asked, should one take an arc lamp into an explosive atmosphere at all, when a glow lamp answered all purposes and was infinitely more suitable.

Dr. Mond remarked that he understood the author to claim an economy in the working of the lamp, due to the dispositions of the carbons. But the question was what was the candle power produced for a given consumption of current.

Dr. Squire replied that the lamp gave an illuminating power of about 1,200 candle power per half a Board of Trade unit. But the power was not all in the same direction. Most of it was at an angle of 75, and the rest at various angles. The light produced in an arc was to a certain extent a measure of the resistance of that arc. With a greater resistance the energy was converted into heat, and that heat was realised upon the points of the carbons. Therefore if the arc were increased in length, the temperature of the carbon would be increased, because the resistance was greatest there. An ordinary arc lamp upon the usual circuit of 100 volts required a resistance interposed in order to get the arc of such a length as to bring down the voltage to about 40. This reduction involved considerable loss. It did not follow that a longer arc would produce more light, because the light came from the carbon, and only to a small extent from the arc itself. The longer arc, that is to say, the greater resistance, enabled a current of less ampèreage to heat the carbons to the required temperature.

Mr. Gaster said that his experience of such lamps was that over 85 per cent. of the current went away, and he did not see any advantage in the larger arc. With a greater surface of carbon they would have not more light but heat, and that was just what was not wanted.

Dr. Squire, interposing, said that was just what was wanted, as it was because of the high temperature of the upper carbon that the light was evolved. Electricity as electricity could not be used as a source of light. It must first be converted into heat in such a way that some substance, usually carbon, as in both the arc and glow lamps, was heated to incandescence. All bodies, without exception, offered at ordinary temperatures more or less resistance to the electric current, and were therefore more or less heated. Quite as much heat as was developed in the electric arc would be developed in a long wire offering the same amount of resistance, but as the heat was in that case spread over a long resistance, the temperature developed would be low. In the case of the arc, the resistance, and therefore the heat, being all at one point, a very high temperature was attained. The higher the temperature the greater the amount of light.

Mr. Gaster, resuming, said that what was wanted was the largest possible amount of light from the smallest possible surface. He would like to know at what intervals the results shown on the diagram before them had been taken, and if they had been checked at various times to ascertain if they agreed.

Dr. Mond said that it might interest members to know that he had accidentally learnt a fact that day which corroborated Dr. Squire's statement that the light of the arc increased with the resistance between the carbons. It had been generally supposed that the heat of an arc could not be carried beyond the volatilising point of carbon, which was estimated at 3,500° C., but M. Moissan had, in the course of his experiments on the melting of titanium, attained temperatures in the arc which he estimated at between 4,000° and 5,000° C.

A Member inquired if there was a deposit of carbon in the inner envelope sufficient to interfere with the emisson of light.

Dr. Squire replied that a slight deposit did occur upon the inner glass, but the matters forming this deposit when given off from the arc would be, and in fact were, carried by convection to the upper part of the glass and there deposited, so that the deposit was invariably above the line of light, and in no way interfered with the illuminating power. It was easily removed by the attendant when changing the carbons.

The Chairman thought that the lamp, owing to its great power of diffusion, was likely to be of considerable advantage in illuminating large spaces. The theoretical points raised during the discussion were of great interest and deserved further consideration. It was obvious that in a lamp of this nature the light produced must be at least equal to that of an ordinary arc light, but from the fact that practically no crater was formed in the carbons, the consumption of carbon must be much less.

ON ATTEMPTS TO CONVERT OLEIC ACID INTO CANDLE MATERIAL.—I.

BY DR. J. LEWKOWITSCH.

One of the most fascinating technical problems in the fat industry, is the conversion of oleic acid into solid fatty acids or, at all events, into solid substances which we comprise under the name "Candle Material."

Several years ago I was led to attack this problem from a technical point of view, and I set myself the task of examining the various processes proposed hitherto in the

hope that a thorough and methodical investigation might lead to some practically feasible method.

The great commercial importance of this subject becomes at once apparent if we consider the difference in the prices of "stearine" (commercial stearic acid) and "oleine" (commercial oleic acid)—round about 15*l.* per ton at present price—bearing in mind that the candlemaker obtains, even by the best methods known, 30 per cent. of oleic acid. This acid must rank in the candle works as a by-product, the sale of which for soapmaking becomes more and more difficult the greater the pressure of cottonseed oil and other cheap oils is on the market.

At the first glance the problem seems to resolve itself into the simple addition of two hydrogen atoms to oleic acid with formation of stearic acid, the most desirable candle material. But whereas the lower members of the acrylic or oleic series are readily converted into saturated acids, oleic acid itself resists all attempts at hydrogenisation. I have carried out a large number of experiments in this direction under most varied conditions, but hitherto all of these gave negative results. It has been tried to act with hydrogen *in statu nascendi*—evolved by electricity—on oleic acid under pressure, but the process has not proved a commercial success.

The remarkable power of resistance oleic acid offers to the assimilation of hydrogen becomes also apparent, if hydrogenisation is attempted in a roundabout fashion, *viz.*, by first converting it into the dibromo-addition product, and then reducing the latter by hydrogen. Dibromo-stearic acid is readily formed, but on attempting to reduce the latter, I always obtained the original oleic acid.

It is true that reduction can be effected by means of phosphorus and hydriodic acid, but the palpable hopelessness of such a process, from a commercial point of view, has not even elicited an attempt to try this process on a semi-large scale. By heating oleic acid with 1 per cent. of iodine to 270°—280° C. P. de Wilde and Reychler succeeded in obtaining about 70 per cent. of stearic acid. But as only one-third of the iodine could be recovered, the process had to be abandoned. Even when for part or the whole of the iodine the cheaper bromine or chlorine was substituted, commercial failure resulted. The process had a fair trial on the large scale in a Belgian candle works, but the large amount of valueless by-products, coupled with the fact that no autoclave could resist the corrosive effect of the halogens, showed once more that beautiful laboratory experiments but too often lead to disastrous results in the works.

The action of chlorine on oleic acid and the reduction of the chloro-derivatives under pressure by means of zinc, or iron powder, &c. has been made the subject of a German patent by Zürrer, who claims to have obtained solid saturated fatty acids. I have not finished yet the examination of this process, and am, therefore, not in a position to offer any remarks, but I may say that as far as I am aware, it has not been tried yet on the large scale.

The conversion of oleic acid into its solid isomeride elaidic acid has figured of long in text books, and has been propounded as a commercial process as often as it has been shown to be a valueless process to the candlemaker. To the reagents effecting this change, *viz.*, nitrous acid and sulphurous acid, there has been added lately sodium bisulphite, which Saytzeff allows to act on oleic acid under pressure at a temperature of 175°—180° C.

At this juncture I may mention that about two years ago Lidoff made the statement that by the action of nitrous oxides at 80°—85° C. on oleic acid, an increase of weight of the material takes place, and that the resulting product absorbs 9 per cent. only of iodine, whereas oleic acid assimilates 90 per cent. My own experiments, however, showed me that elaïdic acid is formed and the apparent low iodine value is due to the action on potassium iodide of nitrous acid not washed out completely from the product.

Great hopes were at one time entertained of the application of Varrentrapp's reaction, *viz.*, the conversion of oleic acid into palmitic and acetic acids by means of caustic potash. The process has been described and its commercial results stated in two papers read before this Section by the late Lant Carpenter (this Journal, 1883, 98; 1884, 200). It is true large quantities have been worked by Mr. Radisson in Fournier's works at Marseilles, and the candles made from the artificial palmitic acid created some sensation in technical circles at the Paris Exhibition of 1878. But on account of their rank odour and their greasy touch the candles found no buyers, and although further improvements were heralded, the process seems to have or has died a premature death. At the last Paris Exhibition I vainly looked (Lewkowitsch, Technical Report on the Paris Exhibition, Chemiker Zeitung, 1889, 1190) for the candles made with Radisson's artificial palmitic acid, and on enquiry I elicited the fact that the process had been abandoned, not only for the reasons stated above, but also on account of the high cost and the great danger attending its working in consequence of the concomitant evolution of hydrogen.

Another promising process, *viz.*, M. v. Schmidt's zinc chloride process has met with no better fate. The process and its products have been examined on a laboratory scale by the late R. Benedikt and his paper (*cf.* Chemical Analysis of Oils, Fats, Waxes, &c. by J. Lewkowitsch, p. 664), well repays perusal on account of the beautiful illustration it affords of the capabilities of the modern processes of fat analysis as a means of reseach. I have examined the process myself, and, as a matter of course, I could but confirm Benedikt's statement with that exception, however, that I found no solid saturated acids other than hydroxy-acids. The temperature at which the interaction of oleic acid and zinc chloride is to take place must be kept at exactly 185° C. as the inventor directs. Deviation from this point leads to an increase of liquid substances. Unfortunately the solid candle material must be distilled and the considerable proportion of β-hydroxystearic acid (melting point 82° C.) in the crude product is seriously diminished by the partial conversion of this acid into oleic and iso-oleic acids. Thus, from a candlemaker's point of view, a substance of high melting point is rendered practically valueless. Schmidt's process was tried on the large scale in an Austrian candle works. The quantity of liquid unsaponifiable substances obtained was, however, so large that commercial success was out of the question. It only remains to be added that the inventor has allowed his patent to lapse.

The action of concentrated sulphuric acid on oleic acid is well known in candle works, and practical application of it is made in the partial conversion of oleic acid into solid substances. I say partial, as out of the 47 per cent. of oleic acid with, *e.g.*, tallow, would yield only 17 per cent. are converted into candle material. As stated already, 30 per cent. of oleic acid still result as a by-product.

The sulphuric acid process seemed to offer the greatest hope of success, and I have devoted considerable attention to it.

I have also examined and have still under examination several other reactions not mentioned above, and I hope to lay the results of this research at some future time before this Society.

My reason for bringing some of my results regarding the action of sulphuric acid on oleic acid before the Society, incomplete though they are, is that David has recently taken out a patent for a process which to some extent covers results I have obtained some time ago. Whilst preparing these notes I find that there has also appeared a paper by the same chemist in the Comptes Rendus, an abstract of which is published in last month's issue of our Journal.

It is not my intention to criticise David's paper, but I must remark that his "stearolactone," the proportion of which is stated by him to be 18—20 per cent. of the crude product, is practically nothing but β-hydroxystearic acid. At any rate, in my own experiments I obtained chiefly β-hydroxystearic acid, even when closely following his direction. The impure substance had an acid value of 189·12 and an ether value of 14·29 only, so that the amount of lactone was insignificant. Besides, the melting point of the crystals obtained by the first crystallisation from alcohol, and therefore still very impure, was 60°—60·5° C., whereas stearolactone melts at 47°—48° C. The melting point of β-hydroxystearic acid is 81° C. Experiments made with

mixtures of concentrated and fuming acids (see below) gave β-hydroxystearic acid and not the lactone.

I. Action of Sulphuric Acid on Oleic Acid.

The action of sulphuric acid on oleic acid is a very complicated one, and although a number of papers have been published by different authors a satisfactory explanation of the reactions taking place is still a desideratum.

No doubt this is chiefly due to a formula satisfactorily expressing the chemical constitution of oleic acid not having been established yet. We shall perhaps best get an insight into the difficulties besetting this subject by briefly examining its theoretical aspect.

At present we know four fatty acids of the empirical formula $C_{18}H_{34}O_2$, *viz.*, oleic acid, elaïdic acid, iso-oleic or solid oleic acid, and rapic acid.

The last-mentioned acid, rapic acid, is certainly not identical with any of the other three acids, for it does not solidify on cooling; nor does it give the elaidin reaction. Being but little studied the acid need not engage our further attention.

Elaïdic acid is undoubtedly the stereometrical isomeride of oleic acid, and its formula will be readily derived from that of oleic acid.

On attempting to arrive at a rational formula of oleic and iso-oleic acids, the safest plan will be to try the inductive way by building upon well ascertained facts. Oleic acid, on being heated with melted caustic potash, yields palmitic and acetic acids. This reaction would appear to be in favour of the simplest expression, *viz.*, $C_{15}H_{31}CH=CH-COOH$. But as iso-oleic gives the same products, it tells us no more than that under the influence of caustic potash an intramolecular change takes place, one acid being converted into its isomeride before being finally broken up. We might, therefore, assign to oleic acid and iso-oleic acid the formulæ $C_{13}H_{27}-CH_2-CH_2-CH=CH.COOH$ and $C_{13}H_{27}-CH_2-CH=CH-CH_2COOH$ respectively, or *vice versâ.*

A more definite answer is supplied by the sulphuric acid reaction. On treating *oleic acid* with this acid at a low temperature and boiling the product with water, one obtains, amongst other substances, (1) a solid anhydride, the corresponding acid of which does not exist in its free acid, and (2) a hydroxystearic acid.

The solid anhydride $C_{18}H_{34}O_2$ (having the same empirical formula as oleic acid, but differing from it by its physical state and by not absorbing halogens) must be considered as the inner lactone of a hydroxy acid, $C_{18}H_{36}O_3$, and reasoning from analogy, its stability would lead us to look upon it as a γ-lactone, so that its formula would be expressed by—

$$\begin{array}{l} \qquad\qquad\quad\ \gamma \\ C_{13}H_{27}-CH_2-CH-CH-CH-CO \\ \qquad\qquad\quad\ \ |\qquad\qquad\qquad\ | \\ \qquad\qquad\quad\ O\!-\!-\!-\!-\!-\!-\!-\!-\!-\!-\!-\!- \end{array}$$

and its corresponding acid thus—

$$\begin{array}{l} C_{14}H_{29}-CH-CH_2-CH_2-COOH \\ \qquad\qquad\ \ | \\ \qquad\qquad OH \end{array}$$

This would fix the γ-carbon atom as one of the doubly-linked carbon atoms in oleic acid, and it would only remain to choose between the formulæ of a γ-δ or a β-γ unsaturated acid, thus—

$$C_{13}H_{27}-CH=\overset{\gamma}{C}H-CH_2-CH_2-COOH$$

or—

$$C_{13}H_{27}-CH_2-\overset{\gamma}{C}H=CH-CH_2-COOH.$$

The hydroxystearic formed besides the lactone just described yields, on being subjected to distillation *in vacuo*, two unsaturated acids, *viz.*, oleic and iso-oleic acids. a portion of the original acid passing over unchanged at the same time.

This behaviour of the hydroxystearic acid is completely analogous to that of all known β-hydroxy acids derived from lower fatty acids of the acrylic series, and analogy thus speaks for the formula—

$$\begin{array}{l} \qquad\qquad\qquad\quad\ \beta \\ C_{14}H_{29}-CH-CH-CH-COOH \\ \qquad\qquad\ \ |\qquad\ | \\ \qquad\qquad\ H-OH \end{array}$$

On treating *iso-oleic acid* with concentrated sulphuric acid and boiling the product with water, two hydroxystearic acids are formed, one of which is identical with the hydroxy acid yielded by oleic acid. The proportions of the two hydroxy acids vary according to the temperature employed during the interaction, the yield of the second hydroxy acid decreasing as the temperature rises. This second hydroxystearic acid distils without decomposition *in vacuo*, it yields no lactone and cannot therefore be a γ-hydroxystearic acid. Reasoning again from analogy this behaviour of the acid would correspond to that of an α-hydroxy acid, thus—

$$\begin{array}{l} \qquad\qquad\qquad\qquad\quad\ \alpha \\ C_{14}H_{29}-CH-CH-CH-COOH \\ \qquad\qquad\qquad\ |\qquad\ | \\ \qquad\qquad\quad\ \ H\quad\ OH \end{array}$$

Since both oleic and iso-oleic acids lead to the same hydroxystearic acid it is evident that both acids must have, in common, one carbon atom which is linked by a double bond to its neighbour; and as we fixed the γ-carbon atom as one of these for oleic acid, the carbon atom common to both acids must be either the β or the δ carbon atom, so that we would have to adopt for iso-oleic as formula, either a δ-ϵ or an α-β unsaturated acid.

The above-mentioned analogies render it more likely that oleic and iso-oleic acid have the β-carbon in common as one of the doubly linked carbon atoms, so that we should have as the likeliest formulæ for oleic and iso-oleic acids the following:—

$$C_{13}H_{25}-CH_2-\overset{\gamma}{C}H=\overset{\beta}{C}H-CH_2-COOH \text{ and—}$$

Oleic acid.

$$C_{13}H_{25}-CH_2-CH_2-\overset{\beta}{C}H\quad \overset{\alpha}{C}H-COOH-$$

Iso-oleic acid.

I have avoided adducing the formation of palmitic acid as an additional proof, for the conclusion derived from the behaviour of an unsaturated acid with melting caustic potash—formerly held as conclusive—has lost much of its force, and one might, therefore, rule out of court whatever evidence is afforded by the formation of palmitic and acetic acids. But I would think the reasoning given above speaks in favour of the formulæ as stated, and I may therefore omit discussing the formula—

$$C_8H_{17}-CH=CH-(CH_2)_7-COOH$$

and proposed by Baruch (Berichte, 1894, 173).

I have dwelt at some length at these theoretical speculations as they are of the utmost importance for the solution of our problem. I merely will remark by the way that the same reactions, *mutatis mutandis*, occur in the case of erucic, brassidic, and iso-erucic acids.

From the candlemaker's point of view the conversion of oleic acid into iso-oleic would be an important step, for the latter acid melts at 47° C., and does not share the fate of elaïdic acid when worked into candles, for commercial distilled stearine does contain considerable quantities of iso-oleic acid.

I have made some experiments with oleic acid and sulphuric acid diluted with an equal volume of water (*cf.* Fittig, Liebig's Annalen, **283**, 51), but no practical result was obtained. As the conversion into iso-oleic acid by means of iodostearic acid and alcoholic potash had to be left out of the question for obvious reasons, I was forced to fall back upon the known interaction of concentrated sulphuric acid on oleic acid.

The products of this reaction at a low temperature are chiefly β-hydroxystearic acid, sulphostearic acid, and stearolactone.

Adopting the formula $C_{13}H_{27}-CH=CH-CH-COOH$ for oleic acid, we may explain the interaction by the simple addition of $SO_3H.OH$ to oleic acid, much as hydrobromic acid is assimilated.

According as the SO_3H group is assimilated by the γ or the β carbon atom, two different sulphostearic acids will be formed, and, as there is no reason why one acid should be formed in preference to the other, it is reasonable to assume that both acids are formed, say, in equal proportions, and that we thus obtain the two sulphostearic acids—

$$(1.)\ C_{13}H_{27}—\underset{SO_3H}{\underset{|}{CH}}—\underset{OH}{\underset{|}{CH}}—CH_2—COOH \quad \text{and}$$

$$(2.)\ C_{13}H_{27}—\underset{OH}{\underset{|}{CH}}—\underset{SO_3H}{\underset{|}{CH}}—CH_2—COOH$$

(One might as well write the formulæ of these acids—

$$C_{13}H_{27}—CH(SO_4H)—CH_2—CH_2—COOH \text{ and}$$
$$C_{13}H_{27}—CH_2—CH(SO_4H_2)—CH_2—COOH$$

but the mode of expression adopted renders the chemical changes more conspicuous to the eye.)

These acids are not very stable, and, by merely allowing the crude product to stand, they absorb moisture enough to be partially split up into SO_4H_2 and hydroxy acids, *viz.*, β and γ hydroxy acids—

$$C_{13}H_{27}—CH_2—\underset{OH}{\underset{|}{CH}}—CH_2—COOH \quad \text{and}$$

$$C_{13}H_{27}—\underset{OH}{\underset{|}{CH}}—CH_2—CH_2—COOH$$

the latter of which immediately undergoes dehydration, forming γ-stearolactone. Another portion of the sulphostearic acids will remain unchanged.

On boiling the resulting mass with water, the sulphostearic acid is further converted into hydroxystearic acids, Thus a suitable candle material would be obtained, *if subsequent distillation could be avoided*, whereby, as explained already, β-hydroxystearic acid is changed into oleic and iso-oleic acids. (I purposely omit here following up the possibility of β-hydroxystearic acid forming products of condensation, *viz.*, hydroxystearohydroxystearic acid $OH.C_{17}H_{34}COO.C_{17}.H_{34}COOH$ and the dibasic anhydride—

$$O\left\langle\begin{array}{l}C_{17}H_{34}COOH\\C_{17}H_{34}COOH\end{array}\right.$$

as suggested by Juillard.)

In the first instance it seemed necessary to direct efforts to the production of a crude material containing the smallest amount of unsaturated products. It is known that the best conditions to effect this are found in working at a low temperature, and by employing concentrated acid and dry oleic acid.

I describe my *modus operandi* briefly :—Oleic acid was cooled to 0° C. by ice ; the congealing of the acid does not interfere with the subsequent operation, as the temperature rises soon. Sulphuric acid, also cooled down, was then allowed to fall drop by drop into the oleic acid, whilst the mass was agitated vigorously. The temperature was not allowed to rise beyond 5° C., although no detrimental effect was observed when the temperature rose to 25° C. towards the end of the operation, as was the case in experiments on the large scale. No evolution of sulphurous acid was noticeable, and the material was not blackened. This is in contradistinction to the action of vitriol at higher temperatures which was carried out for the sake of comparison (see below). The product became, on standing, a buttery mass. It was boiled with water repeatedly until the wash-waters were free from acid.

The decomposition with water just described seemed in some experiments to yield a better material when the mixture of oleic acid and sulphuric acid was allowed to stand 24 hours; other experiments, however, did not bear this out, and seemed to prove that time was not an important factor. This is a point to be ascertained yet on the large scale.

In the following table (Table No. 1) a number of experiments is given, the quantities of sulphuric acid, calculated to one molecule of oleic acid. being varied. The amount of unsaturated substances was measured by the iodine number of the product. The original oleic acid was prepared from good tallow ; it absorbed 80 per cent. of iodine.

Table No. 1.

Action of Sulphuric Acid of Varying Strength on Oleic Acid.

Oleic Acid.	Sulphuric Acid.		Iodine Number of the Product.
Molecules.	Containing SO_4H_2	Molecules.	
	Per Cent.		
1	95	1	39·83
1	95	1	33·73
1	95	1	47·23
1	103 (fuming acid.)	1	26·26
1	103 (fuming acid.)	1	20·43
1	95	2	10·96
1	95	2	10·86
1	95	2	10·28
1	95	2	11·15
1	95	2	14·99
1	92	2	23·02
1	92	2	24·06
1	103 (fuming acid.)	2	10·28
1	100·5 (mixture of fuming and concentrated acid.)	2	14·40
1	100·5 (mixture of fuming and concentrated acid.)	2	14·41
1	95	2·5	16·73
1	92·5	2·5	23·60
1	93	2·5	19·61
1	95	3	6·74
1	95	3	8·46
1	103 (fuming acid.)	8	16·04

This table is full of suggestions for a number of experiments that are being carried out on the large scale. Contrary to expectation, the greatest amount of saturated products is not obtained when acid was used of 100 and more per cent. of SO_4H_2. There appeared this further drawback in the employment of acids of such high strength, that the product was liquid at the ordinary temperature, and gave emulsions with water, so that the sulphuric acid had to be washed out by means of a solution of Glauber salts, whereas in all other cases washing with water gave two well-defined layers. The same drawback was experienced in the case of treating oleic acid with vitriol at 132° C. Also the quantity of water mixed with the crude product has apparently some influence on the chemical changes.

It should be noted here that practically no evolution of sulphurous acid was observed, whilst working at low temperatures; therefore no loss of substance occurs, and the yield of the raw product must be more than 100 per cent. of the oleic acid worked upon. If we assume for a moment that addition of sulphuric acid to oleic acid takes place exclusively, according to the equation—

$$C_{18}H_{34}O_2 + SO_4H_2 = \underset{\text{Hydroxystearo-sulphuric acid.}}{C_{18}H_{34}O_2(OH)SO_3H.}$$

and that on boiling with water the decomposition takes place, according to the following equation :—

$$C_{18}H_{24}O_2(OH)(SO_3H) + H_2O = C_{18}H_{35}O_2(OH) + SO_4H_2$$

141 grms. of oleic acid should yield 150 grms. In an actual experiment 145 grms. of the crude product were obtained, but, of course, this is not meant as a confirmation of the assumption made. For not only are anhydride (stearolactone) and other products formed, but the action of sulphuric acid on oleic acid reaches, from a practical point

of view, a limit, as a glance at Table No. 1 will show (see above).

The crude products obtained by the experiments described in Table No. 1 were examined as set out in Table No. 2. Hitherto, it has only been possible to carry out, on a semi-large scale, using over 1 ton of material, the interaction of 1 mol. of oleic acid with 1 mol. of sulphuric acid of about 95 per cent. of sulphuric acid.

TABLE No. 2.

Crude Product obtained by the Interaction of Sulphuric Acid and Oleic Acid at a Low Temperature.

No.	Oleic Acid.	Sulphuric Acid.		Laboratory Experiments.			Works Experiment.		
	Molecule.	Containing Per Cent. SO_4H_2	Molecules.	Acid Value.	Saponification Value.	Iodine Value.	Acid Value.	Saponification Value.	Iodine Value.
1	1	95	1	157·8	179·2	31·8	98·4	198·3	24·8
2	1	100·5 (mixture of fuming and concentrated acid).	1	136·1	192·6	23·1	..	..	..
3	1	103 (fuming acid).	1	120·8	169·5	24·3	..	..	..

The fact that β-hydroxystearic acid on being distilled undergoes considerable deterioration from a candlemaker's point of view—for hydroxystearic acid has a melting-point of 82° C. in its pure state—suggests the idea of removing it from the crude product, before subjecting it to distillation. The use of solvents is, of course, out of question, and the only practically feasible process is hydraulic expression. This is being tried on the large scale, and it is hoped that, by suitable processes, the necessity of distilling may be obviated.

In the experiments described below, with the exception of that given in Table No. 6, the hydroxystearic acid was not eliminated before distilling.

The crude products characterised in Table No. 2 were subjected to distillation, which, in the case of the laboratory experiments, was carried out in a flat bottomed cylindrical copper still (a by no means suitable apparatus), and in the case of the works experiments an ordinary still was used. Superheated steam was employed, and the distillation was effected, for reasons of convenience, under atmospheric pressure instead of *in vacuo*. It will, of course, be understood that these experiments are comparable in a limited sense only; the distillates were broken up into fractions when they appeared to change in consistency, and the residues varied considerably in colour, thus proving that distillation might have been carried further in some cases. In no case, however, was distillation continued until pitch only remained in the still.

The results thus obtained are given in the following Table, No. 3, the numbers 1—3 given in the first column correspond to those in Table No. 2.

TABLE No. 3.

Distillation of Crude Products with Superheated Steam.

No.	Crude Product obtained from Oleic Acid and Sulphuric Acid containing—per Cent. SO_4H_2.	Laboratory Experiment.						Works Experiment.					
		First Fraction.	Second Fraction.	Third Fraction.	Fourth Fraction.	Residue.	Loss.	First Fraction.	Second Fraction.	Third Fraction.	Fourth Fraction.	Residue.	Loss.
	1 mol. of oleic acid and 1 mol. of SO_4H_2:—	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
1	95	27·4	42·7	20·4	1·9	4·0	3·6	30·0	30·0	26·5	..	6·2	7·3†
2	100·5 (mixture of fuming and concentrated acid).	26·6	46·5	17·6	..	6·2	3·1	..	..	..	..	..	..
3	103 (fuming)	19·5	16·9	33·7	14·9	11·7	3·3	..	..	..	..	..	..
	1 mol. of oleic acid and 2 mols. of SO_4H_2:—												
4	93	11·0	13·8	12·2	52·7	6·5	3·8	..	..	..	..	..	..
5	96·5	9·5	11·9	13·6	51·5	6·6	6·9*	..	..	..	..	..	..

* Still leaked a little. † Of course, this high loss is due to the exceptional circumstances.

The several fractions thus obtained are further characterised in the following Table No. 4.

TABLE No. 4.

Constants of the Several Fractions obtained from the Crude Products No. 1—3, Table No. 3.

———	Crude Product, No. 1. Laboratory Experiment.			
	First Fraction.	Second Fraction.	Third Fraction.	Fourth Fraction.
Iodine number	58	84·3	83·8	82·4
Acid "	192	196·5	196·9	188·1
Saponification number	201	201·2	199·9	194·9
Melting point, 0° C.	Liquid.	27·2—28·3	27·2—29	25·5—29

———	Crude Product, No. 1. Works Experiment.			
	First Fraction.	Second Fraction.	Third Fraction.	Bulk.
Iodine number	82·8	85·2	65·8	77·1
Acid "	197·5	197·3	149·1	197·5
Saponification number	200·7	200·0	188·9	201·3
Melting point, 0° C.	20·2	25·3	24·9	23·45

TABLE No. 4—*continued.*

—	Crude Product, No. 2. Laboratory Experiment. First Fraction.	Second Fraction.	Third Fraction.
Iodine number..........	56·6	78·5	82·2
Acid „	197·6	198·2	194·6
Saponification number ..	201·7	199·9	198·4
Melting point, 0° C.	Liquid.	26·6—28·3	26·6—29

—	Crude Product. No. 3. Laboratory Experiment. First Fraction.	Second Fraction.	Third Fraction.	Fourth Fraction.
Iodine number..........	61·9	73·4	81·4	84·1
Acid „	204·5	200·5	198·9	192·8
Saponification number..	209·1	203·3	199·7	194·5
Melting point, 0° C.	Liquid.	Liquid.	26·1—27·2	29—29·4

It should be borne in mind, that the iodine numbers do not correspond to the equivalent amounts of oleic acid, the solid iso-oleic acid absorbing the same amount of iodine as the ordinary oleic acid. A glance at the melting points shows that iso-oleic acid has been formed in considerable quantities, whereas stearolactone can be present but in small quantities judging from the small differences between saponification and acid values. This is remarkable as stearolactone is supposed to distil without decomposition.

For the sake of comparison I add the results obtained on treating oleic acid with 1 mol. of concentrated SO_4H_2 at 132° C. See Table No. 5.

TABLE No. 5A (LABORATORY EXPERIMENT).

Product obtained by the Interaction of Sulphuric Acid and Oleic Acid at 132° C. and then distilled.

—	Yield.	Acid Value.	Saponification Value.	Melting Point.
	Per Cent.			° C.
First Fraction ..	10·8	181·16	201·3	Liquid
Second Fraction	39·0	167·38	193·9	25·5—30
Third Fraction..	16·6	152·68	191·9	25·5—32
Residue.........	29·4 (corrected, 26·8)	..	..	..
Loss	4·2	..	..	..

The residue is very high indeed. In mitigation of this high number it must be stated that it contained 9 per cent. of ash (Glauber salts from the washing, see above), but this still leaves for the corrected number 29·4 − 2·6 = 26·8 per cent. The proportion of stearolactone is, however, higher than in the case of crude products No. 1—3, Table No. 4.

The same experiment was carried out on the large scale. The results thus obtained are given in Table 5B.

TABLE No. 5B (WORKS EXPERIMENT).

Product obtained by the Interaction of Sulphuric Acid and Oleic Acid at 132° C. and then distilled.

—	Iodine Number.	Acid Value.	Saponific Value.	Melting Point.
				° C.
Crude product	53·90	169·8	..	..
Bulk distillate from crude product.	71·60	180·2	202·0	20·70
Bulk distillate :—				
Cold-pressed cake ..	69·45	195·5	207·8	30·80
„ oil	69·45	184·8	202·2	Liquid
Hot-pressed cake...	72·10	202·8	206·7	43·05 (titer test)

It is evident that acidification at the high temperature can never be a paying process, on account of the great loss, and it can only be a question of examining more closely acidification at a low temperature.

From the practical point of view, the most important factor, next to the yield, of the pressed cake is its melting point, as some minor technical difficulties will, no doubt, be overcome. A report on the experiments on the large scale must be deferred to a future occasion.

In conclusion, I submit the results of an experimental distillation of a crude product obtained by the interaction of 1 mol. of oleic acid and sulphuric acid of 95 per cent. SO_4H_2, freed from hydroxystearic acid.

TABLE No. 6.

Distillation of Crude Product freed from Hydroxystearic Acid.

—	Yield. I.	Yield. II.	Melting Points. I.	Melting Points. II.
	Per Cent.	Per Cent.		
First fraction ...	24·0	23·4	Liquid	Liquid
Second „	24·0	29·2	24—29	Oily
Third „	(32·9)	(13·9)	24·4—29·4	24·4—27·7
Fourth „	(7·8)	(22·9)	20·0—25·5	26·6—30·5
Residue.........	5·1	5·7	..	..
Loss	6·2	4·9	..	..

The considerable amount of liquid products obtained must not be looked upon as consisting entirely of unchanged oleic acid. These liquid substances consist to some extent of anhydrides, and further experiments must decide whether they contain ordinary oleic anhydride—which I prepared some years ago by heating oleic acid with acetic anhydride (this Journal, 1890, 666)—or products of polymerisation.

DISCUSSION.

Dr. RIDEAL, while admitting the great scientific value of the paper thought that it would have been appropriate if it had been read before the Chemical Society, as at present the results of the author's experiments did not seem to warrant any commercial application on a large scale.

Mr. W. MANSBRIDGE considered the statement put forward by the author to be extremely interesting from the theoretical point of view, but that, from the standpoint of the candle-maker, they offered very little encouragement. Even the best fractions of the material treated had very low melting points, and further distillation appeared to be necessary, the result of which would be partially to return them to a liquid condition, unfitting them entirely for the manufacture of candles. They had already many cheap sources of stearic acid of low quality, what was wanted was something of a high quality. Some years ago he had met with a sample of stearic acid made by Radison's process, which gave very fair candles, but its use was abandoned because the process of re-distillation was found to be too expensive. It appeared to him that in the present case they would be met with a similar difficulty. The margin between the ordinary brown oleic acid and the hard stearine of cotton oils was less than 3*l.*, and the cost of re-distillation would soon do away with that margin. He therefore did not see much prospect of oleic acid coming into use as a source of candle-making material.

Dr. J. LEWKOWITSCH, in reply, while admitting the general correctness of Mr. Mansbridge's views, explained that in the hurry of summarising his paper he had omitted a point which would seem to promise better results. The crude product referred to undoubtedly gave candle-making material, but it required pressing, and the question resolved itself purely into one of yield. The raw product contained the two substances, stearolactone and hydroxystearic acid, and it was now for them to endeavour so to treat it as to avoid the production of the liquid acid. From the candle-maker's point of view distillation was undoubtedly a drawback. That point was fully dealt with in the paper, and his view was that they should try to arrive at some means short of distillation which would yield a white material suitable

for the purpose. Some of the experiments were made at a very low temperature—near freezing point—and some at a high temperature, 130—and the latter yielded a considerable loss. He did not attach any importance to the apparently considerable loss in the works experiment because those experiments were made very roughly in unsatisfactory apparatus, and the results were only given as an auxiliary to the laboratory experiments.

The CHAIRMAN regretted that from want of time the discussion had been so restricted. He did not agree with Dr. Rideal's view, for unless chemists saw the theoretical side of the problems they had to deal with, such problems would have a poor chance of solution.

NOTE ON OZONE GENERATORS.

BY EDWARD BEANES.

HAVING read, with a considerable degree of interest, the paper of Mr. E. Andreoli on "Ozone: Its Commercial Production," &c. (this Journal, 1897, 89—93), and having also noted the number of patents recently taken out on this subject, I thought that probably a brief recital of a few points of hard-earned experience from an old worker in this difficult field would be acceptable.

For the commercial application of ozone, the generator with glass plates, which was introduced years ago by me, would appear to be the only way, so far as can be at present seen, to obtain a large area. Point-bearing electrodes have been advocated, I know, but experience has shown me that points should be avoided. For the successful production of ozone, the electrodes should be as highly charged with electricity as possible, but such charging cannot be adopted with point-bearing electrodes because sparks would be produced, and ozone, already formed, would be destroyed. With very highly charged electrodes (most conducive to ozone formation) even specks of dust or dirt might give rise to sparks through the formation of points. The only way to avoid sparks with point-bearing electrodes is to avoid charging these electrodes so highly as to reach the sparking point, but this would be at the sacrifice of capacity for the production of ozone; yield would be greatly diminished.

The air to be ozonised should be dried, and filtered through cotton-wool; otherwise small solid particles may become deposited on the electrodes, and sparking may ensue, as already explained.

There is no doubt that nitrous vapours would be formed as the result of sparking, and with these a peculiar odour, but ozone would be destroyed. Looking at the character of a large number of so-called "chemical patents" annually issued, it is not impossible that the conditions laid down in sundry of those patents, ostensibly for the production of ozone, will rather favour the formation of oxides of nitrogen —in other words, of nitrous fumes.

I may, in conclusion, perhaps, just call attention to a slip in a statement in Mr. Andreoli's paper, *loc. cit.*, page 89, col. 2, and 30 lines from the bottom, where it is stated that "Ladd constructed an ozone generator, which consisted of parallel sheets of glass," &c. It is true that Ladd constructed such a generator, but he did so as ordered and employed by me: it was my generator, and the first in which sheets of glass were used.

Liverpool Section.

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The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Committee:* A. Smetham and Frank Tate.

SESSION 1896–97.

Meeting held on Wednesday, April 7th, 1897.

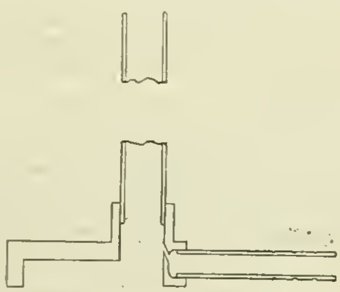
DR. C. A. KOHN IN THE CHAIR.

AN IMPROVED FORM OF BUNSEN BURNER.

BY HUGH MARSHALL, D.SC., F.R.S.E.

A GREAT drawback with the ordinary form of Bunsen burner is the liability of the central jet to become choked up by anything falling down the tube. A single drop of water is often sufficient to extinguish the flame, and a fused borax bead is still more objectionable, owing to the difficulty of properly clearing the jet. This is troublesome enough in ordinary laboratory work, but is much worse with a large practical class. Various methods of getting over the difficulty have been tried more or less successfully. I think, however, I have now succeeded in evolving a form of burner which is a considerable improvement on preceding ones.

The improvement consists in replacing the central jet by a suitably inclined lateral opening in the wall of the burner itself. The burner is left open right through, and the side air holes are done away with. An air regulator can be



fitted on the base in the form of a pivoted diaphragm. A simple form of the burner is illustrated in the diagram. This represents a section through the gas-supply tube and inlet (to the right of drawing) and one limb of the tripod base (to the left). The upper tube screws into the lower portion. This consists of a very shallow tripod with circular opening for the tube. At one side of the opening is a block into which the gas-supply tube is fixed and through which the gas inlet is drilled. The inclination of the inlet and its diameter depend somewhat on the richness of the gas used.

The advantages of the burner are, that the gas inlet does not become choked; anything dropping down the tube passes right through to the bench. Further, if the gas inlet is properly arranged, the flame can be turned down very low without it striking back or the air supply requiring regulation. Again, the air regulator fitted on the bottom cannot jam in the way the usual circular regulator does; in fact, it can be completely removed by the aid of a screw-driver in a few seconds; it is unnecessary except when a luminous flame is desired.

The burner is now in use in Edinburgh and Aberdeen Universities, and works well. It is manufactured only by Messrs. Baird and Tatlock, being provisionally patented.

THE MANUFACTURE AND INDUSTRIAL USES OF PERSULPHATES.

BY HUGH MARSHALL, D.SC., F.R.S.E.

When requested by the Chairman of the Section to give a paper on "The Manufacture and Industrial Uses of Persulphates," I explained to him that whilst I had prepared considerable quantities for my own use and studied many of their reactions, I had no practical experience of these salts from a manufacturer's point of view. It followed, therefore, that any paper would consist largely in a compilation of material already published, though not in a connected form readily accessible to members of the Society. Your Chairman thought, however, that such a paper, giving an account of my own work, and a *résumé* of the most important work done by others, might still be of use, if only to draw attention to the possibilities of these new oxidising agents. On the Continent the persulphates have received, and are receiving, a considerable amount of attention, but in this country they seem to have been totally neglected, so far as I am aware.

The anhydride of persulphuric acid was first prepared by Berthelot in 1878. He obtained it by submitting a mixture of dry oxygen and sulphurous anhydride to the silent electric discharge in an ozonising tube. It is an oily liquid, freezing to white crystalline solid at lower temperatures. It can be sublimed at a moderate temperature. It decomposes spontaneously in the course of a few days, forming sulphuric anhydride and oxygen. It dissolves in water, giving an acid solution, which soon decomposes, forming sulphuric acid and oxygen; it dissolves in sulphuric acid, and this solution is more stable. The composition of the anhydride is represented by the formula S_2O_7.

Berthelot also showed that the oxidising liquid formed round the anode during the electrolysis of fairly concentrated sulphuric acid contains a substance other than hydrogen peroxide, which had up till then been supposed to be the sole cause of the oxidising action. The properties of the electrolysed sulphuric acid were exactly similar to those of a solution of persulphuric anhydride in sulphuric acid, and Berthelot assumed that during the electrolysis of the acid a hydrate of persulphuric anhydride—persulphuric acid—had been formed.

Berthelot studied the conditions affecting the formation of persulphuric acid by electrolysis, more especially as regards the concentration of the sulphuric acid. He did not prepare the salts; in fact, it was for some time supposed that these could not exist, persulphuric acid being classed along with hydrogen peroxide as an "indifferent" substance. This view had immediately been put forward by Mendeleef, whose ideas of the "periodic law" did not admit of a salt-forming oxide of sulphur higher than SO_3. Subsequently, however, Berthelot showed that persulphates certainly could exist in solution.

In 1891, while engaged in an investigation on the electrolytic oxidation of cobalt salts, I incidentally obtained a small quantity of a potassium salt, which analysis proved to have the composition represented by the empirical formula KSO_4, and which therefore was a salt of Berthelot's persulphuric acid. The general properties of the salt also agreed with those of persulphuric acid. Only a small quantity of the substance was obtained, and attempts were made to prepare further quantities by simply electrolysing potassium hydrogen sulphate solution in the apparatus employed for the cobalt electrolyses (Chem. Soc. J. 1891, 771). This consisted of a platinum basin of 200 c.c. capacity, cooled externally by a stream of water. In the centre of this was placed a porous jar containing dilute sulphuric acid, into which dipped a thick coil of platinum wire to act as cathode. The basin contained a saturated solution of potassium sulphate in dilute sulphuric acid, and itself constituted the anode. The current was supplied by a secondary battery, and averaged in the experiments about 3 ampères. Solid persulphate began to separate after a day or two, and thereafter deposited fairly rapidly. When a considerable amount had collected, it was drained off and dried on porous plate. The mother-liquor was diluted somewhat and resaturated with potassium sulphate, after which the electrolysis was continued. It was found that the crude persulphate thus obtained could be purified by dissolving it in boiling water, filtering quickly, and cooling the liquid. The loss was small, although slight decomposition did take place.

Similarly, it was found that the corresponding ammonium salt could easily be prepared by employing ammonium sulphate in place of potassium sulphate. It separated solid in the anode cell in the same way, and was similarly treated. The loss on purification in this case was much greater owing to the ammonium salt being much more soluble in water. (Ice-cold water dissolves only 1·8 per cent. of the potassium salt, but 58 per cent. of the ammonium salt; so that a strong solution of ammonium persulphate produces an abundant precipitate of potassium persulphate when added to a strong solution of, say, potassium acetate.)

Other sulphate solutions gave no deposit of persulphate on electrolysis, the normal or acid salts being apparently less soluble in the mother-liquor, and separating out first. Certain other persulphates were, however, prepared indirectly; the ammonium salt formed the starting point, so it was prepared in much greater quantity than the potassium salt.

In order to prepare ammonium persulphate in quantity more conveniently than was possible in the small apparatus first employed, a new apparatus with much larger basin was tried. Berthelot in his early papers had stated that he preferred to use a small anode (of platinum wire), to prevent as much as possible decomposition of persulphuric acid in contact with platinum. As, however, I had not observed in my former experiments any special tendency to such decomposition, although the surface of platinum was large, and as the apparatus had given good results in preparing large quantities of cobaltic solutions, I expected to get equally good results with it in the present case. In this I was disappointed, no persulphate separating even after some days with a current rising to 5 ampères. The apparatus was therefore completely changed so as to use a smaller anode, still in a large vessel. The latter consisted of a beaker standing in an outer vessel containing cooling water. In the beaker, to one side, was suspended a porous jar containing dilute sulphuric acid in which dipped the cathode, formed of a sheet of thin platinum foil. The anode was composed of a long, thin platinum tube in which cold water could be circulated. The beaker was filled with a saturated solution of ammonium sulphate in dilute sulphuric acid. With this apparatus, using a current of 3—5 ampères, good yields were obtained, especially when a very small anode, got by dipping the tube only a short distance into the liquid, was employed. It was found advisable to keep two solutions going, one being saturated with ammonium salt whilst the other was being electrolysed. If the solutions were not changed frequently, ammonium persulphate crystallised out when the sulphate was added, showing that there was deficiency of ammonium salt in the liquid. Sometimes a perforated vessel containing crystals of ammonium sulphate was suspended in the upper layer of solution during the electrolysis. When solid persulphate had collected in the beaker to a considerable depth, the mother-liquor was transferred to a similar beaker, in which the operation was continued. The proper draining of the persulphate mud was at first somewhat difficult. The plan which succeeded best was to sink in it a porous jar closed with an indiarubber stopper, from which a glass tube led to a large bottle, from which the air was pumped. The liquid was gradually sucked out, and a dry, hard mass of crude ammonium persulphate resulted. If so desired, this could then be further purified by recrystallisation from water.

Several kilos. of ammonium persulphate were prepared in this way without difficulty.

The impossibility of preparing persulphates when a very large anode was employed, led me to change my views as to the way in which they were formed. The formula which had at first been adopted for the persulphates was $M'SO_4$. This was based on a determination of the electrical conductivities of solutions of the potassium salt, the values found agreeing with those given by Ostwald for potassium perchlorate. If that formula were correct, we might assume that persulphuric acid was formed simply by the

discharge of the HSO_4 ions conveying the current, these, and not SO_4, forming the majority of the anions in solutions of sulphuric acid or acid sulphate of the strength employed. In that case it was difficult to see why the amount formed should be so diminished by increasing the anode surface. If, however, the true molecular formula for persulphate should be $M'_2S_2O_8$, then the case is entirely different. Persulphuric acid would then be formed by two discharged HSO_4 ions uniting together, and there would be greater opportunity for such union the closer the ions were packed together at the moment of their discharge, that is to say, the higher the current density. To obtain a high current density with a large anode means a very strong current; but with moderate current the same effect is procured by correspondingly diminishing the anode area. To decide the matter definitely, estimations of the molecular weights of the various persulphates were made by freezing-point determination. These all pointed to the doubled formula. Similar results were obtained by other observers. An experiment of a different kind was also tried. A solution of barium persulphate was electrolysed in a U-tube, with platinum electrodes. Barium sulphate rapidly formed round the cathode, but not at all round the anode. If the persulphate ion were SO_4 we should expect it when discharged to give at least some sulphuric acid by acting upon the water. Instead of that, however, we get only persulphuric acid and oxygen; so that this experiment also, if anything, favoured the double formula. All doubt was finally removed by a redetermination of Ostwald's conductivity values for potassium perchlorate, which were shown to have been formerly inaccurate.

We may therefore consider the formation of persulphate from sulphate by electrolysis as very similar to that of tetrathionate from thiosulphate by the action of iodine. Persulphate is in fact analogous to tetrathionate, as exhibited by the corresponding formulæ $(MSO_3)_2O_2$ and $(MSO_3)_2S_2$; just as anhydrosulphate, $(MSO_3)_2O$, corresponds to trithionate, $(MSO_3)_2S$. The names persulphuric acid and persulphate are therefore now somewhat unfortunate, as the persulphates have no close relation to the perchlorates and permanganates.

To get good yields of persulphate it is evident, then, that we should have: first, an anode solution containing the largest possible proportion of MSO_4 (practically HSO_4) ions; second, high current density; third, low temperature. The first ensures that the largest possible portion of the current goes to provide the HSO_4 groups required for the formation of persulphate; it implies the removal of already formed persulphate as rapidly as possible from the anode. The second secures that a large proportion of the discharged HSO_4 groups unite together to form persulphate, and do not cause other, unproductive, reactions. The third is necessary because the acid liquid containing persulphate easily decomposes when warmed; possibly also there would be less persulphate actually formed, the other reactions predominating as the temperature rose. It is probable that persulphuric acid is the substance formed in the electrolysis of the various salts, the potassium or ammonium persulphate resulting by double decomposition with the potassium or ammonium sulphate present.

As a matter of fact, these conditions, so far as they relate simply to the electrolysis of sulphuric acid, had been carefully studied by Richarz as early as 1885, but I was unacquainted with his original paper (Ann. d. Phys. u. Chem. xxiv, 1885) at the time of my work. He gives the results of whole series of experiments on the effects produced by varying the above conditions. Another point discovered by Richarz is that, after an anode has been in use for a short period, the yield of persulphuric acid unexpectedly fails, but that the yield resumes its former amount if the anode is withdrawn and made red hot. Apparently the surface of the anode is acted upon in some way, a slight roughening being produced.

More recently the conditions affecting the formation of persulphuric acid have been very fully studied by Elbs and Schönherr (Zeitschrift für Electrochemie, 1895—1896), who were at first also unaware of the full extent of Richarz's work. Their results bear out those of the latter, and as they cover more ground, I shall refer to them later.

I need only say a few words about the other persulphates which I prepared. None could be obtained by direct electrolysis of the sulphates in dilute sulphuric acid. Those tried in this way were sodium, lithium, magnesium, zinc. Attempts to get pure crystallised specimens of them by various double decompositions—for example, through the barium salt—were also unsuccessful; they are apparently very soluble in water. Small quantities of approximately pure sodium salt were prepared by the action of pure sodium hydroxide on strong solutions of the purified ammonium salt, and evaporation *in vacuo*. They could not be got to crystallise, however, and gave only crystalline crusts contaminated with sulphate. The barium, strontium, and calcium salts are easily obtained pure by the action of the corresponding hydroxides on a strong solution of ammonium persulphate, and evaporating the filtered solutions *in vacuo*. They all crystallise with 4 mols. of water, and are very soluble, especially the calcium salt. They dissolve in absolute alcohol, the solution in the case of the barium and strontium salts soon depositing a monohydrated salt, practically insoluble in the liquid. The calcium solution does not do so, and the addition of ether to it throws down the tetrahydrated salt unchanged. A small quantity of lead salt was prepared by treating the barium salt with dilute sulphuric acid in slight excess and dissolving lead carbonate in the persulphuric acid solution thus obtained. It is very soluble and difficult to crystallise. It contains 2 or 3 mols. of water of crystallisation. Lead persulphate was of a certain interest, as it had been suggested that its behaviour might throw light on the chemistry of secondary batteries. It decomposed very quickly, forming lead sulphate and free acid, but no peroxide, so that it is very unlikely that it plays any part in the charging of a secondary cell. The contrary view is nevertheless held by some chemists, though it is strongly combated by Elbs.

I shall now give a few of the principal results obtained by Elbs and Schönherr in their investigation already mentioned. Their experiments are of importance, supplying data for obtaining the best yields by electrolysis; but it is to be remembered that the yields stated refer to the amount formed in solution, and not to the quantity of solid persulphate obtainable.

The apparatus employed consisted of a divided cell formed by a porous jar of 100 c.c. capacity, standing in a beaker. A cylinder of lead or platinum of 150 sq. cm. surface, surrounding the jar, formed the cathode. The anode was a platinum wire or foil dipping into the jar. The cell was surrounded with ice. Sometimes the arrangement was reversed, a ring of platinum wire in the outer cell forming the anode, and a coil of lead pipe in the jar acting both as cathode and as a cooling worm. The liquids to be investigated were of course placed in the anode division, sulphuric acid always in the cathode division.

Influence of Acid Concentration.

Current density = 100 amp. per sq. decim. Temp. 5°—6°.

The maximum effect was obtained with acid of 1·45 sp. gr.

Yield for 1st hour, 69·5 per cent. of possible.
" 2nd " 40·5 " "

The fall during the second hour is probably largely due to the accumulated persulphuric acid itself undergoing electrolysis.

Influence of Current Density at Anode.

Acid of 1·38 sp. gr. Duration of exp. = 50 min.
Current density = 4 amp. per sq. decim.
Yield = 0·7 per cent. of possible.
Current density = 28 amp. per sq. decim.
Yield = 53·3 per cent. of possible.
Current density = 500 amp. per sq. decim.
Yield = 67·5 per cent. of possible.

Influence of Temperature.

Acid of 1·38 sp. gr. Duration of exp. = 60 min.
Temperature − 2° C. Yield 64 per cent. of possible.
" 27° C. " 33 " "
" 60° C. " 0·9 "

Influence of other Substances dissolved.

A great many experiments were carried out in which other sulphates besides sulphuric acid were present. In many cases. as, for example, with zinc and magnesium, these had no noteworthy effect. In others there was a large increase in the persulphate yield. The sulphates which produced the best results were those of nickel, potassium, ammonium, and aluminium, the increase being most marked in the latter case; sodium sulphate, strange to say, had practically no action.

It was further found that a very small quantity of hydrochloric acid added to the sulphuric acid (0·03 grm. in 50 c.c.) caused a most remarkable increase in the yield; for example, in one case the yield rose from 43·9 per cent. to 69 per cent., and in another, from 10 per cent. to 57·3 per cent. Possibly the high result in the case of aluminium sulphate was partly due to the fact that the sample used contained a trace of chloride.

Generally speaking, the results obtained in the experiments under this head are quite inexplicable.

Influence of the Condition of the Anode Surface.

An ordinary platinum anode must be frequently removed and ignited, the surface being kept as bright as possible. A platinised electrode gives very poor results, but also becomes efficient when made red hot, the surface then assuming a metallic lustre. An anode placed horizontally is better than a vertical one, as in the former case the convection currents caused by escaping gas remove the newly-formed persulphuric acid more quickly from the neighbourhood of the anode, which is of importance.

Finally, the authors conclude from their various results that ammonium persulphate is the cheapest and most convenient to prepare, using a moderately high current density, a strong solution of the sulphate, with or without sulphuric acid, and adding ammonium carbonate from time to time to keep up the supply of ammonium salt. If potassium persulphate is wanted, it is better to prepare it from the ammonium compound by double decomposition.

In such experiments the percentage yield can be got by determining the oxidising power of a known fraction of the solution and comparing it with the theoretically possible for the current during the time of the experiment (1 ampère-hour could produce, at most, 3·63 grms. persulphuric acid); or by comparing the quantity of oxygen evolved with that evolved in a voltameter placed in the circuit, the yield of persulphuric acid being proportional to the difference between them relatively to that evolved in the voltameter. Instead of the volume of oxygen given by the voltameter, half the volume of the hydrogen obtained at the cathode in the apparatus itself might be taken for an approximate result.

A fresh development in connection with the preparation of persulphates has taken place comparatively recently, and may be of considerable importance. Some time ago, Löwenherz applied for, and was granted, a German patent for the preparation of sodium persulphate by the action of caustic soda on the ammonium salt. Later, he took out a new patent for the preparation of it directly in the electrolytic cell. This he effects by thorough cooling of the liquid, high current density, and the occasional addition of soda to the solution. The apparatus is the usual divided cell, a porous jar standing in a vessel of similar height and of about three times the capacity. The vessel is of metal (presumably lead), contains sulphuric acid and water mixed in about equal volumes, and itself constitutes the cathode. The inner cell contains a saturated solution of sodium hydrogen sulphate, and the anode is a piece of platinum wire or foil. The liquid is cooled by cold water, circulating in a coil in the inner cell, and surrounding the outer vessel. The current employed is about 3 ampères per sq. cm. of anode, at 15 volts, but may rise considerably above that. From time to time during the process, sodium carbonate, either in solution or fused in its water of crystallisation, is added to the inner cell. In course of time solid sodium persulphate separates at the bottom of the porous jar. When sufficient has collected the jar is removed, and the solution poured off into another jar, in which the process is continued. The process is continuous but for this changing of the cells, necessary once in about 12 hours.

If this process is economical as regards current, the preparation of sodium persulphate should rank before that of the ammonium salt, as the original material is cheaper. In use, the sodium salt would also be much preferable to the potassium or ammonium salts. It is far more soluble than the former, and more generally applicable than the latter—if alkaline solution was desirable, for example.

It seems to be somewhat generally supposed that the persulphates are very unstable salts, and cannot be kept for long. This is certainly true of solutions, and of those salts which have water of crystallisation, but does not apply to those salts which can be got pure and dry. Potassium salt is the best example of such, its sparing solubility rendering it capable of easy purification. It undergoes practically no change at the ordinary temperature. Samples of it prepared five or six years ago, recrystallised and carefully dried, still have bright shining crystal faces and are almost quite free from sulphate. The ammonium salt cannot be easily prepared so pure as the potassium salt to start with, and does not keep quite so well. Still, a sample prepared five and a half years ago, which had been once recrystallised from water, powdered, treated with ammonia gas, and dried, still contains 97 per cent. of persulphate. Other samples not so carefully treated, now contain much more sulphate than the above. In presence of water the decomposition of persulphates is much more rapid.

The most striking chemical properties of the persulphates is their powerful oxidising action, whether used in neutral, acid, or alkaline solution. In many cases, however, the action is probably not a direct oxidation, a molecule of persulphate $(MSO_4)_2$ taking up two atoms of a second metal to form 2 mols. of sulphate. Such action would correspond more to the direct action of bromine or iodine. Many reactions of the persulphates are most simply explained in this way. Among these are the following:—

Action on chlorides, bromides, iodides; sulphate and free halogen are produced (most of these persulphate reactions proceed slowly at ordinary temperatures, more rapidly on warming): $K_2S_2O_8 + 2KI = 2K_2SO_4 + I_2$.

The conversion of ferrocyanide into ferricyanide is similar: $K_2S_2O_8 + 2K_4FeCy_6 = 2K_2SO_4 + 2K_3FeCy_6$.

Also that of manganate (in alkaline solution) into permanganate: $K_2S_2O_8 + 2K_2MnO_4 = 2K_2SO_4 + 2KMnO_4$.

An interesting example is the action on solution of thiosulphate, resembling that of iodine, tetrathionate and sulphate being produced—

$$K_2S_2O_8 + 2K_2S_2O_3 = 2K_2SO_4 + K_2S_4O_6$$

This affords an easy method of preparing tetrathionates from the corresponding persulphate and barium thiosulphate. In this case insoluble barium sulphate and soluble tetrathionate are directly produced—

$$(NH_4)_2S_2O_8 + 2BaS_2O_3 = 2BaSO_4 + (NH_4)_2S_4O_6.$$

There is a considerable evolution of heat in this reaction, as may be observed by mixing concentrated solutions of sodium thiosulphate and ammonium persulphate. A gradual but very marked rise of temperature takes place.

Somewhat similar to these actions is that on ferrous salts, converting them into ferric. Thus ferrous sulphate with ammonium persulphate and water gives a brown solution from which iron ammonium alum crystallises out—

$$2FeSO_4 + (NH_4)_2S_2O_8 = Fe_2(SO_4)_3 + (NH_4)_2SO_4.$$

This reaction is employed for the quantitative determination of persulphate, with the aid of a standard solution of permanganate.

The action of persulphate solutions on the metals themselves is also interesting, many of the latter dissolving more or less rapidly; so that we have here a case of a metal dissolving in a solution of a normal salt and producing a solution containing normal salts only.

Zinc, aluminium, cadmium, and magnesium simply dissolve up without any specially noticeable phenomenon—

$$K_2S_2O_8 + Zn = K_2SO_4 + ZnSO_4$$

Cobalt also dissolves, giving a pink solution, and forming a slight dark deposit. Nickel sheet or wire is hardly attacked at all, but an electrolytic deposit of the metal dissolves more rapidly.

Mercury is rapidly attacked. If pure solution of potassium persulphate is poured on metallic mercury, a slight black deposit quickly forms, but soon gives place to a considerable yellowish-white precipitate. In presence of free sulphuric acid, a white precipitate only is produced. This is also the case with ordinary solutions of ammonium persulphate.

Copper is dissolved, giving a solution of cupric sulphate, while a slight reddish deposit is also noticeable on the metal.

Lead is attacked only superficially, the bright surface tarnishing from the formation of a thin film of sulphate, which apparently prevents further action.

Silver is dissolved by persulphate solution. If a piece of wire or foil is suspended in the solution it becomes gradually corroded, while a black deposit forms on the metal and at the bottom of the vessel. This afterwards dissolves with evolution of gas. Probably silver sulphate first forms: $K_2S_2O_8 + 2Ag = K_2SO_4 + Ag_2SO_4$. From this silver peroxide is formed by a second reaction—

$$Ag_2SO_4 + K_2S_2O_8 + 2H_2O = K_2SO_4 + 2H_2SO_4 + 2AgO.$$

Metallic iron dissolves very easily in solution of persulphate; with concentrated and slightly acid solution of ammonium persulphate the action is violent. If ordinary iron is used, a deposit of carbon remains. Iron is quickly attacked by the solution, even if it contains free ammonia, ferric hydroxide being formed; and, as it is probable that no carbon compound is produced under such circumstances, this might form a convenient method of obtaining the total carbon in iron analysis. The reaction is being investigated for this purpose.

Gold and platinum are not attacked by persulphate solutions.

Persulphates cause more or less complete precipitation of peroxides when added to solutions of certain metallic salts; for example, those of manganese, silver, and cobalt. In other cases, as those of lead and nickel, precipitates of peroxides are obtained only after the addition of alkali. In the case of manganese the oxidation can be carried still further by digesting the mixture on the water-bath for some time, when a pink solution of permanganic acid or of alkaline permanganate is slowly formed. Similarly, in presence of alkali, chromic salt can be converted into chromate by means of potassium persulphate.

Many organic substances are oxidised by acid or alkaline persulphate solution. Thus, many colouring matters are easily bleached by it; for example, indigo, litmus, turmeric.

The above are some of the most interesting reactions of persulphates which I have investigated. Recently I have been studying some of them, along with others, somewhat more fully, and hope to be able to make a communication on the subject later.

As regards the technical uses of persulphates, I can give little information of a definite character. Numerous suggestions for their employment have been made, but whether or not these have really found practical application it is difficult to discover. Thus, they have been proposed as bleaching agents, and for use in medicine as disinfectants. For several years potassium and ammonium persulphates have been manufactured in quantity by Dr. A. Curchod, Nyon, Switzerland, and he has supplied them to dealers and various manufacturers. In many cases they were wanted for experimental investigations in dyeing, bleaching, the manufacture of dyestuffs, &c., and apparently have found a certain applicability in some of these departments.

Some time ago potassium persulphate was put upon the market, under the name of "anthion," as a hypo-eliminator for use in photography. The special advantages claimed for it were that it completely destroyed the thiosulphate without attacking the image. It undoubtedly destroys the thiosulphate, but the resulting tetrathionate is probably as difficult to remove by washing, and, if allowed to remain at all in the negative or print, would probably be quite as objectionable as thiosulphate: further, as persulphate solution does attack metallic silver, there is almost certain to be some effect on the image.

Although I have been so unsuccessful in obtaining definite particulars of the industrial employment of persulphates, perhaps some members of the Section who employ oxidising agents on the large scale may obtain hints as to possible uses of them from the diverse reactions which have been already noted. Of course, a good deal depends upon the price at which persulphates can be produced on a manufacturing scale. On this point I have no data. The present retail price in small quantity is about 3s. to 4s. per lb. A year or two ago it was double that. The commercial potassium salt contains as much as 99 or more per cent. of persulphate; the ammonium salt very much less.

Even though persulphates should not become of much use on a large scale, they are convenient substances to have in a laboratory. In many cases they can be used in place of hydrogen peroxide, bromine, iodine, &c.; and, as we have seen, can be preserved for a long time without decomposition.

Meeting held on Wednesday, May 5th, 1897.

DR. C. A. KOHN IN THE CHAIR.

THE VOLUMETRIC ESTIMATION OF ZINC.

BY EDWARD GEO. BALLARD, A.R.S.M., F.I.C.

In the volumetric determination of zinc by a standard solution of sodium sulphide, more or less difficulty is often experienced in ascertaining the exact point at which all the zinc is precipitated and the sodium sulphide becomes present in excess. This is usually accomplished by adding to the ammoniacal solution of zinc under examination some flocculent ferric hydrate, and watching for the moment that the darkening of the flakes takes place, which happens as soon as an excess of sulphide has been added. This needs much care and practice in order to obtain uniform results, owing to the fact that unless the solution of zinc be kept in constant agitation the iron is very liable to blacken before all the zinc is precipitated. This, of course, may be overcome by filtering off small portions of the solution from the suspended zinc sulphide and testing a drop of the filtrate with ferric hydrate on a slab, or by using a solution of lead in the same way, or by employing lead paper. I have found the following method very delicate and rapid in determining the end of the titration, and it is based upon the fact that the suspended sulphide of zinc has no action on metallic silver, whereas the smallest excess of sulphide of sodium in the solution will produce a stain upon a bright silver surface. A small bright plate of silver is procured, and at intervals during the titration a drop of the zinc solution containing suspended ZnS is taken out on a glass rod and placed upon the silver plate, and allowed to remain there for 10 to 20 seconds. No blackening of the silver surface occurs until there is an excess of sodium sulphide present, when the stain upon the silver plate is evident at once, and the titration may be considered finished.

The number of drops of the standard solution of sodium sulphide required to produce the stain in the time mentioned, may be ascertained in a quantity of water equal in bulk to that of the zinc solution operated upon, and afterwards deducted from the total used in calculating the result. One part of Na_2S in 20,000 of water will produce a stain.

Another way to use the silver-plate indicator is to run in the sulphide to small excess, and then titrate back with a solution of zinc of known strength, watching the disappearance of the stain on the silver plate in this instance. Time is thus gained when testing a substance containing an unknown quantity of zinc for the first time, but in cases where the amount is approximately known, the former method suffices, the greater part of the sulphide being run in at once, and the silver-plate indicator applied during the addition of the last portions only.

Precaution.—Although it has been stated that the precipitated sulphide of zinc has no action upon the silver plate, yet in the presence of a large excess of ammonia in the cold there is a slight action; therefore it is desirable to

observe the precaution to avoid, as far as possible, adding more ammonia than is required to redissolve the precipitate first formed in rendering the solution of zinc under examination alkaline. However, if the temperature of the solution of zinc be raised to about 180° F., a much larger excess of ammonia may be added without interfering with the accuracy of the test or the final estimation of the zinc. Under any circumstances I prefer titrating the solution of zinc hot.

The first appearance of the stain on the silver plate can be more easily distinguished in a diffused light, such as that reflected from a sheet of white paper, or a white card, and more especially if the drop be removed at the end of 10 or 20 seconds by means of a small blotting pad or piece of folded filter paper.

A porcelain dish is the most convenient vessel in which to perform the titration. Another point to ensure accuracy is to be careful that the silver plate is clean and free from grease. A little chalk and ammonia is useful for this purpose.

APPARATUS FOR GAS ANALYSIS.

BY J. KENT SMITH AND J. W. TOWERS.

READ BY J. KENT SMITH.

Some two years ago one of us had occasion to perform a large number of gas analyses in connection with the changes taking place in producer-gases of different compositions after passing through the regenerators of a Siemens steel furnace, and a short time previous to this a large number of analyses of coal-gas obtained in the Semet-Solvay process of coking. The necessities of these cases, together with some experimental work entailed, were the origin of the present apparatus. The results obtained, and the very favourable comments of several chemists who have seen the apparatus working, prompted us to put it before you this evening.

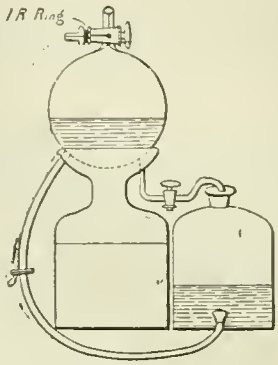
We first submit to you a modified gas sampler, more especially adapted for taking continuous or average samples of gas, and working with mercury. That mercury is the only liquid which may be employed for *accurate* work is, we think, universally established. Many operators recommend the use of water, previously saturated with the gas to be examined. This I, personally, have found unsatisfactory, especially in regard to the analyses of illuminating coal-gas. Some time ago I had occasion to draw some average samples of coal-gas, representing a 24 hours' make, both raw and purified gas being sampled. The collecting bottles were filled with water taken direct from the large station meter in which the *raw* gas was measured. Samples were also collected "dry" at short intervals over the 24 hours and analysed for H_2S and CO_2. While in the dry samples the percentages of CO_2 and H_2S gave as an average 2·6 per cent. in the crude gas and 0·7 per cent. in the purified gas, in the samples collected over gas-water the results were respectively 2·0 per cent. and 1·3 per cent., thus showing that CO_2 had actually been dissolved in the one case and taken up from the water in the other.

This is certainly an extreme case. The sampling bottles were subjected to a considerable range of temperature, but this is a point very generally to be encountered when drawing average samples. Again, the saturated water was saturated in both cases with "raw" gas. In spite of this, we are of the opinion that the above figures are a strong proof that "saturated water" is anything but an advisable containing liquid for an average gas sampler.

Our sampler (Fig. 1) consists of a hollow sphere of stout glass mounted on a solid stand, having a capillary blown on to the upper extremity carrying a three-way glass tap of 1½ mm. bore. At its lower extremity on the one side is a tubulure of 2 mm. bore, and on the other side a capillary carrying a single-way glass tap of 1 mm. bore, the exit tube of which is bent to a seal, thus preventing over-running and consequent loss of sample. The upper tap has a ridge blown on it just beyond the glass casing, and a strong rubber band fits into the groove, forming a washer, which prevents the tap from springing out of place. The tubulure is attached by thick rubber pressure tubing to an ordinary Orsat bottle, which acts as a mercury reservoir. About midway up the rubber tubing a screw slip is placed.

The sampler is filled completely with mercury by raising the Orsat bottle, opening the sealed tap till the mercury flows out, closing it, and keeping the mercury rising until

Fig. 1.



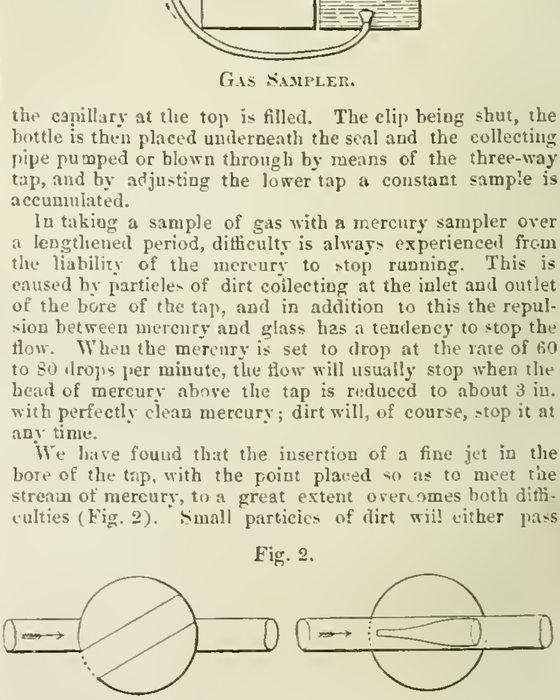
Gas Sampler.

the capillary at the top is filled. The clip being shut, the bottle is then placed underneath the seal and the collecting pipe pumped or blown through by means of the three-way tap, and by adjusting the lower tap a constant sample is accumulated.

In taking a sample of gas with a mercury sampler over a lengthened period, difficulty is always experienced from the liability of the mercury to stop running. This is caused by particles of dirt collecting at the inlet and outlet of the bore of the tap, and in addition to this the repulsion between mercury and glass has a tendency to stop the flow. When the mercury is set to drop at the rate of 60 to 80 drops per minute, the flow will usually stop when the head of mercury above the tap is reduced to about 3 in. with perfectly clean mercury; dirt will, of course, stop it at any time.

We have found that the insertion of a fine jet in the bore of the tap, with the point placed so as to meet the stream of mercury, to a great extent overcomes both difficulties (Fig. 2). Small particles of dirt will either pass

Fig. 2.



through the jet or fall on one side and collect in the space round the jet, and the mercury will continue to run until the head is reduced to about 1 in.

There is this objection to the use of the jet: the tap can be used at one speed only, because the jet must always be at full bore. It is very easy, however, to make the jets; a minute or two suffices to make one, if the one in use is not of right size. They are made by pushing a tapering capillary tube through the plug of the tap until the point is reached at which it fills the bore; this point is marked, the tube withdrawn and heated for a second in a small flame at about an eighth of an inch from the mark, then drawn out quickly to the size required, and the tapering piece, about a quarter of an inch long, altogether cut off.

In order to make the speed of aspiration more uniform, a tube of 1 to 2 ft. may be attached, if desired, to the sampler, to give a greater fall; this must, of course, be bent at the lower end to form a seal.

For drawing an average sample of gas at different periods of the day the apparatus may be connected up for the first sample, and an aliquot amount of gas drawn. It may then be left *in statu quo*, and previous to drawing the next portion of the sample the pipe is cleaned of "stale gas" by blowing or pumping through the three-way tap according as the gas to be examined is under pressure or suction.

The sampler is particularly adapted to drawing samples for the purposes of checking the work of boiler firemen.

It is advisable in all cases that the apparatus should be as near the gas flue as possible. The collecting pipe should be as short as possible and of a narrow bore. It may conveniently be widened at the intake end, and any dust arrested by means of a plug of loosely packed asbestos. A piece of stout walled, narrow bore glass tube may be used, enclosed in an outer iron tube, the intervening space being packed with asbestos. The whole sampling apparatus may be carried in a small separate case if desired.

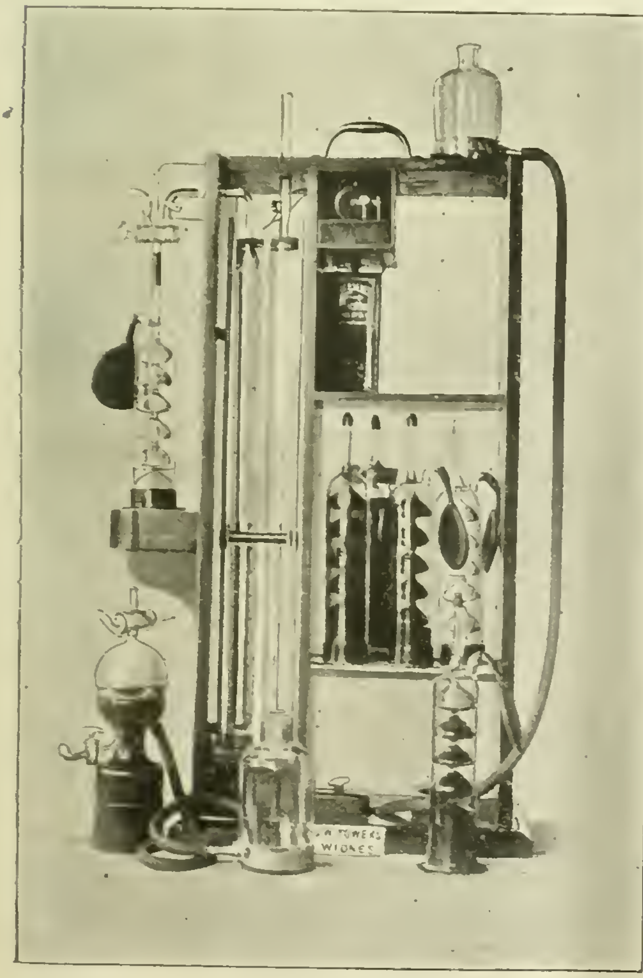
The Gas Analysis Apparatus.

This apparatus presents many features common to a great many of the forms of apparatus in the market. From the results of two years work done in it we feel we are justified in saying that we have produced an apparatus which is—

(1.) Accurate.
(2.) Rapid.
(3.) Portable.
(4.) Simple, and last but not least,
(5.) Easily cleaned.

(1.) Accurate, in that—
We work over mercury.

Fig. 3.



We use a large sample (*i.e.*, up to 100 c.c.) of gas.
We have no capillary tubes which are not under actual measurement.
Our risk of leakage is reduced to a minimum, and
We have a special adjustment for accurate reading without the aid of a cathetometer.

(2.) Rapid, in that the adjustment of the absorption bulbs is very quickly performed, and the pipettes themselves offer special advantages for rapid absorption.

(3.) Portable, in that the whole apparatus, together with—
Four sets of absorption bulbs,
Battery and coil for explosion, and
The sampler already described, is carried in the case before you.

(4.) Simple, in that the whole apparatus carries only two taps, one of these being used for regulating.

(5.) Easily cleaned, the eudiometer being removable by simply disconnecting one rubber joint and drawing out the fore part of the split shelf.

This last point will, we think, recommend itself strongly to chemists who are required to make a considerable number of gas analyses.

The apparatus consists of—

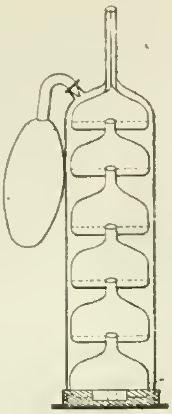
A eudiometer tube fitted with platinum electrodes drawn to a capillary at its upper end, this capillary being bent twice at right angles and fitted with a three-way tap (sketch). The lower end of the eudiometer is drawn out to a tube having a bore of 2 mm., and is connected to the first upright of a horizontal F piece. The second upright of the F piece is connected to a balance or pressure tube of 8 mm. bore, contracted near the top to a fine calibre, and opening out again for a length of 10 cm.

This contraction prevents projection of the mercury from the pressure tube should the levelling bottle be brought up quickly. The pressure tube is of sufficiently wide bore to overcome any error in levelling due to capillary attraction, and enables the apparatus to be worked with very much less mercury than would be necessary if the eudiometer and pressure tube were of the same diameter. The horizontal arm of the F piece is connected to an ordinary Orsat bottle, a large single-way tap of 2 mm. bore set in the base of the apparatus, being interposed. By means of this tap levelling is much simplified. To prevent the tap springing under the high mercury pressure put upon it a mahogany slot-fastened casing is superimposed. Both eudiometer and pressure tube are contained in glass jackets closed at both extremities, and having milk-glass backs. The upper cork of the eudiometer jacket is split, and platinum wires connected to the electrodes are led through it. Arrangements for a constant water flow may be adapted.

The eudiometer is graduated to 100 c.c. in one-fifths, the zero point being at the outer extremity of the bore of the three-way tap. It is supported by a split shelf, through which the lower end of the tube runs, while the rectangular bend at the top is supported by a baize-lined groove in the wooden casing of the stand. The exit capillary tube on the three-way tap has a bulb blown upon it. A sliding, removable spirit level is carried by a pair of uprights in front of the eudiometer and balance tubes, and is fitted with a hair-line, for the purpose of securing an exact reading. With this 0·05 c.c. may be easily read.

The absorption vessels (Fig. 4) are of a special form, consisting of a vertical series of bulbs, mushroom shaped,

Fig. 4.



Absorption Bulbs.

to prevent any gas bubbles being intercepted at their upper end, and flattened at the bottom. The connecting-tube of each bulb in the set rises slightly above the bottom of the bulb immediately above, so that in each bulb a layer of the absorbent liquid is retained. The gas is thus exposed to the action of several successive layers of the absorbent, giving the bulb a greatly increased absorbing capacity, with a minimum of bulk. In addition, the fact that the gas entering at the top is first spread out, and meets with the freshly-wetted sides of the bulb, and then has to pass through a narrow space, where it has practically to bubble through a very thin layer of liquid, greatly promotes absorption, while the absorptive surface of liquid is nearly six times the area of the bulb section at its widest part.

The containing jar is contracted at the top, and fused on to the stem of the bulbs. It contains a tubular opening, through which it can be filled by means of a small funnel with a bent stem. A rubber hand-ball is attached to the tubulure, which serves as a pressure-vessel for forcing the reagent up the bulbs after adjusting them, and protects the reagent from oxidation. A small rubber cap closes the mouth of the bulbs when not in use. They can be emptied by simply removing the rubber ball, inverting the bulbs, and shaking gently. The bulb for copper chloride solution is not fused to the containing jar, but suspended by means of a rubber stopper. This enables a piece of copper gauze to be placed in the containing jar, outside the bulbs.

A comparative trial of the bulbs was made with a sample of producer-gas, selecting CO as the most "obstinate" constituent. A large sample was taken and entirely freed from CO_2 and oxygen. The percentage of CO contained in the CO_2 freed gas was 25·0 per cent. Acid copper chloride solution was used which had been previously used several times. Each trial absorption vessel was filled at the same time with the same solution, and an equal quantity of gas was drawn into the eudiometer for each experiment.

(1.) In a plain glass bell-tube of the usual type, 270 mm. × 26 mm., packed with glass tubes, 18 per cent. of the total amount of CO present was absorbed at the first pass (about 30 seconds duration).

(2.) Using the same sized glass bell-tube, but substituting copper gauze for the packing tubes, as is the practice of many chemists, 28 per cent. of the total amount of CO was absorbed in the first pass of the same duration. Copper gauze is objectionable for accurate work, owing to its strong tendency to retain gas bubbles.

(3.) With our own form of bulbs, using the same amount of gas and the same copper solution, 65 per cent. of the total amount of CO present was absorbed in the first pass, whilst practically the whole of the CO present was absorbed in five passes.

Other trials of our bulbs have, when producer-gas of a somewhat similar composition is worked with, given average results of well over 60 per cent., the best figure reaching to nearly 70 per cent., and the worst being no lower than 55 per cent.

The stand is of mahogany, and has a cupboard which may be rack-fitted for packing six absorption pipettes. It is fitted with shelves holding a dry cell, and a $\frac{5}{16}$-in. spark induction coil. It also contains a drawer for holding the eudiometer tap, a shelf for holding the sampler and a bracket (with packing block) for holding the absorption bulbs in place. It has sliding doors completely enclosing it back and front, and small auxiliary slot-fastened covers are fitted to protect the only two slightly projecting glass parts. A handle for carrying the case is attached.

The apparatus is worked as follows:—100 c.c. of gas is drawn in through the three-way cock, the excess gas over 100 c.c. being expelled in the usual way. The levelling must be very carefully done, as it must be remembered that a difference of 1 mm. mercury is equal to over 13 mm. of water.

The three-way cock is then turned so as to give a through passage from the inlet to the outlet. The first absorption bulb is slipped on, with a well-fitting connecting piece, of medium thick rubber. By pressure on the hand-ball the reagent is made to rise through the bore of the three-way tap until it stands in the upright portion of the outlet tube. By turning the three-way tap quarter round, communication is made between the eudiometer and the absorption pipette, and the reagent falls a little, pulling with it a small quantity of gas from the eudiometer. Pressure is then put on by means of the mercury bottle, and the whole of the gas driven into the pipette. The mercury bottle is then lowered, the speed of the gas travelling back checked by means of the lower tap as the absorbent rises in the pipette, and the three-way tap shut off the moment the absorbent comes up to the tap. After the constituent to be estimated is completely absorbed, the three-way tap is turned half round, and the pipette quickly disconnected and capped. The capillary may be cleansed by drawing in and expelling a little water by suction at a short piece of rubber kept fixed on the outlet of the three-way. Any number of successive absorptions may be made by a moderately skilled manipulator without a bubble of air or a drop of absorbent reagent being drawn into the eudiometer.

Reagents.

(1.) *Ethyl Alcohol* (1 c.c. on mercury followed by 1 c.c. of water on mercury) removes "hydrocarbons." This may be conveniently done by utilising the sampler as the absorption pipette. Butterfield says these hydrocarbons are benzene and its homologues and are identical with the condensation products on cooling to 22° C. (Deville) Sutton says they consist of hydrocarbons of the series C_nH_{2n+1} and C_nH_{2n+2}.

(2.) *Cadmium Chloride* in strong solution, slightly acidified, may be used for the H_2S estimation, but this is preferably estimated in a separate portion of gas with standard iodine.

(3.) *KHO.*—Good caustic potash not purified by alcohol should be used. A solution of 40° Tw. to 50° Tw. is a strength sufficient for all general work.

(4.) *Alkaline Pyrogallol Solution* is undoubtedly the best reagent to use for the determination of oxygen. Clowes has pointed out that when the proportion of oxygen present in the gas is large, carbon monoxide is frequently evolved, the amount generally being greater the higher the ratio of pyrogallic acid to potash in the absorbing solution. Even practically pure oxygen may be wholly absorbed by using a solution of 160 grms. of potash with 10 grms. of pyrogallol in 200 c.c. of solution without evolution of CO. For ordinary work a solution rather weaker in alkali and with a greater proportion of pyrogallol may safely be used.

(5.) *Fuming Sulphuric Acid* of the greatest obtainable strength is used for heavy hydrocarbons = C_2H_4, &c. Before use it is diluted with about 10 per cent. of its volume of ordinary 1·84 H_2SO_4. This reagent gives a higher absorption than bromine water. The latter reagent is, moreover, very troublesome when working over mercury, as it corrodes the mercury surface and makes reading difficult, and frequent cleaning necessary.

It has been shown by many chemists that fuming sulphuric acid removes a small amount of oxygen from the gas. With this statement our experience coincides.

The "reduction products" and SO_3 vapour must be removed by KHO before reading.

(6.) *Cuprous Chloride* may be used in either a hydrochloric acid or an ammoniacal solution. The latter is perhaps slightly the quicker absorbent, but is much more troublesome to prepare.

The HCl solution may be made by boiling 70 grms. of fine copper oxide with 1,000 c.c. of hydrochloric acid for half an hour, adding gradually during the first 10 minutes 56 grms. of copper powder, or fine filings. The solution is made up to its original volume with gradually added water and decanted into a tightly stoppered flask containing rolls of copper gauze. The solution should be colourless after standing a little. It turns brown when in contact with oxygen.

Acetylene and ethylene are both absorbed by ammoniacal cuprous chloride.

The HCl vapour has a very low tension, but in accurate work it may be removed by KHO before reading.

Order of Estimation should be :—"Hydrocarbons," CO_2H_2S, O, C_2H_4, &c., CO.

The H and CH_4 may then be estimated by explosion with oxygen.

CO may also be estimated together with CH_4 and H by explosion, but this we do not recommend. The somewhat tedious CO absorption is certainly done away with, but an additional oxygen absorption must be made. In addition nitrogen free oxygen gas must be used, or else air (in which the per cent. N is known). In the latter case the explosion must be performed on a smaller quantity of gas, not much exceeding 10 c.c.

After the discussion the working of the apparatus was demonstrated.

Manchester Section.

ROOMS OF THE CHEMICAL CLUB, VICTORIA HOTEL.

Chairman: George E. Davis.
Vice-Chairman: Peter Hart.
Committee:

F. H. Bowman.	J. M. Irving.
R. Forbes Carpenter.	*M. J. Lang' n.*
R. S. Dale.	J. Lewkowitsch.
R. Le Neve Foster.	E. Schunck.
B. W. Gerland.	*W. Thomson.*
H. Grimshaw.	D. Watson.
J. Grossmann.	

Hon. Local Secretary:
J. Carter Bell,
Bank House, The Cliff, Higher Broughton, Manchester.

The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Committee:* E. Knecht, I. Levinstein, A. Liebmann, A. Ree, and C. Truby.

SESSION 1896–97.

Meeting held on Friday, May 7th, 1897.

THE PRESIDENT IN THE CHAIR.

A METHOD OF SEPARATING TIN, ARSENIC, AND ANTIMONY.

BY WILLIAM DANCER, B.SC.

JUDGING from the number of papers which have been read before various Sections of the Society of Chemical Industry during the past 12 months, it is very evident that serious efforts are being made at the present time to solve the problem which has long been a source of trouble to analysts, the old methods of separating these metals being not only tedious, but also yielding results which troubled the peace of mind of the analyst considerably as to their accuracy.

I have now the pleasure of bringing before the notice of the Society another attempt to arrive at a solution of the problem by a method upon which I have been working for some few months as opportunity has allowed. At first I intended to claim it as simply a qualitative separation of the three metals, but having obtained some fairly good quantitative results, especially as regards the separation of tin from antimony, I venture to submit the process to the consideration of my brother analysts, premising that I have not fully investigated all the reactions which take place in the course of the analysis, merely giving details of the process and a few of the results obtained, hoping that someone having more time at his disposal and better qualifications than myself may take the question up and work it out thoroughly.

The method depends mainly upon the reactions which take place when the sulphides of the metals are treated with a solution of calcium hydrate.

Antimony sulphide dissolves rapidly and completely as a colourless sulphantimoniate or antimonite of calcium.

Arsenic sulphide is also quickly dissolved and decolorised, but after a short time partially precipitates as arseniate or arsenite of calcium.

Tin sulphides on treatment with a sufficient quantity of lime water are speedily decolorised, and at the same time converted into an insoluble crystalline compound, the exact composition of which I have not determined. Microscopical examination shows three distinct forms of crystals, needles resembling calcium sulphate forming the largest portion of the precipitate, small grains of calcic carbonate, and a third form of crystal which varies according to the conditions under which the reaction has taken place; in the cold, these crystals appear as six-rayed stars. If, however, the temperature has been raised to near the boiling point, cubical crystals are formed.

I think these crystals are stannic or metastannic oxide. According to all the literature I have been able to find on the subject, the precipitate is a sulphostannate or stannite of calcium, but the following reactions I think prove this to be incorrect: it is soluble in cold dilute hydrochloric acid, the solution having a faint opalescence; it is also soluble in dilute nitric and sulphuric acids, hydrated stannic oxide being precipitated on boiling the solutions of the two last-named acids.

Caustic and carbonated alkalis only partially dissolve the precipitated tin, but the whole of it may be dissolved in sulphide of ammonium.

The main fact, however, with which we have to deal is that the tin is entirely precipitated from the solution of sulphide and hydrate of calcium; it settles rapidly and filters easily, and can be washed with boiling water. It will be evident from the above that the qualitative separation of tin from antimony becomes a very simple operation, the mixed sulphides washed free from acid requiring only to be diffused in a sufficient quantity of fresh, clear lime water to entirely decolorise them; this may be done at the ordinary temperature, but heating to about 80° C. accelerates the reaction and renders the tin precipitate denser; the filtrate from this will contain all the antimony, which may be recovered as sulphide by adding an excess of acid. I may here remark that it is immaterial whether the tin is in a stannous or stannic condition, but it is not advisable to have an excess of sulphur present, as sulphide of tin is slightly soluble in polysulphide of calcium; there must be an excess of hydrate of calcium to effect the total precipitation of the tin.

If arsenic be present together with the tin and antimony in quantity not exceeding 5 or 6 per cent. of the tin, the whole of it will be found in solution with the antimony, from which it may be separated in the following manner:—Strong ammonia is added—about 5 c.c. to each 100 c.c. of the solution, which is then heated in a covered beaker to near boiling; glacial acetic acid is added drop by drop until a slight permanent precipitate is formed; it is then boiled until the precipitated sulphide of antimony becomes dense and settles quickly, leaving the liquid clear and slightly acid to litmus paper, though still having a rather strong smell of ammonia. After settling, the solution may be filtered whilst still hot, and the sulphide of arsenic be thrown out by adding an excess of hydrochloric acid.

When a considerable quantity of arsenic is present a portion of it will be precipitated with the tin, and a separation may be effected by treating the precipitate with sufficient yellow sulphide of ammonium to dissolve all, except the calcic carbonate, which is always present when the analysis has been made in an open vessel. The solution is diluted considerably and boiled, caustic lime made by igniting pure calcic carbonate is added gradually, and the boiling continued until the ammonia is nearly all expelled: it is then allowed to cool, and filtered. The sulphide of arsenic may be precipitated from the filtrate by hydrochloric acid.

The quantitative separation of the three metals is conducted on the same lines as above described, with, of course, the necessary precautions of well washing the precipitates, &c.

Perhaps the simplest way of explaining the process will be to describe an analysis of the three metals taken in nearly equal quantities; and I may here remark that, owing to the slight solubility of both hydrate and sulphide of calcium in water, only small quantities of tin can be taken for analysis: otherwise large volumes of liquid would require to be filtered and dealt with, 0·1 grm. of tin as sulphide taking nearly 300 c.c. of lime water to convert it entirely into the insoluble condition.

Taken—

	Grm.
Pure tin	0·1
Antimony as tartar emetic	0·1
Arsenious anhydride	0·1

Fused in small porcelain crucible with 1 grm. dehydrated potassium carbonate and 1 grm. sulphur. After burning off as much of the last-named substance as possible, and cooling, the melt was dissolved in a small quantity of hot water and filtered from a trace of sulphide of iron into a beaker capable of holding 600 c.c., the filtrate—which, together with the wash water, should not be more than 50 to 60 c.c.—carefully treated with hydrochloric acid in slight excess, sufficient only to ensure that all the potash is converted into chloride; pure calcic carbonate is added in sufficient quantity to make it neutral, and 300 c.c. fresh, clear lime water poured in, the beaker placed on gauze over a lamp and heated to about 80° C. for four or five minutes. A bulky nearly white precipitate is formed, and the liquid becomes pale yellow in colour; after cooling and settling, 50 c.c. of the solution are passed through a filter, using preferably one of the hardened filters; an equal volume of clear lime water is added. If no further precipitation takes place, the whole of the solution is decanted through the filter; the precipitate is washed twice by decantation with lime water and then with hot water, leaving as much as possible of the precipitate in the beaker. The filtrate is now placed over a lamp to boil down, whilst the precipitate is dealt with in the following manner:—The portion on the filter is washed off with hot water into the beaker containing the rest by means of the wash bottle. The filter is subsequently moistened with yellow sulphide of ammonium and thoroughly washed with hot water into the beaker, 2 or 3 c.c. more sulphide of ammonium are added, the whole made up to about 400 c.c. and heated to boiling; pure caustic lime is now gradually added, about 1 grm. in all, and the boiling continued until the tin falls out of solution; this will occur when the ammonia is all driven off. The solution is pale yellow in colour, and when cold should give no further precipitate on addition of lime water.

After filtering, the precipitate is dried and ignited, and separated from the calcium by boiling in dilute nitric acid, again filtered, well washed, and weighed as stannic oxide.

When treated in this manner, the precipitated tin retains its crystalline condition, and filters and washes easily. The separation of arsenic from tin by this method is not complete if, in the alloy or compound taken for analysis, the arsenic is in excess of the tin; in such a case the above process would require to be repeated.

The filtrates from the tin containing the antimony and arsenic are added together and boiled down to about 400 c.c., 30 c.c. of ammonia (sp. gr. 0·880) run in, and glacial acetic acid added drop by drop until a slight permanent red precipitate is formed; the boiling is continued until the sulphide of antimony becomes dense and settles quickly, leaving the solution clear and slightly acid to litmus paper. The boiling must not be too long continued, or the sulphide of antimony may become so finely divided as to pass through the filter paper. After settling, the sulphide of antimony is filtered off, and as it still contains a small quantity of arsenic, it is dissolved in ammonia and lime water, a few drops of sulphide of ammonia being added, if necessary, and the solution again precipitated, boiling with acetic acid. The antimony is determined by titration with iodine in alkaline tartrate solution, dissolving the sulphide in strong hydrochloric acid in presence of a little iodine—a process devised by Dr. J. Clark (this Journal, 1896, 255).

The arsenic is precipitated from the filtrates from the antimony by addition of rather a large quantity of strong hydrochloric acid as sulphide, and only completely after long standing. After filtering and washing, the sulphide of arsenic is dissolved off the filter into a large flask with strong ammonia, diluted considerably with water, and boiled until free from sulphuretted hydrogen, a few drops of dilute sulphuric acid added, and again boiled until clear and free from precipitated sulphur; it is then cooled, made alkaline with carbonate of soda, starch solution added, and titrated with iodine.*

Results of analysis:—

Tin	0·1003 found.
Antimony	0·100 „
Arsenic	0·098 „

Separation of Tin from Antimony.

Taken.		Found.	
Tin.	Antimony.	Tin.	Antimony.
Grm.	Grm.	Grm.	Grm.
0·100	0·100	0·103	0·0988
0·1045	0·152	0·1046	0·156
0·042	0·152	0·043	0·151
0·100	0·100	0·103	0·101

Separation of Tin from Arsenic.

Taken.		Found.	
Tin.	Arsenic, As_2O_3.	Tin.	Arsenic, As_2O_3.†
Grm.	Grm.	Grm.	Grm.
0·100	0·109	0·1006	0·094
0·100	0·025	0·1018	0·0198

Taken.		Found.	
Tin.	Arsenic, As_2O_3.	Tin.	Arsenic, As_2O_3.
Grm.	Grm.	Grm.	
0·100	0·035	0·100	Not determined.
0·100	0·086	0·101	„ „

Separation of Antimony from Arsenic.

Taken.		Found.	
Antimony.	Arsenic, As_2O_3.	Antimony.	Arsenic, As_2O_3.‡
Grm.	Grm.	Grm.	Grm.
0·100	0·100	0·101	0·099
0·1098	0·0866	0·110	Not determined.
0·0549	0·0433	0·050	„ „

* This is a modification of De Clermont and Frommel's process, Comptes Rend., vol. 66, p. 828.

† The arsenic determined as magnesia salt.

‡ Arsenic determined by boiling solution of sulphide in ammonia with large quantity of water and titrating with iodine in alkaline solution.

I may here note that arsenic may be separated from antimony by evaporating the calcium solution of the sulphides to complete dryness: the sulphide of arsenic is decomposed; the sulphide of antimony remaining unchanged may be dissolved out of the residue by repeated washings with lime water, filtering off, and reprecipitating as sulphide by adding hydrochloric acid. The residue, containing arsenite of calcium and carbonate, is cautiously treated with dilute sulphuric acid, filtered off from excess of sulphate of lime, and a little free sulphur boiled to expel sulphurous acid, made alkaline with carbonate of soda, cooled, and titrated with iodine.

Taken.			Found.		
Tin.	Antimony.	Arsenic.	Tin.	Antimony.	Arsenic.
Grm.	Grm.	Grm.	Grm.	Grm.	Grm.
0·100	0·103	0·100	0·1003	0·101	0·088

I have made a few experiments with hydrate of barium on the sulphides of the three metals, but, so far, they have not proved very satisfactory.

In conclusion, my thanks are due to Messrs. Roberts, Dale, and Co., of this city, for permission to publish these results, which have been obtained in their laboratory.

NOTE ON THE ADULTERATION OF SILKS.

BY M. I. LANGDON, PH.D.

After a highly interesting historical sketch of the silk industry, and a slight *résumé* of the processes in vogue for the weighting of silk, Sir Thomas Wardle, at the last meeting of the Section, attributed the decay of the English silk industry to this weighting or, as termed in the lecture, adulteration of silk. I must take strong exception to this ascribed cause of decay, which, however, certainly did commence about the same time as the Swiss and French dyers started the chemical weighting of silk. This weighted silk was used for the manufacture of low-priced fabrics, and a great demand for these arose, and was not catered for to any degree by the English silk manufacturers, either through want of energy on their part, or else inability to have their yarns weighted to the desired point by the English silk dyers. If the former, then the cause of the decay is self-evident; if the latter, were any steps taken to meet the demand?

After the reading of the paper, I put the following question to Sir Thomas Wardle:—Can the English silk dyer weight black warps up to 26—28 oz., and black souple up to 40—42 oz.; and, if not, has any attempt been made, as in the U.S.A., by means of imported talent, to arrive at this result? The reply was, that yarns could be weighted up to 24—26 oz. in this country; consequently I must assume that the English silk dyer cannot supply the required demand for the heavier weighted silk yarns, and I therefore should like to draw the attention of the English silk dyer to two possible ways of meeting this demand and the foreign competition: firstly, by following the example of the leading calico printers of this country, and engaging highly trained chemists to work out the different processes for them (I believe English calico printing has benefited largely by this step, and other industries might well follow suit, especially as there is now a fair supply of highly trained *English* chemists), or else by importing Swiss or French foremen silk dyers, to teach the most modern continental methods of silk dyeing. This latter was the means adopted in the U.S.A. Since the importation of one dyer about 25 years ago, an industry has been built up by periodic importations which now employs some two or three thousand men under the necessary scientific direction, both chemical and mechanical, in works fitted up with the most modern machinery. Now, to meet this demand for highly weighted silk yarns, and to turn the same out in quantity, if the English silk dyers cannot or will not do it (although I feel sure there are several English silk dyers who, if only properly supported, would come to the front), why do not the manufacturers combine and start a co-operative dyehouse provided with modern machinery and under scientific direction to dye the yarns in quantity. By this I do not mean dyebecks to dye about 50 lb. of yarn at a time, but becks capable, for colours or black warps, of holding at least 100 lb., and for black souples 250 lb. (I may say from practical experience that becks this size are in use): and I feel convinced that if this is done, and the manufacturers cater for the taste of the public, and not try to force on them what they do not require, the English silk industry would soon occupy its old predominant position.

There is a silk association in this country which certainly has succeeded in obtaining some little employment for the Spitalfields weavers, but has evidently missed the true question, *viz.*, of meeting the demand of the public in this and other countries for a low-priced article, and also the advisability of calling in chemical assistance—not of chemists who have learnt a little qualitative and quantitative analysis during the course of a year or two at college—but of men trained in research work. I believe practical and well-trained chemists are conspicuous by their absence in the Leek and Macclesfield districts, although I am told one or two of the dyers do sometimes seek outside assistance in this line.

Much as it would be pleasing to see Sir Thomas Wardle's idea of the practical abolition of silk weighting carried out, such a scheme in my opinion would not be feasible, and might even be considered visionary. Some little good might be done if the dyers of other countries would support the Swiss dyers in their endeavours, as mentioned by Sir Thomas Wardle, to limit the weighting. This they would doubtless have to do in self-protection owing to the dangers of excessive over-weighting: and in furtherance of this view would suggest the following limits:—

	Oz.
White and coloured warps	16—18
" " souples	18—20
Black warps	22—24
" souples	40—42
" cardinette	80—84

There is another view of the question, *viz.*, Can the silk manufacturer make use of the weighted yarns when he receives them, and does he know what weighted yarn is required for any fabric he may be required to match? This I do not think, however, comes within the province of this Society.

I trust that these few remarks may cause a discussion on this interesting subject, which should not be lightly passed over by this Society.

Discussion.

Mr. Harry Grimshaw thought it most important to ascertain the cause of decline in the silk industry of Great Britain. Sir Thomas Wardle had suggested that it was due to adulteration, while Mr. Langdon attributed it mainly to the lack of competition between English dyers and those abroad in the production of a cheap silk. He was inclined to think that the distribution of wealth would have a tendency to produce a great change in the demand for cheaper silks; but then there was the question of cheap labour abroad, which would have to be taken into account before we could compete successfully with the foreigner.

Dr. Bowman remarked that it was difficult for the British silk trade to compete with that of other countries, inasmuch as the foreigner had not the same desire to comply with the Adulteration Acts as was the case in England.

Dr. Bailey thought the question of adulteration in the silk trade was a very wide one, and the constant change of fashion was in a great measure responsible for the demand for a cheap article. It was a question which could not be considered from a chemical point of view.

A NOTE ON THE THEORY OF DYEING.

BY ERNEST BENTZ, F.C.S., AND FRANK J. FARRELL, B.SC.

To explain according to the chemical theory the nature of the phenomena of dyeing in the case of wool and silk, it has been assumed that the fibres in question are amido-

carboxylic acids, the constitution of which may be provisionally represented by the formula—

$$X{<}\begin{matrix}-NH_2\\-COOH\end{matrix}$$

in which X is an unknown radical or radicals.

According to this view the acidic colouring matters (sulphonic acid derivatives of azo dyestuffs, nitro-phenolic compounds, eosins, &c.), in dyeing, saturate the amido groups, while the dyeing with basic colouring matters is a result of the attraction exerted upon the easily dissociated colour bases by the carboxyl groups. Without entering into any discussion of the nature of the process of dyeing wool and silk, it is sufficient to point out that the conclusion regarding the chemical natures of wool and silk (about which exceedingly little is known) has been largely deduced from a consideration of the dyeing properties of the fibres. The evidence apart from this is meagre, and, as far as the presence of carboxyl groups in silk is concerned, is principally the fact that carboxy compounds are formed when that fibre is decomposed by barium hydrate (Schützenberger and Bourgeois), dilute sulphuric acid (Cramer, Waltenberger, and Städeler, and Weyl), or alcoholic potash (Richardson). The compounds produced when wool is similarly decomposed have not been so closely studied. With regard to the amido groups, P. Richard (Bull. Soc. Ind. Mulhouse, 1888; this Journal, 1888, 841) has shown that wool and silk, when they are treated with nitrous acid, become diazotised and give coloured compounds on bringing them into alkaline solutions of phenols (see also Richardson, this Journal, 1893, 426; Dreaper, *ibid.* 1894, 95; Watson Smith, *ibid.* 1896, 245). Doubt has been expressed concerning the interpretation of this reaction. We have, therefore, repeated Richard's work. We find the products of the action of nitrous acid on wool and silk behave in all respects as diazo compounds, combining with phenols and amido bases, undergoing the general reactions for this class of substances observed by Griess and Sandmeyer, &c.; and, further, that the wool and silk azo compounds are decomposed when heated with an acidified solution of stannous chloride, the regenerated wool and silk being capable of being again diazotised and combined with phenols and amido compounds.

The quantity of diazotisable nitrogen present in wool is only 1·0—1·2 per cent., that is, $\frac{1}{15}$—$\frac{1}{12}$ of the total nitrogen present. Notwithstanding this, it might be supposed that the dyeing with acidic colouring matters took place through the medium of the diazotisable nitrogen (presumably the free amido group), the rest of the nitrogen being unaffected by the processes adopted for the removal of the diazotisable amido group, since there is sufficient of the latter to account for the absorption of the amounts of colouring matters usually applied. We find, however, that the wool which has been deprived of its diazotisable amidic nitrogen, dyes with acidic colouring matters apparently as well as does wool in its ordinary state. The dye-baths are exhausted in both cases to the same extent, and the colours, so far as our experiments range, are as fast to soaping and to light. As might be expected, the basic colouring matters dye the "de-amidated" wool equally as powerfully as they dye ordinary wool.

The method of "de-amidating" wool referred to consists in treating it with nitrous and hydrochloric acids at the ordinary temperature for 10—12 hours, washing, and boiling with (1) alcohol, or (2) cuprous chloride and hydrochloric acid, or (3) water. The colours of the wool thus treated are respectively (1) brown, (2) very pale buff, and (3) brown. The modified wool, after treatment again with nitrous acid, does not in any of the three cases yield colours when acted upon by alkaline phenol solutions. It contains, as mentioned above, 1·0—1·2 per cent. less of nitrogen than the original wool.

Silk may be similarly "de-amidated," and the product behaves towards colouring matters like ordinary silk.

It would appear, therefore, that in the dyeing of wool and silk with acidic colouring matters the amido groups present in the fibre-substances play either no part at all or at most an unimportant one.

POTASSIUM PERCARBONATE, AND THE OXIDATION OF THE LEUCO BASE TO ACRIDINE YELLOW.

BY ARTHUR H. LYMN.

Potassium percarbonate is obtained by electrolysing a saturated solution of potassium carbonate at low temperatures, about -10°. The salt is deposited in the anode space, and is filtered and dried rapidly to prevent decomposition. It is at first bluish, and on drying becomes white.

When heated above 100° it is decomposed as follows:—

$$K_2C_2O_6 = K_2CO_3 + CO_2 + O.$$

In ice-cold water it dissolves without being decomposed. The decomposition is quicker and more complete according as the temperature is raised, and takes place in accordance with the following equation:—

$$K_2C_2O_6 + H_2O = 2KHCO_3 + O.$$

When brought into contact with alkalis and gently heated a current of pure oxygen is evolved—

$$K_2C_2O_6 + 2KOH = 2K_2CO_3 + H_2O + O.$$

With acids, carbon dioxide is evolved and hydrogen peroxide is formed quantitatively—

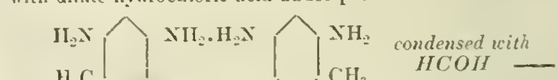
$$K_2C_2O_6 + H_2SO_4 = K_2SO_4 + 2CO_2 + H_2O_2.$$

From the above it appears that the salt oxidises in acid, neutral, and alkaline solutions.

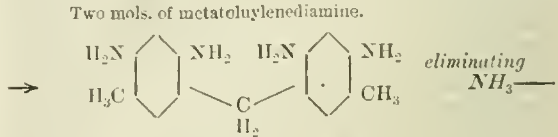
Drs. Constam and von Hansen have tried its bleaching powers on cotton, wool, jute, silk, hair, &c., with more or less success, the experiments having been only conducted on a small and incomplete scale.

An abstract on the salt will have been seen in the Journal of this Society, 1896, 815, and further details will be found in the Journal of the Society of Dyers and Colourists, Vol. XII., No. 12; also in the "Zeitschrift für Electrochemie," Vol. III., p. 445, a new article has just come out under the title "Ueber die Darstellung and Eigenschaften des Kaliumpercarbonats," by A. von Hansen.

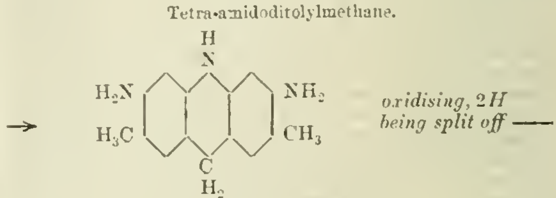
Oxidation of the Leuco Base to Acridine-Yellow.—Ger. Pat. 52,324 (Leonhardt and Co., in Mühlheim). As is well known, the leuco base is prepared by the condensation of metatoluylenediamine, C_6H_3, $\overset{1}{C}H_3$, $\overset{2}{N}H_2$, $\overset{4}{N}H_2$, with formaldehyde in presence of sulphuric acid to tetra-amidoditolylmethane, from which the leuco base is formed, through elimination of one molecule of ammonia, by heating with dilute hydrochloric acid under pressure.



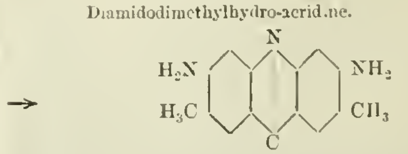
Two mols. of metatoluylenediamine.



Tetra-amidoditolylmethane.



Diamidodimethylhydro-acridine.



Diamidodimethylacridine or acridine yellow.

The condensation can take place either in methyl alcoholic or aqueous solution, preferably in the latter.

According to the patent, the final oxidation is carried out by means of ferric chloride, air being also technically used; bichromate, nitrous acid, &c., can also take their place as oxidisers. The dyestuff itself in dilute solution gives a characteristic yellow and green fluorescence. This hydroacridine takes up oxygen easily, the dye being somewhat precipitated on exposure to air, and entirely by boiling the solution for some time or passing air through in the heat. The dry leuco base is fairly stable, and is readily soluble in water and dilute acids.

The product obtained by heating 7 grms. of the tetra-amidoditolylmethane with 18 grms. hydrochloric acid (concentrated), and 50—55 c.c. water in a sealed tube at 150° C. for six hours, was diluted with water, and to this was added potassium percarbonate and acid. On addition of acid the liquid frothed considerably, which froth disappears slowly. The percarbonate was added until no further precipitate was formed. The filtrate gave no precipitate with ferric chloride, or in passing air through in the heat, from which one may assume that the oxidation was complete. The reddish-brown precipitate was filtered, washed, and dried in a vacuum desiccator, and weighed 3·5 grms. The substance so obtained was redder and had a purer appearance than the commercial product. It was soluble in water, the dilute aqueous solution showing the characteristic yellow and green fluorescence of acridine yellow. It dyed silk with the same strength as the product made by the author by oxidation with ferric chloride, but, if anything, somewhat purer in appearance, and had a very much greater colouring power than the commercial product, a ½ per cent. skein dyed with this substance being more strongly dyed than a 2 per cent. skein with the commercial product. The percarbonate would therefore seem to be a very suitable oxidising agent in this case. It possesses the great advantage that the bicarbonate which is formed on oxidation in neutral solution, as also the chloride or other salt formed in acid solution, are easily washed out of the product with water, and it is thus unnecessary to wash with hydrochloric acid, as is the case after oxidation with ferric chloride.

Experiments on a larger scale.—125 grms. of the condensation product obtained from 203 grms. of metatoluylenediamine with formaldehyde and sulphuric acid were heated for about eight hours under 8—10 atmospheres pressure in an autoclave with 324 grms. concentrated hydrochloric acid and 1 litre of water. The product so obtained consisted of a dark red liquid, a sediment of hydroacridine crystals, and a difficultly soluble sediment. The whole was diluted, stirred up, and divided as equally as possible into four parts, two of which were oxidised with ferric chloride, one with air, and one with potassium percarbonate. Practically speaking, the yields were equal, 26 grms. being obtained by ferric chloride (with double quantities), 13 grms. with air, and 12 grms. with percarbonate—in each case about 25 per cent. of the theoretical quantity from the metatoluylenediamine employed. The oxidation with the percarbonate was executed under precisely the same conditions as in the previous experiment. These products were not quite as soluble in water as the products made on a small scale, and leave considerable ash on burning. The commercial product shows in these respects very similar properties.

In order to ascertain whether the oxidising action of the percarbonate in acid solution is due to the intermediate formation of hydrogen peroxide according to the equation—

$$K_2C_2O_6 + 2HCl = 2KCl + 2CO_2 + H_2O_2$$

the following experiments were undertaken.

Four sealed tubes, each containing—

	Grms.
Condensation product	31
Concentrated HCl	9
Water	27

were heated in the same furnace for six hours at 150° C. In each case the contents were previously made as homogeneous as possible. The contents of—

No. 1 were treated with hydrogen peroxide alone, whereby practically no precipitate was formed; but on addition of more acid the substance came down freely.

No. 2 were treated with hydrogen peroxide and acid.

No. 3 were treated with potassium percarbonate without additional acid.

No. 4 were treated with potassium percarbonate and acid, as in previous cases.

In each case the oxidising agent was added until no further precipitate was formed; moreover, ferric chloride produced no more precipitate in each portion. The differences in the yields are probably due to the fact that it is impossible to subject the tubes to exactly the same temperature in the furnace.

Results.—No. 1 gave 1·2 grms. chocolate-brown product.
No. 2 gave 1·8 grms. light-brown product.
No. 3 gave 1·3 grms. brown product.
No. 4 gave 1·0 grm. chocolate-brown product.

All four products are soluble in water, possess the characteristic fluorescence, and give the same effect on silk. These substances have a different appearance one against another, and their densities also seem to vary, insomuch as the one collects at the top and the other at the bottom of the liquid.

Experiments with Isolated Hydro-acridine in a Carbonic Acid Gas Atmosphere.—The hydroacridine is partly oxidised by air at the ordinary temperatures when in a wet state, but can be isolated in a fairly pure state by filtering the contents of a sealed tube in an atmosphere of carbon dioxide under suction and quickly transferring to a vacuum desiccator. This product melts above 300° under decomposition. Four small portions were dissolved in water.

To No. I. hydrogen peroxide was added without acid, and very little precipitate was formed.

To No. II. hydrogen peroxide and acid in excess were added. In this case a copious precipitate was formed as above.

To No III. potassium percarbonate was added without additional acid and the precipitate was formed.

To No. IV. potassium percarbonate and acid were added. The precipitate came down freely, as in the last two cases.

On repeating the experiments with isolated hydroacridine in the air with an hydrogen peroxide purchased in England, the author found that the precipitate was copiously formed without additional acid as in the other cases. This may perhaps be accounted for by a difference in the amount of available oxygen in the two hydrogen peroxides, or a difference in the quantity of acid in solution.

It is more economical to work with the percarbonate with excess of acid than otherwise, since in the latter case much of the oxygen formed escapes. The experiments seem to confirm the assumption that the action of potassium percarbonate in acid is due to the intermediate formation of hydrogen peroxide.

All the products had a much higher dyeing power than the commercial product, both on silk and on cotton mordanted with tannin. On cotton the colours are of a greenish-yellow, but the percarbonate product shows a much purer yellow than the others. On silk the difference is not so noticeable.

The action of ammonium persulphate was also tried under the same conditions as the percarbonate, but gave a very dark grey substance which neither produced in water solution the characteristic fluorescence, nor was it completely soluble. It dyed silk brown, at the same time partly taking away the gloss.

Summary.—In the oxidation of diamidodimethylhydroacridine to acridine yellow—

Potassium percarbonate with additional acid gives very good results.

Potassium percarbonate without additional acid gives also good results, though in this case the action is slower and not so economical.

Hydrogen peroxide with additional acid acts similarly to to the percarbonate under these conditions.

Hydrogen peroxide without additional acid also gives the precipitate.

Ammonium persulphate gives an entirely different product, which was not further investigated.

Attempts to oxidise the leuco base to crystal violet with potassium percarbonate also made in the Polytechnikum laboratories did not prove successful.

The above investigation formed part of that made for obtaining the diploma in the technical chemical course of the Federal Polytechnic School in Zürich, and the author wishes here to express his most sincere thanks to Prof. Gnehm for his kind help and suggestions in it, and to Dr. Constam for furnishing some notes on the salt as well as a sample of the same.

Dr. Grossmann exhibited an apparatus for testing indigo and other colouring matters, a full description of which had been read by him the previous evening at the Bradford Section of the Society of Dyers and Colourists.

Newcastle Section.

Chairman: F. S. Newall.

Vice-Chairman: G. T. France.

Committee:

A. Allhusen.	John Morrison.
P. P. Bedson.	John Pattinson.
T. W. Hogg.	W. W. Proctor.
H. Louis.	W. L. Rennoldson.
T. W. Lovibond.	G. Sisson.
N. H. Martin.	J. E. Stead.

Hon. Local Secretary and Treasurer:
Saville Shaw, Durham College of Science, Newcastle-on-Tyne.

SESSION 1896-97.

THE SAMPLING AND ANALYSIS OF COKE, AND VALUATION OF COAL FOR COKING PURPOSES.

BY W. F. KEATING STOCK, F.I.C.

Erratum in Discussion.

(*This Journal*, 1897, 308.)

In Mr. Stock's reply, read from line 6 as follows:—With regard to Mr. Proctor's question, he (Mr. Stock) thought that the writer in the *Comptes Rendus* was at fault in that he attempted to extract his phosphorus with *aqua regia*; for it was well known that if much peroxide of iron was present the acid mixture had little effect upon it until the nitric acid was largely broken up.

And from line 13 read:—He had not noticed the occurrence of titanic acid in coal ash, although local fire-clays, associated with coal seams, sometimes contained as much as 3 per cent. In presence of titanic acid it would be difficult to extract the phosphorus, &c.

Nottingham Section.

University College, Nottingham.

Chairman: F. J. R. Carulla.

Vice-Chairman: J. M. C. Paton.

Committee:

L. Archbutt.	F. E. Lott.
R. M. Caven.	*T. Mason.*
F. Clowes.	J. J. Sudborough.
H. Forth.	*C. Taylor.*
W. G. Johnston.	G. J. Ward.
J. F. Kempson.	

Treasurer: S. J. Pentecost.

Hon. Local Secretary:
J. T. Wood, 29, Musters Road, West Bridgeford, Nottingham.

The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Chairman:* J. O'Sullivan. *Committee:* S. F. Burford and F. J. R. Carulla.

SESSION 1896-97.

Meeting held at Wyvern Hotel, Leicester, on Wednesday, April 28th, 1897.

MR. F. J. R. CARULLA IN THE CHAIR.

AN ATTEMPT TO PREPARE METHYLENE.

BY J. J. SUDBOROUGH.

After drawing attention to the great interest—from a theoretical point of view—attaching to the preparation of compounds of the type of carbon monoxide, a compound which stands in quite an isolated position as regards the numerous other carbon compounds, the author pointed out the various attempts which had been made to prepare the hydrocarbon methylene CH_2, drawing special attention to those of Butlerow.

The communication concluded with an account of some experiments which the author himself had undertaken some three years ago at the suggestion of Prof. Victor Meyer. The substance made use of in each case was methylene iodide, CH_2I_2. When heated to its boiling point for some time, a considerable amount of iodine separated out, but little or no gas appeared to be evolved. When the iodide was dropped on to sand contained in a flask and heated to about 500°, it was completely decomposed; much carbon was deposited, and hydrogen iodide, together with other gaseous products, were also formed, but no trace of ethylene—a compound which is generally formed on the decomposition of methylene iodide—could be found. Methylene iodide reacted vigorously when warmed with an excess of fine silver powder: a certain amount of carbon was deposited and a considerable quantity of gas evolved. The greater part of this latter consisted of ethylene, C_2H_4, since it readily combined with bromine, yielding ethylene dibromide (b. pt. 130° C.). A certain amount of a hydrocarbon which was not absorbed by bromine, but no hydrogen could be found in the residue. The hydrocarbon on analysis proved to be methane, CH_4, no doubt formed by the decomposition of methylene into methane and carbon.

Discussion.

Prof. F. Clowes said that personally he was gratified to hear the record of Dr. Sudborough's experiments. A record of failures was by no means without instruction. While listening to the paper, it had struck him that acetylene, C_2H_2, might be persuaded to part with half its carbon, and thus act as a starting point. There would, of course, be difficulties in the way, as the author had mentioned, but there seemed to be no reason why C_2H_2 should not part with some of its carbon.

The CHAIRMAN suggested that some substance might be found that would hold methylene in solution much in the way that ammonium was retained by mercury in the well-known ammonium amalgam.

Dr. J. J. SUDBOROUGH, in reply, said that Prof. Clowes seemed to think that he (the author) had succeeded in decomposing methylene into carbon and methane, CH_4. But, judging from the experiments, he thought that there was no chance whatever of achieving such a result. Although ethylene could be decomposed into carbon and methane, it was doubtful if the temperature at which the reaction occurred was high enough to bring about such a decomposition. It was more probable that the methane was obtained by the decomposition of the methylene first formed.

THE REMOVAL OF MINERAL OIL STAINS FROM COTTON FABRICS.

BY S. J. PENTECOST, F.C.S.

AMONG the many difficulties which beset the path of the bleacher and dyer of cotton fabrics, perhaps none are more serious than those arising from the presence in the finished goods of mineral oil stains. The lace bleacher finds it extremely difficult to get rid of the blacklead that is used to lubricate the lace machine, and which occasionally finds its way on to the fabric. But in this case he can see clearly when the black stain has been removed; while in the case of a mineral oil stain, he knows that though it has apparently been completely removed, it is liable to reappear, after goods have been stocked a few weeks, in the form of a light yellow stain.

The presence of these unsaponifiable oils on the fabric, arises generally from one or both of two causes :—

1. It often happens that drops of oil from the machinery or shafting above the fabric, fall upon it and thus cause a stain; while possibly a bearing which has been allowed to get hot and to "squeak," has a superabundance of oil applied to it, with the result that the heated oil is thrown on to the piece, thus causing a large number of small stains. These spots of oil often contain iron or brass dust carried from the bearing.

2. Some manufacturers of lace and hosiery goods find it necessary to lubricate their yarns before putting into the machine, and for this purpose a good saponifiable fat or oil is generally used, which of course helps, rather than hinders, the cleansing of the goods in the bleaching process. But the temptation offered by cheap oil leads some to use so-called stainless oils, which are mixtures of mineral and vegetable oils, the former in some cases largely preponderating. So long as a good emulsion is produced by mixing it with a little dilute alkali or with soap, the presence of the mineral oil is not noticed by the maker of the fabric, and the latter is thus rendered liable to be faulty when finished.

It is much to be deplored that mineral oils should be used for this purpose, but if the question of cost makes it imperative or desirable, the proportion of hydrocarbon oil mixed with the saponifiable oil should never exceed 50 per cent.

Of the stains from these two causes, the most persistent are those produced by the heated bearing or shafting, and if the goods containing them have lain in stock for some time—say three months or more—the difficulty of removing them is much increased. Indeed, it is often found, when the goods have been bleached, that the fibre has become disintegrated, and a small hole in the fabric is the result where the stain had previously appeared.

In the case of fabrics which require "gassing," such as some lace fabrics, muslins, cambrics, &c., no doubt the heat causes the mineral oil to penetrate more deeply into the fibres, and thus renders the stains more difficult of removal.

Various methods have been proposed for the elimination of these oil stains besides those used by many bleachers who keep their processes secret. The value of many of these latter may well be doubted, especially if that mentioned by a bleacher who gave evidence in a recent law case may be taken as a type. Speaking of the possibility of removing mineral oil stains from cotton fabrics, he said that this could be done by a process of his own, which he could not divulge, but that even this process tendered the fabric. No wonder the judge remarked that in that case it was quite as well to keep the process a secret.

Perhaps one of the most usual methods of dealing with these stains in detail, as well as one of the most effective, is to impregnate them with olive oil or olein, allow it half an hour to thoroughly permeate the stained part, then boil the goods in a caustic soda lye for six or eight hours.

It should be remarked here that if the mineral oil used is mixed with an equal proportion of a vegetable oil, it is much more easily removed, and the ordinary process of bleaching is generally sufficient, if the stains are fresh. In a paper published in the Bull. de la Soc. d'Industrielle de Mulhouse in 1889, E. Scheurer shows that a mixture of 5 parts of Scotch petroleum with 5 parts of cotton, rape, or olive oil is removed by one lime boil of six hours, followed by a sour, and ley boil of soda ash and resin for 10 hours, while a mixture of 6 parts of Scotch petroleum with 4 parts of vegetable oil was *not* removed by that treatment, and involved danger of the after-development of the stains on the finished fabric. He also found by experiment that a vegetable oil mixed with the mineral oil was more easily removable than a mixture of *fatty* acid (*viz.*, olein) and unsaponifiable oil, when boiling with soda ash and resin. Further, a mixed oil containing 6 parts of Scotch petroleum and 4 parts of vegetable oil was more readily removed when the soda ash and resin boil was preceded by a lime boil and sour. He suggested that this was probably owing to the lime soap formed with the cotton fats, absorbing the mineral oil from the interior of the fibre, in the same way that pipeclay and plaster act when applied to remove grease stains from finished fabrics. In boiling with caustic soda he found the addition of a small quantity of resin to the boil (about 1 oz. to 2 galls. ley) was an advantage in removing the oil; it formed a sufficient quantity of soap to emulsify the natural fats of the cotton.

Another method which has been successfully used for the removal of these stains is to steep them all night in a bath containing—

Soap	1 lb.	4 galls.
Ammonia, 0·880 sp. gr.	4 oz.	
Turpentine	2 oz.	

afterwards boiling and bleaching in the usual way.

Quite recently E. Schweitzer has proposed the use of aniline oil or phenol in soap solution for removing mineral oil stains. In an article in the Bulletin de la Société d'Industrielle de Mulhouse, he says: Since aniline possesses the property of dissolving many bodies which are insoluble in ordinary solvents, he conceived the idea of trying its effect for the removal of stains (*e.g.*, mineral oil stains). As a result of his experiments he patented a process in which the stained goods are steeped in a bath of 5 quarts aniline oil, 9 lb. soap in 20 gallons of water for half to five hours, and then boiled and bleached in the usual way; an alternative method is to add the aniline oil or phenol to the lye in the kier just before boiling, and to proceed as usual. When phenol is used half the quantity suffices. A patent for a similar process was applied for about the same time by Messrs. Ferguson and McMeekin, of Belfast, who immerse the fabric in a liquid said to be capable of dissolving the mineral oil. The composition of the said liquid is :—

H_2O	10 to 15 galls.
Soap	8 lb.
Soda ash	4 „
Ammonia extract of soap	2 „
Oil	1 pint.

The oil may be shale oil, paraffin oil, resin oil, aniline, or phenol.

The fabric is immersed for 5 to 12 hours, and it is claimed the treatment removes the oil stains and improves the finish of goods. The first of these two methods was reported to be satisfactory by M. Jacquet after a trial in which 2,000 pieces of cotton cloth were bleached; but he

admitted that a number of the stains still remained after the treatment.

Pressure of business has prevented me making the series of bleaching trials which this treatment appears to me to demand; but a short trial was made on some well-twisted cotton fabric which had been previously bleached.

The fabric was impregnated with ordinary American mineral oil over one half its surface; then divided into four equal parts (each having half stained). They were then left in a stove at 110° F. for two hours, and treated as follows:—

No. 1, boiled 1 hour with 1 oz. of soap, $\frac{1}{4}$ oz. of phenol in 1 pint of water.

No. 2, boiled 1 hour with 1 oz. of soap, 1 oz. of aniline oil in 1 pint of water.

No. 3, boiled 1 hour with 1 oz. of soap in 1 pint of water.

No. 4, steeped in $\frac{1}{4}$ oz. of turpentine, $\frac{1}{2}$ oz. of ammonia, and 1 oz. of soap in 1 pint of water, and then boiled for five minutes.

After rinsing and drying, the stain appeared to be removed in every case; but on re-examining them two days after, Nos. 1 and 2 showed the stain distinctly, while Nos. 3 and 4 had a very faint appearance of stain.

The idea implied in M. Schweitzer's article, that the aniline or phenol acts as a solvent for the mineral oil, is, I believe, a mistaken one. Probably their penetrating power may enable them to permeate the cotton wax and substance of the fibre, and to bring through the cell walls the mineral oil which has found its way to the centre of the fibre, which the alkalis alone cannot touch. In other words, they may assist the emulsive action of the soap and alkali.

It is found in practice that if, before boiling, the fabric is well scoured in a dolly or in washing stocks with a soap solution and the addition of a little carbonated alkali, the mineral oil stains are more thoroughly and easily removed, than when boiled in the kier at once. By this procedure also, any iron or brass dust off bearings or shafting is removed before being fixed by the acid, &c. used in bleaching.

Much of the unsatisfactory work arising from these stains might doubtless be avoided if in the bleaching process sufficient time were given in the first boil for the natural oil and cotton wax of the fibre to be completely saponified and removed. Scheurer has shown that with the best materials for the ley boil, goods must be boiled for six hours before the cotton oil can be saponified and the way cleared for the emulsion of any remaining waxy and unsaponifiable matter; but in the lace trade, at least, customers often insist upon the bleacher returning goods in that time (*viz.*, six hours), bleached and dried. The short boiling thus enforced only removes the oil on the surface, and the major part of the mineral oil is imprisoned by the cotton wax, &c. remaining, and forms an objectionable stain on the finished fabric; or, if at first apparently cleared, it subsequently oozes to the surface of the fibre and develops into a pale yellow stain.

The ley boil should be done under pressure with caustic soda, a little resin, and a small quantity of a good tallow soap. The speed at which the liquor circulates in the kier influences the emulsive power of the detergent liquor, a quick circulation being advantageous. A good tallow soap (being cheaper than olive-oil soap) should be used in preference to the mottled soaps made from bone grease, &c., which latter are so largely used by bleachers in consequence of their low price. It will be found that a sound tallow soap is much more effective where obstinate stains have to be dealt with.

The dolly, a machine used by lace and hosiery bleachers, is almost a necessity for removing the worst of these stains. Its action is to beat the goods while immersed in the detergent liquor. Those stains which survive this treatment can be taken singly and hammered on a bench with a small wooden mallet, dipping them occasionally into a strong soap solution.

In the case of removing stains from dyed and finished fabrics, which cannot be wet out again without spoiling, benzene, which acts as a solvent for mineral oils, can be used. The stain is impregnated with the solvent and then pressed between blotting paper or other absorbent material, thus removing the benzene with the oil in solution.

This solvent is not used on the large scale by the bleacher, as, apart from other objections to its general use, the expense is prohibitive.

Discussion.

Mr. H. Forth admired the author's pluck in tackling this subject. Talking of mineral oil stains on fabrics always frightened bleachers and dyers. Notwithstanding most careful treatment, these stains appeared months after the goods had gone away. Many attempts had been made to remove these stains, but usually without success. He agreed with the author that it was oil from the bearings or other parts of the machinery which caused the mischief. Fine white cashmere shirts with silk in them often showed stains after a very long time. Oil stains also appeared on the papers in which the goods were wrapped. The paper absorbed the oil, and the customer of course condemned the articles.

Mr. S. F. Burford had recently had some difficulty with regard to slight stains, which he attributed to mineral oil. He would be glad to hear of a test to ascertain whether they were mineral oil stains or not.

Mr. J. T. Wood asked whether benzoline could not be used. It was used now on a very large scale for extracting grease from leather. A machine which recovered the benzoline would involve little or no expense except for plant.

Dr. Sudborough thought that by no single process could these stains be removed. They were due to various substances. Mineral oils could probably be removed by extraction by benzine or benzoline. Those mineral oils which were used for lubricating were allied to benzoline. The stains due to small particles of brass or iron from the bearings could not of course be removed in the same way as mineral oils. Possibly they might be removed by some of the methods mentioned. At any rate, the stains due to metals ought to be removed before the fabric was bleached, as otherwise the material would be liable to rot in the process of bleaching. The stains due to mineral oils could be removed before or after—it was immaterial. From a common-sense point of view was not prevention better than cure? Were there no means at hand by which they could prevent the staining of the fabric? Could they not design a guard to screen some of the bearings from the fabric?

Dr. Clowes said that in the course of a long experience at the University College at Nottingham, the question of these stains had arisen on more than one occasion, and both the causes referred to by the author were present in the fabrics which were submitted to himself. There were brown stains in the finished fabric due to iron and due to mineral oil. In one instance they proved that the stain was due to iron by burning the raw fabric and estimating the quantity of iron in the ash, and then treating the finished fabric in the same way. The ash of the finished fabric contained a large quantity of iron, obviously picked up in the course of making and finishing. There was no process in the finishing which would remove the iron. The bleaching would tend to fix the iron rather than to remove it, and unless the iron were removed specially as suggested there seemed no chance of getting rid of it. In one case the source of certain stains alleged to be caused by mineral oil was ascertained, not by examining the stains themselves, but by examining the oil which had been used for preparing the yarn. Yarn was commonly greased with lard before it was passed through the rollers, which had iron surfaces. In the case of iron stains, the oil which had been used for preparing the yarn was found to be acid, and the iron was introduced by the action of the acid on the iron surface of these rollers. In another case mineral oil was undoubtedly present in the oil used for this purpose. He supposed that that was an acknowledged operation. It was known to be mixed with vegetable oils. They proved this by simple experiment. The fact of the presence of mineral oils explained why there was such difficulty in removing the stains. They would expect to remove them by washing with soap, but this was found to be impossible after the goods had been kept. They could remove the appearance

of the stains, but the goods were ultimately sent back again, the stains having reappeared. Mineral oil stains of some standing could not be removed by simple mechanical treatment, or short treatment with solvents. No doubt it was possible to get a thin film of the oil off, but not the bulk of the oil which had penetrated into the fabric. The goods stood by, and the oil from beneath gradually worked its way to the surface again. Probably the fabric would require very prolonged treatment to get rid of all the oil.

The CHAIRMAN agreed with Dr. Sudborough, that "prevention was better than cure"; but in order to say whether prevention was possible, one should know the character of the machinery. Perhaps Mr. Paton, as an engineer, would enlighten them.

Mr. PATON said hot bearings ought not to exist. There was always a reason for bearings getting hot, and it was preventable. While he had no special knowledge of these machines, yet on general principles he might say that the flying of oil could be prevented. Bearings were now made which required oil once in six months, and during the whole of that time there was not the slightest appearance of oil. The more troublesome of the two classes of stains appear to be from the bearings, and these were usually preventable. Now the subject has been brought forward, some engineering members of the Society might be called in to confer with the chemists in order that the difficulty might be remedied.

Mr. R. M. CAVEN said if, as seemed to be the case, the use of mineral oil almost necessarily involved stains, was it not courting failure to put mineral oil in that used for dressing fabrics? Should not vegetable oil be used, entirely to the exclusion of mineral oil?

Mr. PENTECOST, in reply to Mr. Forth, said that he had merely dealt with the difficulty as regarded cotton fabrics; and the treatment recommended for cotton would, of course, be too severe for silk and wool. With regard to Mr. Wood's remark as to whether benzoline could not be used on a large scale where the benzoline was recovered, there seemed to be no likelihood of bleachers complicating their plant any further. It would be too expensive, taking into account the price paid for bleaching. There was also the danger of fire, but this could be overcome. It would also be necessary to store the goods before treating them, if the process was to be effective. He questioned whether many of these mineral oil stains could be completely removed by an ordinary treatment with benzine without the preliminary removal of moisture. He confessed he could not satisfactorily explain why old oil stains were found to be tender. He had found some that were somewhat tender before the goods passed through the bleaching process. The metal dust mixed with oil from bearings might account for it. Dr. Sudborough asked whether these stains could not be prevented. They could, and easily. The best method of prevention was simply to use oils which were saponifiable. This could be done at an infinitesimally small extra cost; but the desire to get a cheap oil led people to adulterate with mineral oil, and the buyer did not know that the oils so adulterated caused such mischief. The bleacher did not generally find much difficulty in distinguishing between an iron stain and a mineral oil stain. The former was removed by treatment with strong HCl, and subsequent thorough rinsing in water and weak alkali to remove all trace of acid. The iron stain never reappeared after this treatment. Dr. Clowes had alluded to the oil which he examined. He probably referred to the same oil which was sent to him (the author) on a piece of lace two or three weeks afterwards. The piece, with one end stained in the oil, after passing through the usual bleaching process, showed a large yellowish stain distinctly visible. He at once set it down to mineral oil, and returned the piece to the dollies, and gave it a 10 hours' boil. The stain was completely removed. On making enquiry 9 or 10 months after this he found that there had been no reappearance of the mineral oil stain.

Dr. CLOWES explained that the goods to which he had referred might have stood with the oil in contact, so that the oil would be much more likely to penetrate. Possibly in the case mentioned by Mr. Pentecost the penetration was not allowed.

THE OLEOREFRACTOMETER.

BY S. F. BURFORD.

Mr. BURFORD described the oleorefractometer of Messrs. Amagat and Jean, which he had found especially valuable in analysing a large batch of samples—of butters, for example—and by its means he was able to select those "deserving of special attention." He would not condemn a sample on the strength of these indications, but as a means of sorting the good from the bad he considered the instrument invaluable. The instrument was described in an article by Dr. Muter in the *Analyst*, May 1890, pp. 87—89. M. Jean had discovered a typical oil that gave no refraction, and with which the outer receptacle was always filled. Mr. Burford had found that lard oil answered the same purpose.

ERRATUM.

This Journal, 1897, 310, col. 1. The sketch which should represent a suspended "vacuum-jacketed tube" has been accidentally placed in a wrong position. It should have been turned 90° to the right, so as to bring the twisted wire by which the tube is suspended into a vertical position.

Yorkshire Section.

Chairman: Thos. Fairley.
Vice-Chairman: Christopher Rawson.

Committee:

H. E. Aykroyd.	*W. Leach.*
J. Cohen.	*A. G. Perkin.*
J. R. Denison.	F. W. Richardson.
N. Farrant.	A. Smithells.
T. Glendinning.	Geo. Ward.
A. Hess.	*T. Whitaker.*

Hon. Local Secretary and Treasurer:
H. R. Procter, Yorkshire College, Leeds.

The names in italics are those of Members of Committee who retire at the end of the current session.
The following have been elected to fill the vacancies, and will take office in July next:—*Committee:* F. W. Branson, J. J. Hummel, W. McD. Mackey, and H. R. Procter.

SESSION 1896-97.

Meeting held on Monday, March 29th, 1897.

MR. T. FAIRLEY IN THE CHAIR.

A METHOD OF ESTIMATING THE WEIGHT OF SOLID MATTER IN THE AIR.

BY J. B. COHEN, PH.D.

IN connection with a series of experiments, which were undertaken with the view to estimating the impurities in the atmosphere of Leeds, I attempted to determine the weight of suspended matter in the air. I roughly appraised the quantity at not much above 1 mgrm. in 100 cb. ft., which would necessitate therefore a large volume of air and extreme accuracy in weighing. The first method, subsequently relinquished, consisted in aspirating the air by means of a small electric Blackman fan and intercepting the dust by a glass plate smeared with glycerin, which was then washed, the washings filtered and the solid matter weighed. The volume of air aspirated was measured by an anemometer. I mention the method in case anyone may feel disposed to improve upon it, but under the conditions I employed I found it unsatisfactory. The second plan was to filter a smaller and more carefully measured volume of air and to weigh the solid matter as before. I had the use of a very delicate balance for the purpose.

I employed, to begin with, an open zig-zag tube coated inside with a thin layer of vaseline, which permitted a rapid flow of air. In order to determine if vaseline lost weight in the course of aspirating large volumes of air over its surface or gained by absorption of moisture, the tube was heated to 100°, and air, filtered through cotton wool, aspirated rapidly by means of a vacuum pump for four hours. In another experiment the same process was conducted for five hours at the ordinary temperature and in a third experiment at a slower rate (24 cb. ft.) for six hours. The loss in the first case was 1 mgrm. and in the last two, 0·2 mgrm. There was therefore a slight loss at the ordinary temperature.

Four experiments were then made on unfiltered air with the following result:—

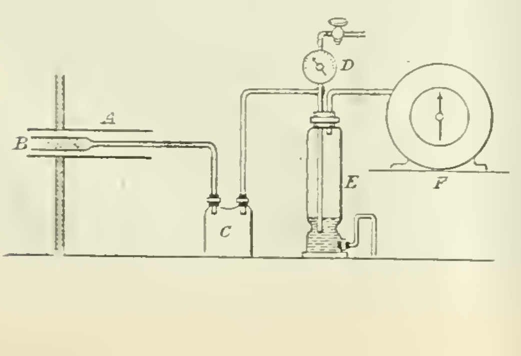
Number of Experiment.	Time.	Number of Cubic Feet Aspirated.	Gain in Weight.
	Hours.		Mgrms.
1	6	27	0
2	6	27	0·3
3	6	36	0·3
4	6	35	0·3

Adding on the 0·2 mgrm., which the vaseline loses in the same time, the average gain in weight would correspond to 1·36 mgrm. in 100 cb. ft. of air.

On examining the zig-zag tube with a lens, the quantity of dust in the bends gradually decreased, until the last bends appeared entirely free from specks, from which it might be concluded that the dust was completely removed in its passage through the tube. The method finally adopted as giving the most accurate results was the following:—

A cotton-wool plug was used as the filtering medium. In order to ascertain if the size of the plug to be employed was effective as a filter, it was fixed into a wide opening of a similar zig-zag vaseline tube to that previously used, and air aspirated through it for several hours. No specks appeared on the vaseline in any part of the tube. This plug was fixed into an open wide glass tube with a constricted tail piece for filtering, and transferred to a stoppered bottle for weighing. Some difficulty was experienced at first in obtaining the plugs of a constant weight. Cotton-wool is exceedingly hygroscopic, and after being dried at 100° will rapidly absorb moisture from the air as the temperature drops. I found on one or two occasions when the plugs were accidentally left in the steam oven a short time after steam was turned off; the temperature having dropped a few degrees, that they invariably weighed more, even though left in a desiccator over sulphuric acid during the night.

When the plugs were heated in the steam oven for the same number of hours, placed straightway in the desiccator over sulphuric acid, and weighed against a similar plug, heated and dried under precisely similar conditions, the plugs attained a constant weight. 17 weighings of the check bottle with its plug were made between March 8 and June 4, 1894, and the maximum variation was 0·4 mgrm., the variation from the mean being 0·2 mgrm. The difference between the check and experimental plug, which was adjusted by a few small weights, is obviously less. As a matter of experience whenever tested, the weights were practically identical. The form of apparatus is shown in the diagram.



A is a leaden tube fixed through the window. Through a cork diaphragm within this is fixed an open glass tube B. The wide portion contains the weighed cotton-wool plug. The glass tube B is connected with an empty bottle C, and a Desaga water-jet aspirator, which is fixed into a large wash-tower E, so that air can be aspirated through B and C, and delivered to the experimental gas meter F for measurement.

The experiments were carried on in the Philosophical Hall, near the centre of the town, and air was aspirated through an aperture in a window, about 15 ft. above the ground.

The air was aspirated for about six hours daily. During May and June at the rate of 25—35 cb. ft. a day. Sometimes the plug was weighed after one day, sometimes the experiment was continued two, three, or four days. Two sets of experiments were conducted alternately, giving the following results:—

Number of Experiment.	Cubic Feet Aspirated.	Gain in Weight.	Weight per 100 Cubic Feet.
		Mgrms.	
1	436·94	5·2	1·19
2	681·75	8·1	1·2

from which we may conclude that the weight of solid matter in Leeds air is nearly 1·2 mgrms. per 100 cb. ft. As this solid matter is perfectly black, we may assume that it consists for the most part of soot. Though so small in weight it represents for the whole bulk of Leeds air a quantity approaching 8 cwt. of solid suspended impurities in the atmosphere at every moment of the day.

Discussion.

The Chairman drew attention to the necessity for standardising anemometers, and especially in allowing for the nature of the opening, as similar experiments made with differently shaped apertures gave different results.

Mr. F. H. Richardson enquired whether any experiments had been made with pyroxylin as the absorbent for the dust, as he had used it for the estimation of micro-organisms with much success. He was of opinion that Dr. Carnelly's flasks might be used for these determinations, water or glycerine being employed as the absorbent media. This would be very useful in microscopic work at any rate.

Mr. H. D. Mackey understood that Dr. Cohen's determinations only referred to the *dry* dust in the atmosphere. He would like to know whether the moisture had been estimated also, since micro-organisms and other organic constituents all contain more or less water.

Dr. Cohen, in reply to Mr. Richardson, remarked that he had not been attempting the estimation of micro-organisms in these experiments—hence the small quantity of air that he had aspirated. As regards the weight of micro-organic impurities he considered it almost negligible, for considering the number of these particles in the air it was extraordinary how little increase of weight there was.

SOME METHODS OF ANALYSIS OF CHROME SALTS.

BY PROF. H. R. PROCTER, F.I.C.

The methods which I am about to describe lay no claim to originality in principle, but are simply the result of a search for convenient means of determining the principal constituents of the various chrome liquors now largely used in leather manufacture, and were originally intended for publication in a little book which I have in the press on the methods of analysis in use in the leather industries. Some of my friends, however, expressed the opinion that the information may also be useful to chemists engaged in dyeing and other kindred manufactures, who are not likely to see the book to which I have referred, and I have therefore ventured to bring it before the Society.

The determinations generally required for these purposes consist in that of chromic acid in its free state and in acid and normal salts, of the normal and basic salts of chromium

combined with other acids, and of chrome oxide as obtained in the ash of fabrics and leather after ignition, by which it is rendered extremely insoluble. In this last case the most practicable way of getting the chromium into solution is to convert it into chromic acid by fusion with a mixture of 3 or 4 parts of sodium carbonate and 1 part of sodium peroxide, for 10 or 15 minutes. If the ash is finely pulverised before fusion, this should be sufficient, but if any green residue is left on solution in water, the operation must be repeated. A nickel crucible may be used, but for occasional work the damage to a platinum crucible is not very serious. The alkaline melt must be boiled with water for at least half an hour before acidification to destroy excess of peroxide, as hydrogen peroxide reduces chromic acid, while oxidising it in part to perchromic acid. (*Cf.* this Journal, 1895, 1017; 1896, 155.) The chromic acid may then be estimated in the solution by titration with iodine and potassium iodide and thiosulphate as about to be described. For mere detection of chromium in an ash, fusion in a borax bead is a delicate test, a very intense green colour being produced even in the oxidising flame, while the green produced by ferrous iron is pale, and passes into a yellow-brown in the oxidising flame, becoming much paler as it cools.

The atomic weight of chromium is given by Ostwald at 52·15, oxygen being reckoned as 16, and it is therefore more accurate to take the round number 52 than the frequently adopted 52·5, and certainly sufficiently so for technical purposes; while it has the convenience that CrO_3 is then exactly 100, which considerably facilitates calculation. On this reckoning the molecular weight of potassium bichromate is 294. It must be remembered that chromic acid may be looked at from the double point of view of a dibasic acid, of which $\frac{1}{2}$ grm.-molecule will be required for 1 litre of normal solution; and of an oxidising agent, of which $1\frac{1}{2}$ atoms of oxygen are active, and which will therefore liberate 3 atoms of iodine, &c., and of which $\frac{1}{3}$ grm.-molecule only is required for a normal solution. Similarly, considering bichromate, we see that the molecule contains two chromic acid groups both "half bound," and therefore only saturates as much alkali as the one molecule of free chromic acid, while as an oxidising agent it contains 3 atoms of loosely combined oxygen, and liberates 6 atoms of iodine, and that therefore only $\frac{1}{6}$ grm.-molecule or 49 grm. is required for a normal solution. I must apologise for mentioning such elementary facts, but shall have to make use of them later, and have found that the two different normal strengths are sometimes confusing at first sight. Both acidimetric and oxidising methods may be used advantageously for analytical purposes.

Chromic acid may be estimated gravimetrically by precipitation as lead or barium chromate, but still more easily and satisfactorily by reduction (by alcohol or sulphurous acid) to chromic oxide (Cr_2O_3) and subsequent precipitation by ammonia.

Of the volumetric methods, the one generally recommended depends on the oxidation of a ferrous salt by the chromic acid, as in Penny's method of estimating iron. A convenient method, where only occasional determinations are required, is to weigh about 1 grm. of pure ammonio-ferrous sulphate from a weighing bottle into a flask or beaker, adding a little water, and, say, 10 c.c. of dilute sulphuric acid, and titrate it with the chromic solution of which the strength is to be determined until a drop withdrawn by a glass rod gives no greenish tinge with a freshly prepared solution of potassium ferricyanide on a white plate. Three molecules or 1,176 of the ferrous salt correspond to 1 molecule or 100 of CrO_3, from which the strength of the solution is easily calculated. The principal objection to this method is the character of the end-reaction, which is always more troublesome than one dependent on a change of colour in the body of the solution, and the necessity of weighing out the ferrous salt every time on account of the liability of ferrous solutions to oxidation by the air.

Where many analyses have to be made, I have found the iodometric method of Zulkowsky much more convenient and very accurate and rapid; and especially so with very dilute solutions such as are used in tanning and dyeing. Some of the text-books talk about distillation of the liberated iodine, and others convey the impression that digestion under pressure in a closed flask is necessary for the complete liberation of iodine from potassium iodide by chromic acid, but in my experience the reaction is almost instantaneous at ordinary temperatures, and at least is quite complete in the cold in a few minutes in a stoppered bottle.

The method is conveniently carried out as follows:—A measured quantity of the chromate or chromic acid solution, which should not contain more than about 0·01 grm. of potassium bichromate or its equivalent, is placed in a stoppered bottle of 200 or 300 c.c. capacity, and 10 c.c. of a 10 per cent. solution of potassium iodide and 5 c.c. of concentrated (or a corresponding quantity of dilute) hydrochloric acid is added. The bottle is closed and well shaken, and allowed to stand at least a few minutes, when it is titrated with a N/10 solution of pure sodium thiosulphate (24·8 grms. per litre) until the brown coloration of the liberated iodine has nearly disappeared, when about 1 c.c. of $\frac{1}{2}$ per cent. starch solution is added and the addition of the thiosulphate continued till the violet of the starch iodide has given place to the pale greenish-blue of the chromic chloride. With very slight practice this point is perfectly easy to hit. As the violet tinge sometimes reappears by some secondary oxidation, its first disappearance should be noted as the end-point. As has already been stated, each molecule of chromic acid liberates three of iodine, and consequently each c.c. of N/10 thiosulphate is equivalent to 0·0049 grm. of bichromate, or 0·0033 of chromic anhydride. If pure thiosulphate solution is not at hand, an approximately normal solution is made by dissolving 25 grms. of ordinary "hyposulphite of soda" in a litre of water, and titrating 20 c.c. of N/10 bichromate solution, as above described with iodide of potassium. If the same quantity of the two chrome solutions be used, their strengths will be directly proportionate to the thiosulphate used in each case. A similar method may be used to estimate the thiosulphate in "hypo liquors" by adding the liquor to be tested from the burette to the iodine solution obtained by treating 10 c.c. of 10 per cent. potassium iodide solution, acidified with hydrochloric acid, with 20 c.c. of N/10 bichromate, which will correspond to 0·496 grm. of pure thiosulphate. No accurate results can be obtained by titrating thiosulphate direct with bichromate, as the oxidation is complex, and proceeds further than that with iodine. The iodide used must be free from iodate, and the hydrochloric acid free from chlorine, both of which may be tested by a blank experiment, when, if either are present, iodine will be liberated, colouring the solution brown, and giving the usual reaction with starch. If pure chemicals cannot be obtained, the amount of iodine so liberated must be determined and allowed for.

It is frequently desirable not only to determine the total quantity of chromic acid present, but the relative amounts as free acid and bichromate, or as bichromate and neutral chromate. It is obvious that all three of these cannot exist in the same solution, but it must not be forgotten that free chromic acid may co-exist with other free acids, and that by any alkalimetrical process they will be estimated together. Neutral chromate reacts neutral with phenolphthalein, while bichromate and free chromic acid are acid to it. If, therefore, bichromate and neutral chromate are present, the former can be estimated by adding standard sodium or potassium hydrate solution in presence of phenolphthalein till the latter is reddened. Every c.c. of N/10 alkali will in this case correspond to 0·0147 grm. of potassium bichromate, 0·01 of half-bound chromic acid (present as bichromate), or 0·005 grm. of free chromic anhydride, the question being one of acidity simply, and not of oxidising power; and hence free chromic acid will consume double as much soda as when "half-bound," in bichromate.

If a solution contains 0·1 grm. of CrO_3 in whatever form, it will require 30 c.c. of N/10 thiosulphate to reduce the iodine it evolves from potassium iodide. If the chromic acid is free, it will also require 20 c.c. of N/10 sodic hydrate to make it neutral to phenolphthalein; while if it is present as bichromate, only 10 c.c. will be required, and in the case of neutral chromate none at all. We may therefore deduce the following rule:—

If the N/10 soda required to neutralise the chromic acid is less than one-third the N/10 thiosulphate, each c.c. of soda corresponds to 0·01 of CrO_3 as bichromate or to 0·0147 of potassium bichromate, and the remainder of the chromic acid indicated by the thiosulphate will be present as neutral chromate. If the soda required is more than one-third the thiosulphate, each c.c. in excess of one-third corresponds to 0·01 of free CrO_3, and the remainder indicated by the thiosulphate is bichromate. If the soda exceeds two-thirds of the thiosulphate the whole of the chromic acid is free, and the excess of soda over two-thirds corresponds to some other acid. The pale yellow colour of the neutral chromate scarcely lessens the distinctness of the end reaction with phenolphthalein.

It is also possible to estimate the free chromic acid directly by the methods described by Mr. Carlton Heal and the author in a paper on the "Analysis of Used Chrome Liquors," which was published in this Journal, 1895, 248; and probably for very exact work this would be preferable to the indirect method just described.

The determination of chromium in solutions of chrome salts is, of course, readily performed gravimetrically by precipitation with ammonia, but its rapid volumetric estimation is much less easy. Naturally if it can be converted into chromic acid, its estimation by the methods already given will present no further difficulty. The problem is to find an oxidising agent which is powerful enough to convert the whole of the chromium into chromic acid, but which can itself be completely removed from the solution before titrating, without at the same time reducing any of the chromic acid formed, or which is incapable of liberating iodine or oxidising thiosulphate. Hydrogen peroxide naturally suggests itself, but though it is capable of oxidising chromic to perchromic acid, it also acts on it as a reducing agent with evolution of oxygen. Sodium peroxide in alkaline solution oxidises chrome salts rapidly to chromic acid, but must be completely destroyed by boiling before acidification, which demands half an hour, and with all possible care the results seem apt to be lower than the truth. Nitric and chloric acids are unsuitable from the difficulty of getting rid of them and their products after the oxidation is finished. On the whole potassium permanganate seems to be the most suitable oxidising agent. Crookes, in his "Select Methods," quotes a process suggested by W. J. Sell for the analysis of chrome steel and similar compounds, in which he oxidises with permanganate in a solution rendered acid with sulphuric acid, and after neutralising and filtering off the manganic oxide, estimates the chromic acid iodometrically. However well this may answer the purpose for which it was originally intended, it is rendered inapplicable in the present case by the fact that in tanning, and I suppose in dyeing solutions, chlorides are frequently present, and in acid solution in contact with manganic oxide, chlorine is evolved, which it is almost impossible to get rid of, and which, on the one hand, is apt to make the results too high by liberating iodine, while on the other, free hydrochloric acid in presence of chromic acid leads to loss by partial reduction of the chromic acid, and by volatilisation as chlorochromic acid. Crookes describes another method in which the salts of chromium are oxidised by standard permanganate in alkaline solution, and their amount estimated by the volume consumed, a point being taken as the end reaction when the supernatant liquid was pure yellow, free from any trace of green or pink. I have not found this nearly sufficiently delicate, at least with the dilute solutions with which I have had to deal, but the following modification has been found to give satisfactory results, and to be very easy to work. A measured quantity of the chrome solution, which should not contain more than about 0·05 grm. of Cr_2O_3, is rendered decidedly alkaline with sodium hydrate, raised to a boil, and potassium permanganate added till the supernatant liquor remains pink after boiling for two or three minutes. Alcohol is then added drop by drop to the boiling liquid till the pink disappears, and the solution, after cooling, is made up to a known volume, say, 250 c.c., and filtered. The chromic acid is then determined with potassium iodide and thiosulphate in, say, 50 c.c., as has been already described. The alcohol does not reduce the alkaline chromate solution, at least under the conditions described.

Beside the estimation of chromium it is often desirable to determine the basicity or otherwise of the liquors. I cannot suggest any direct means of doing this, but in absence of ammonia, and of bases precipitated by sodium carbonate, the total acidity in combination with chrome is easily found by a modification of Hehner's method of determining the permanent hardness of waters. A known excess of sodium carbonate is added and the solution boiled for a short time, made up to 100 c.c., filtered, and the sodium carbonate titrated back in an aliquot part. I have not found any necessity for evaporation to dryness. The same method may be applied under the same conditions to alumina salts, and in the writer's hands has given much more satisfactory results than Bayer's method of direct titration in presence of methyl orange, or its modifications. In the case of sulphate of alumina 5 grm. of the sample is dissolved in about 40 c.c. of water, 50 c.c. of N/1 sodium carbonate is added and the mixture is boiled for a short time, made up to 100 c.c., filtered, and 20 c.c. representing 1 grm. of the sample is titrated with N/1 acid and methyl orange.

Discussion.

The Chairman reviewing the methods suggested by Prof. Procter for the estimation of chromium considered that any iodometric method was likely to be superior to those in which ferrous sulphate in any form was used. He thought that after fusing a chromium compound with sodium peroxide, the excess of the latter might be more quickly removed by boiling after addition of sodium bicarbonate since the peroxide is unstable in presence of carbon dioxide. He drew attention to the fact that in reducing a chromate to chromic oxide by means of alcohol, especially if the alcohol is now quite pure, too much of the latter must not be used as some non-volatile organic compound seems to be formed which is capable of preventing the complete precipitation of the chromium. The ammonia should be added only in slight excess.

Mr. C. Rawson said he had used caustic soda with phenolphthalein for the estimation of bichromate but he found that better results are obtained by adding the alkali in excess and then titrating back again, than by direct titration. He had also used lacmoid paper to titrate chromate (alkaline to lacmoid) in presence of bichromate (neutral to lacmoid), acid being run into the solution until a drop of the latter placed on a piece of lacmoid paper causes no change of colour.

Prof. Procter, in reply to Mr. Ward, said that sodium peroxide does not seem to be prepared perfectly pure but generally contains some iron which, however, does not interfere in any way with the estimation of chromium.

Mr. Fairley incidentally mentioned a mode of indicating standard solutions free from doubt or ambiguity. The quantity of substance taken is made the numerator of a fraction, and the volume in which it is finally dissolved the denominator. Thus sulphuric acid solutions may be written:—

Normal sulphuric acid, or $\frac{N}{1}$ H_2SO_4, or $\frac{49 \text{ grm.}}{1,000 \text{ c.c.}}$ H_2SO_4;

Decinormal sulphuric acid, or $\frac{N}{10}$ H_2SO_4, or $\frac{4 \cdot 9 \text{ grm.}}{1,000 \text{ c.c.}}$ H_2SO_4;

Centinormal sulphuric acid, or $\frac{N}{100}$ H_2SO_4, or $\frac{0 \cdot 49 \text{ grm.}}{1,000 \text{ c.c.}}$ H_2SO_4.

&c. &c. &c.

A discussion followed on this proposal.

New York Section.

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The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Vice-Chairman:* T. J. Parker. *Committee:* H. Clementson, W. D. Horne, G. A. Prochazka, Clifford Richardson, and G. W. Thompson.

Meeting held on Friday, April 23rd, 1897.

PROF. CHAS. F. CHANDLER IN THE CHAIR.

Before the business of the evening commenced, Mr. R. T. Antony exhibited Chassagne's New Colour Photographs, and Mr. C. K. Munns exhibited a new Petroleum Welsbach Lamp.

THE DETERMINATION OF THE SPECIFIC GRAVITY OF MOLASSES.

BY J. C. BOOT.

Molasses containing about 40 or 50 per cent. sugar, which is the residue of the sugar works and refineries, has a specific gravity of about 1·400. The determination of the specific gravity of this molasses is very frequently necessary in the sugar laboratories, whilst the viscosity of, and the amount of air contained in, the molasses, makes it impossible to take the correct specific gravity with an hydrometer.

Sidersky (Zeitschrift für Rübenzucker Industrie, 1881, page 192) mentions a method which gives good results. According to this method, the molasses is heated in a funnel about 5 ins. diameter, placed in a hot-water funnel. The tube of the funnel is closed by a glass rod covered with a piece of rubber tubing. The molasses gets thin by the heating, and the air bubbles rises to the surface, forming a scum preserving the molasses from evaporation. When all the air bubbles have reached the surface, the funnel is placed on a support, the glass rod is lifted out of the funnel tube and, after allowing the first part of the molasses to run off, it is caught in a 50 c.c. flask, which has been previously dried and weighed. When the molasses has nearly reached the mark on the flask, the glass rod is lowered into the funnel tube, thus stopping the flow of the molasses. The flask and contents are now cooled to 15° C. and weighed; water at 15° C. is then added to the mark, and the weight is again taken. The water does not mix with the molasses, and it is therefore an easy matter to add water to the mark very accurately.

The specific gravity is then calculated as follows:—

	Grms.
Flask, with molasses	85·346
Flask, empty	15·834
Molasses	69·512

After adding the distilled water at 15° C:—

	Grms.
Flask, with molasses and water	86·280
Flask, with molasses	85·346
Water added	0·934

The 50 c.c. flask contained, therefore, 50 c.c. $-$ 0·934 c.c. = 49·066 c.c. molasses.

The molasses weighed 69·512 grms., and the specific gravity, consequently, is $\frac{69\cdot512}{49\cdot066} = 1\cdot416$.

I have devised a modification of this method which does away with one weighing and makes the calculation very easy. The molasses is heated in the hot-water funnel just as in the method of Sidersky. The molasses is run into the 50 c.c. flask up to about 1 c.c. from the mark. After cooling down to 15° C., a potassium carbonate solution of 1·400 sp. gr. (38·45 per cent. K_2CO_3) is added to the mark. The flask with the molasses and the carbonate solution is now weighed and the specific gravity calculated as follows:—

	Grms.
Flask, with molasses and potassium carbonate	86·618
Flask, empty	15·834
Molasses and potassium carbonate	70·784

$$\text{Specific gravity} = \frac{70\cdot784}{50} = 1\cdot4157.$$

In this example we took a molasses of 1·416 sp. gr. and filled the flask up to 1 c.c. from the mark; we have therefore—

$$49 \times 1\cdot416 + 1\cdot400 = 70\cdot784.$$

The difference is then only 0·0003. When the molasses has a specific gravity of 1·350 or 1·450, the flask being filled up to 1 c.c. from the mark, and potassium carbonate solution of 1·400 sp. gr. is used, the difference is 0·001, which can be considered of no importance.

The potassium carbonate solution should always be made of about the same specific gravity as that of the molasses to be tested. The following table gives the percentage of potassium carbonate present in solutions of different specific gravities at 15° C.

	Specific Gravity.
34·20 per cent. potassium carbonate	1·350
35·05 " " "	1·360
35·90 " " "	1·370
36·75 " " "	1·380
37·60 " " "	1·390
38·45 " " "	1·400
39·30 " " "	1·410
40·15 " " "	1·420
41·00 " " "	1·430
41·85 " " "	1·440
42·70 " " "	1·450

THE DEFINITION OF A LAKE.

BY MAXIMILIAN TOCH.

A COMPOSITE definition, culled from the older and newer chemical and literary authorities, tends to indicate that a lake pigment is an organic dye precipitated on an inorganic base. The word lake implies a colour used for glazing purposes and, formerly, lakes were used to that end almost entirely. Time and fashion have little regard for custom, and what was considered a lake pigment 25 years ago is, in many instances, not manufactured at all to-day.

The United States Customs Tariff of 1894 says that lakes should be dutiable at 25 per cent., and a vermilion containing lead should be dutiable at 6 cents per lb. It further says, that all blues containing ferrocyanide of iron shall be dutiable at 6 cents per lb., and all chromium colours and all blues containing ultramarine at 3 cents per lb. The fact that a lake is not definitely described in the tariff may lead to some confusion, but it can easily be demonstrated that there are a variety of pigments which are of such composition that they may either be lakes, or paints, or both. If the tariff be strictly interpreted, all the eosine colours of the scarlet order would be vermilions containing lead, because they are invariably eosines precipitated by nitrate or acetate of lead on a base of lead oxide, clay, alumina, barium sulphate, starch, or any suitable medium. If a solution of eosine be taken and precipitated by nitrate or acetate of lead a colour is obtained, which is an article of commerce sold under various names and may be regarded as a true lake. It has excellent covering property, is fairly permanent, and can be made in any shade from a pale vermilion to a deep ruby, according to the methods of manipulation and the grade of eosine used. Calculating the amount of lead contained in a sample of this kind, the percentage would be 42 per cent. oxide of lead and 58 per cent. bromofluoresic acid, the change occurring because lead replaces the sodium originally contained in the yellowish shade of eosine. This is unquestionably the highest type of lake that can be made, and in going down the scale, if the same ingredients be used and reduced with 10 per cent. of alumina and 25 per cent. of lead oxide (orange mineral), a colour is obtained, which in one branch of the trade sells as scarlet lake, and in another branch of the trade is used and sold under the name of coach grinders' vermilion. That this is a lake there is no doubt, and that it is a vermilion there is no doubt either. The question naturally arises over an article of this kind, it is dutiable at 25 per cent. as a lake, or is it dutiable at 6 cents per lb. as a vermilion containing lead? There are hundreds of red lakes which look like vermilion, and in some industries are used as vermilions, and in other industries used as lakes, and nearly all of them contain lead as a part of the base. If they contain originally eosine, they contain subsequently lead, because lead is used as the precipitant.

A blue made by precipitating potassium or sodium, ferrocyanide with a ferric salt is known in commerce as Prussian blue, and if part of this blue be precipitated in conjunction with alumina hydrate, a colour is obtained which in some industries sells under the name of blue lake, and in other industries sells under the name of Antwerp blue. As Antwerp blue, this colour is dutiable at 6 cents per lb. because it contains ferrocyanide of iron; as blue lake, this colour is dutiable at 25 per cent. In a like manner are chrome greens precipitated with alumina hydrate for producing certain lithographic ink colours and known commercially as permanent green lake, or such other names as the makers fancy or the consumers whim would dictate.

From the examples quoted and the inferences made, I contend that it is not within the province of the general chemist to decide whether a pigment under examination be a lake or not. Time was when the analytical chemist's opinion was taken conclusively as to the potability of a given water. Matters have changed and now it is the chemist who has the knowledge of bacteriology who decides as to the noxious or innoxious quality of the water. All the published definitions of a lake are so vague and general that they admit of too many exceptions and include a vast number of colours which should be taken or classed separately.

Discussion.

Mr. Ansbacher considered a lake to be a pigment consisting of an organic colouring matter or dye chemically combined with some material, usually aluminium or some derivative combination, which may be either pure or else containing a certain quantity of white pigment, which is added either to dilute the intensity of colour of the organic compound or else to give it the proper working quality.

Many manufacturers called the products that they sent out lakes when they were not really lakes at all; and a colour containing orange mineral could not justly be classified as a lake, because a lake was assumed to be a colour containing but little body with some transparency, which in the case of orange mineral was added only to give it body. A lake was a pigment which was not supposed to have much body, and which was used in connection with other pigments which possessed body to give it intensity of colour.

Mr. J. A. Ullmann took exception to the definition just given because of what was known in commerce, and sometimes called "white lake," *viz.*, hydrate of alumina, which was purely inorganic in character; but it was called white lake because it had the qualities of transparency, which are generally desirable in lakes. The name of lake should be given to every pigment which had some degree of transparency, or else should be confined entirely to the old lakes as they were formerly made.

Mr. Ansbacher had never heard the term "white lake" used, and he had been in the trade for five years.

Dr. Alsberg entirely disagreed with Mr. Ullmann. In the first place, a lake was certainly nothing but a colouring matter, chiefly organic, precipitated by some means on a transparent or semi-transparent substratum, as ordinary aluminium hydrate, to which may have been added starch, simply as an adulterant. He had never heard of a lake such as Mr. Toch specified, which he took to be a slip of the tongue, containing lead oxide. He presumed that he meant sesqui-oxide, red lead, or, as it was known commercially, "orange mineral."

The author referred to the 1894 tariff. That tariff contained two paragraphs, one of which referred to vermilion red containing quicksilver, which was dutiable at 20 per cent. *ad valorem*, and another paragraph referring to vermilion reds not containing quicksilver, but containing or made with lead, which paragraph was probably introduced into that tariff simply for the reason that some time previous to its passage some so-called imitations of vermilion had been made. These imitations were always precipitated on the sesqui-oxide of lead with red lead and the orange mineral of commerce, although some might contain white lead, oxide of zinc, and so on, partly as adulterants and partly as a means of altering the shade. If they contained whites, the resulting colour was not quite so yellowish. It was more of a bluish tone, and the reason they were separated from the vermilion proper was that there was no means of determining their value. For instance, orange mineral, which at that time paid a duty equivalent to about 60 per cent. *ad valorem*, was coloured with a mere trace of eosine so as to avoid paying the duty, which was $3\frac{1}{2}$ cents per lb. People in Europe simply took 100 lb. of orange mineral or red lead, and added a few ozs. of eosine in solution, which they sent to the United States market, and which paid 25 per cent. *ad valorem* instead of 60 per cent. He also dissented from the author's statement about ferrocyanides being lakes. The author stated that a well-known firm, Messrs. Windsor, Newton, and Co., of London, described "Antwerp blue" as a lake. He (Dr. Alsberg) had known the article a good many years and had manufactured many thousand pounds of it. It was nothing but a mixture of ferrocyanide of iron with some aluminium hydrate for certain purposes; but nobody ever thought of classifying that colour as a lake. Nor had he ever heard that certain chromium colours which Mr. Toch did not specify, and which could be only either the oxide or the sesqui-oxide, the article known in commerce as the so-called hydrate. Sesqui-oxide, manufactured according to Binlay's process by the fusion of potassium bichromate with picric acid, and subsequent washing, was the pure sesqui-oxide, sometimes toned up either with a trace of ultramarine blue or ferrocyanide of iron, so as to give it a little more bluish tone instead of the yellowish tint which it had originally. The other chromium colour must be a mixture of lead chromate and ferrocyanide of iron only. Those certainly were not lakes. They lacked every property of a lake. A lake had never been known to mean anything but a colour precipitated on a transparent or translucent substratum.

Mr. Toch, in reply, said that if he mentioned the orange mineral as a lead oxide, he did it on the authority of a very learned man, who said orange mineral has not yet been classified as a complete compound, and should not be anything else, because of various uncertain conditions. He denied that a lake must be precipitated on a substratum more or less translucent, since pure eosine precipitated by nitrate of lead was undoubtedly a lake, and yet it was not precipitated on anything. It was simply a precipitate, and was a compound which was absolutely new. Dr. John W. Draper, in a book which he published in 1842, defined a lake as alumina which carried down a colouring matter similar to that of cochineal or the extract of some of the West Indian woods or some of the Oriental woods. That was so until the newer changes came up. A lake was entirely different at the present day to what it was 25 or 30 years ago. The tariff of 1894 said vermilion red containing lead should pay a duty of 6 cents a lb. Vermilion red containing lead might also be a lake, because if an absolutely true lake might contain 42 per cent. of lead, calculated as oxide, why could not a colour be a lake which only contained 10 per cent. of orange mineral, another form of lead oxide? He brought the matter up to show the absurdity of that very paragraph of the tariff of 1894. He had asked the Board of Appraisers whether they would determine what a lake was. They said they would prefer to leave that for other people. They would simply say whether a certain colour was a lake or not; but a colour might be imported from Germany, for instance, which was worth about 8 marks a lb. To the uninitiated eye it looked exactly like vermilion, but to the Board of Appraisers and their chemists that colour might pass as vermilion red containing lead, at 6 cents per lb. Those things had happened, and it was just because the Government made incorrect discriminations that paragraphs like the above should be excluded. Messrs. Winsor, Newton, and Co. did not claim their Antwerp blue to be a lake. They claimed it to be a ferrocyanide of iron precipitated on alumina, and he merely mentioned it because, according to the old definition, this was a lake because it was precipitated on an inorganic base.

Mr. Ansbacher said that one could not add orange mineral, which was an oxide of lead, to a lake without affecting the colour to such an extent that anyone who

had experience could at once determine that that was a vermilion. Whenever that lake contained orange mineral it no longer was a lake, and should be classified accordingly.

Mr. Ullmann said that in former times certain pigments were known as lakes, and their distinctive character was their transparency; and, therefore, when eosine and similar so-called lakes were introduced, it was on account of their similarity to the lakes that they were brought into commerce under that name. It therefore seemed to him that the name "lake" could justly be given to such colours only that resembled the old lakes, and, as Mr. Ansbacher said, this particular quality was greatly diminished so soon as any such pigment as orange mineral was added. The name "lake" should be given exclusively either to those old-time lakes as they were formerly prepared, or, if it were to be extended, to all colours that possessed these properties. Accordingly it would also apply to those translucent substances that were sometimes known as white lakes, which had no colour at all. If they had to choose between confining the name "lake" to its original use or else extending it considerably beyond, he agreed with Mr. Ansbacher that if anything were added to a lake which modified its distinct qualities it then ceased to be a lake in the proper sense.

SOLUBILITY OF LIME IN PRESENCE OF SODIUM AND POTASSIUM CHLORIDES AT VARIOUS TEMPERATURES.

BY GODFREY L. CABOT.

The following research, which I carried out as a student in Zurich 13 years ago, and which has never been published, was undertaken to ascertain the effect of temperature and of sodium and potassium chlorides in solution upon the solubility of lime.

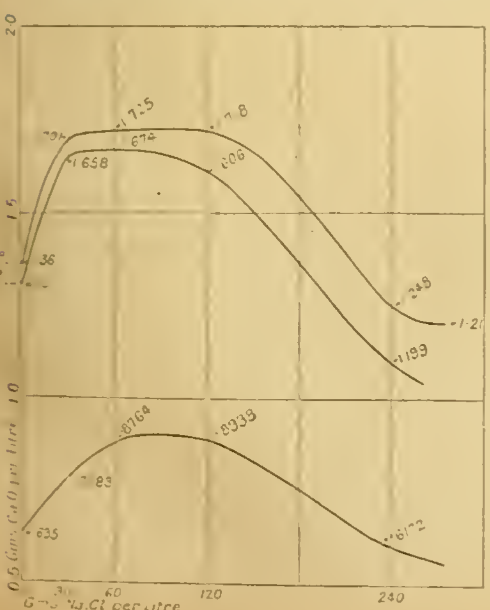
It will be perceived that the solubility of lime in saline solutions diminishes with the increase of temperature; secondly, that the addition of chloride of potassium or sodium increases the solubility of lime till over 6 per cent. of the chloride is in solution, at which point the maximum solubility is passed, and the solubility diminishes to a minimum where the aqueous solution is saturated.

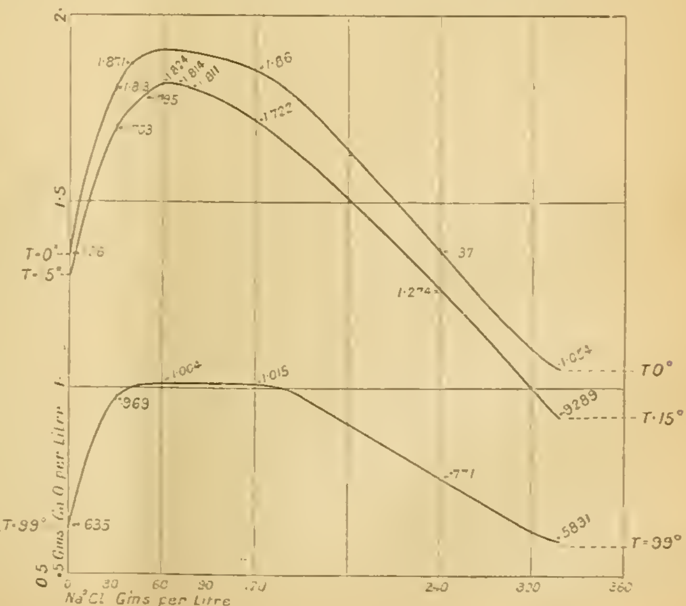
At the boiling point (99° centigrade at Zurich) the saline NaCl solutions containing between 60 and 120 grms. of common salt per litre dissolved a little over $\frac{1}{10}$ of 1 per cent. of lime. The relative increase of solubility with increase of salt seems a little larger at the boiling point than at the zero point. At ordinary temperatures the solubility is less than at zero point and much more than at the boiling point. It further appears that after 60 grms. of salt per litre is exceeded, the solubility of lime in water diminishes, but not so rapidly in proportion to the change of the salinity as it previously increased. In a saturated aqueous solution of salt the solubility of lime is about 25 per cent. less than in pure water at ordinary temperatures. At 99° centigrade it is about 8 or 9 per cent. less than in pure water. In pure water the solubility of lime diminishes with rising temperature less rapidly between 0° and 15° than between 15° and 99°.

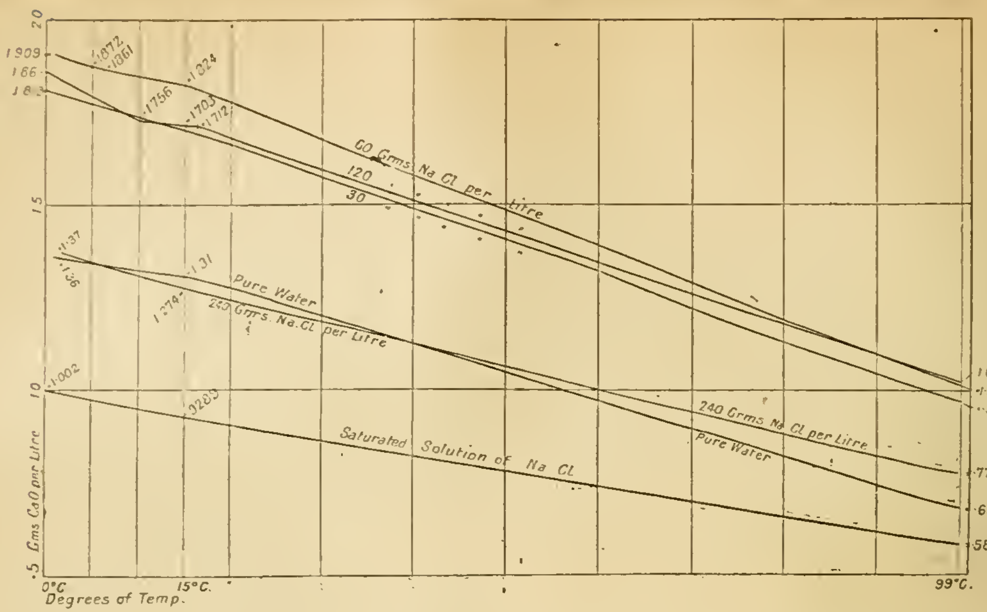
In saturated solutions of either KCl or NaCl it diminishes with rising temperature more rapidly between 0° and 15°, and less rapidly between 15° and 99°. The solubility of NaCl increases so little with an increase of temperature as not to materially affect the result of the experiments in NaCl solutions saturated at different temperatures. I have even seen it stated that it was not at all more soluble at the boiling point than at zero point. I beg, however, to corroborate the opinion of the majority, backed by quantitative experiments, that it does increase in solubility with the increase in temperature. If a saturated solution of common salt at 100° be cooled, out of contact with the air, it will deposit salt. In the case of chloride of potassium the solubility of the lime is uniformly less than in corresponding solutions of common salt. The maximum solubility for zero and ordinary temperatures seems to occur in a little stronger solution than was the case with common salt; just how much I did not ascertain, but the maximum will not considerably exceed the solubility attained with a solution containing 60 parts of KCl per litre. For saturated solutions the problem is made harder by the increased solubility of chloride of potassium as the temperature increases, and we find that the curve of solubility continues out in a straight line downward as far as the increasing solubility of KCl takes it.

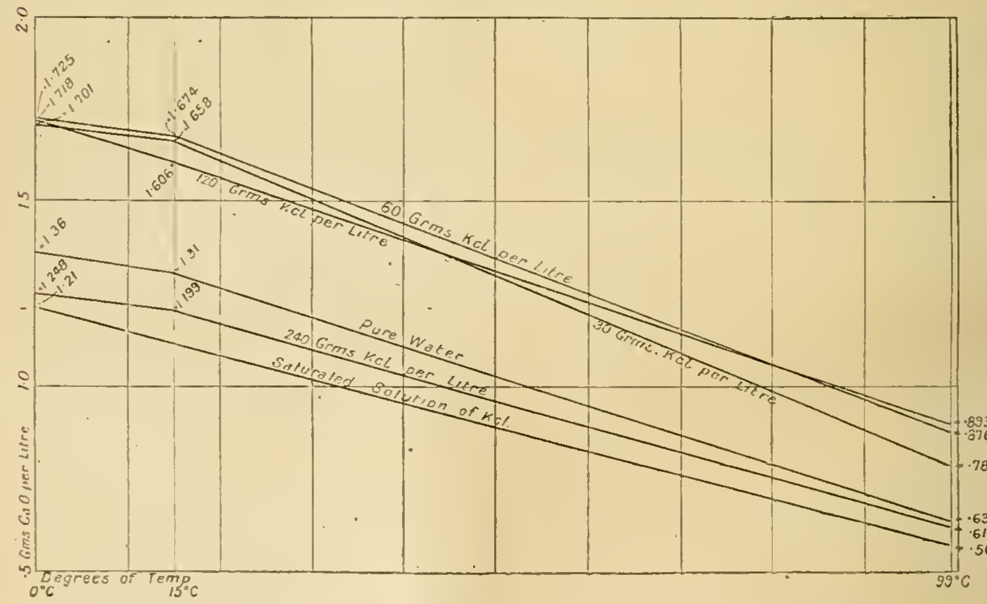
The general relations mentioned in connection with solubilities in salt solutions obtain also in the KCl solution, but there is a peculiarity which I did not mention in connection with the salt solution because it is quite obscure, but which becomes very obvious and apparent in the case of chloride of potassium, namely, that at the boiling point the solubility of lime in either salt or chloride of potassium solutions increases to a much stronger solution of the salt than at ordinary temperatures or the freezing point.

Apparently the maximum solubility of the lime at the boiling point is in both cases where the amount of the chloride in solution is about 120 grms.; but the curve of solubility is so flat on top that this maximum cannot be located very accurately. It is apparently later in the case of chloride of potassium than in the case of chloride of









sodium. It takes a larger proportion of chloride of potassium to dissolve the maximum possible amount of lime than it does of chloride of sodium.

Whether these proportions coincide with the relative molecular weights of chloride of sodium would be difficult to determine, owing to the experimental error in dealing with such minute deviations. On the same co-ordinate paper I have prepared a series of curves to show the effect of temperature on the solubility of lime in different solutions of the chloride. In other words, the temperature varies with the abscissa, and the strength of the solution with the ordinate. This brings out a noticeable fact: that the solution of common salt having 240 grms. per litre dissolves at the zero point a trifle more lime than pure water, and at the boiling point over 20 per cent. more, and the curves of solubility representing 60 grms. and 30 grms. lie pretty close together throughout their length. In the case of chloride of potassium these three lines start, to be sure, at about the same points—in other words, at zero temperature. The solubility of the lime varies little between 30 grms. of chloride of potassium and 120 grms. of chloride of potassium in aqueous solution; but at the boiling point there is a difference of some 12 per cent. or more. The solubility in solutions of chloride of potassium of zero grms. and 240 grms., and saturated at zero centigrade, lie close together, and converge as they approach the boiling point.

To sum up briefly the results of the investigation, we see that the solubility of lime in solutions of either chloride of potassium or of sodium increases to a maximum at about one-fifth of saturation, and then diminishes to a minimum at the saturation point, where it is lower than in pure water. The chloride of sodium solutions dissolve more lime at all temperatures and degrees of concentration than equal solutions of chloride of potassium. That in each case the maximum solubility of lime in the chloride solution is at the lowest temperature, and at about 60 to 120 grms. of the chloride per litre, and that the minimum solubility occurs at the saturation point and at the highest tempera-

ture. That an increase of temperature at all times and in any of these solutions diminishes the solubility of the lime, and at any given temperature the minimum solubility is found in the saturated solution.

I had no very definite practical object in my experiments; they were simply purely scientific research, intended to throw light on the general relations of substances in solution.

I attribute the additional solubility of lime in saline solution to a partial reaction, whereby chloride of calcium and hydrate of sodium are formed in juxtaposition. Such reactions I consider of great importance to the theory of chemistry, and I hope they will be more thoroughly studied in the future. They are not dwelt upon in books, but every practical or theoretical chemist realises their great importance and significance; and a more exact record of these partial reactions would be of great assistance in technology, and save many useless attempts to accomplish the impossible.

Suppose that my research had shown, in place of this wave-like curve, a hyperbola with its cusp at the left; in other words, supposing that the solubility of lime in salt had increased in an increasing ratio with the strength of the saline solution? This might have led us to infer that with chloride of potassium, under pressure, it might have been even possible to avail ourselves of the reaction, and manufacture hydrate of potassium on a commercial scale. The actual results have absolutely closed this particular door, and by so much limited the field for future technical investigators.

THE DETERMINATION OF TANNIN BY MEANS OF HIDE POWDER.

BY JOHN H. YOCUM.

There are two general methods in use for making tannin determinations—the hide powder and the Lowenthal.

The latter method is oxidation in acid solution of all the organic material obtained in an extraction of raw tanning material by means of permanganate of potash, then removing the tannins by means of a gelatine solution and oxidising the remaining organic matter under the same conditions.

In the hide powder method the solids in a tannin solution are determined by evaporating to dryness and weighing, the tannins are removed from a separate portion of the solution by means of hide, and the remaining non-tanning substances determined by evaporation and weighing, the difference being the material removed by hide or tannin.

In the technical examination of tanning material, the method that gives results that are approximately true measures of the tanning value must be accepted, *i.e.*, determines those organic bodies that go with the hide to make leather irrespective of their chemical composition. During the operation of making leather there is opportunity for both chemical and physical reactions. There occurs a chemical reaction between the hide and the tannin, and a physical reaction of the nature of a dyeing operation.

A true chemical union probably occurs between the hide fibre or the inter-cellular gelatine of the hide and the tannins, but this is only a part of the tanning operation, as tannins, colouring matter, &c., dye the fibre, and are held in solid solution, hence a purely chemical method will not determine those organic bodies that go with the hide to make leather, and an empirical method becomes a necessity.

Part of every soluble compound in a tanning solution is taken up by the hide, but it is only those that have the special property of "tanning" that are completely removed when the hide is in excess, the amount of non-tanning materials that are taken up depending upon the condition of the hide, concentration of solution, &c., and is relatively small in any case.

The Lowenthal method does not regard these conditions, but determines the amount of material that will precipitate gelatine, and, aside from the difficulties of operation, particularly the duplication of conditions of oxidation each time, we do not know the amount of oxygen necessary in any individual case for the oxidation of the particular tannin we are working with, and consequently are not able to calculate directly the amount of material removed by the gelatine. For these reasons the Lowenthal method has been superseded by the hide powder method very largely.

The hide powder method has several modifications, probably the best being that adopted by the Association of Official Agricultural Chemists at its recent meeting in Washington. By strict attention to the details, concordant results can be obtained, as the following results show. These results were obtained by three chemists working with this method and using the same hide powder.

Summary of Extract Analyses.

Total Solids.

Analyst.	Sample I.	Sample II.	Sample III.	Sample IV.	Sample V.
A.	42·40	41·07	42·19	42·66	41·93
B.	41·20	39·73	40·78	41·20	40·73
C.	41·31	40·32	40·89	41·32	40·82

Soluble Solids.

Analyst.	Sample I.	Sample II.	Sample III.	Sample IV.	Sample V.
A.	41·20	39·68	41·47	41·88	41·53
B.	40·90	39·17	40·23	40·50	40·30
C.	40·41	39·43	40·24	40·58	39·95

Non-Tannins.

Analyst.	Sample I.	Sample II.	Sample III.	Sample IV.	Sample V.
A.	16·58	17·04	16·27	18·20	16·94
B.	15·18	15·70	14·98	16·73	15·45
C.	15·89	16·47	15·49	16·58	16·86

Tannins.

Analyst.	Sample I.	Sample II.	Sample III.	Sample IV.	Sample V.
A.	24·62	22·64	25·20	23·68	24·59
B.	25·72	23·47	25·25	23·75	24·85
C.	24·52	22·96	24·75	24·00	23·09

This method obviates many of the objections to previous schemes proposed for the use of hide powder as a measure of the tannin in raw materials, but still has the variations due to differences in the hide powder itself.

Washing the hide immediately before adding it to the tannin solution, and making a correction for dilution due to the moisture in the wet hide, removes the soluble hide error.

The use of mechanical means of shaking makes the method a rapid one, and completes the tanning operation before there is time for the production of more soluble hide. The empirical method of filtration gives comparable results on the soluble solids, and the suggestion that 20° C. be the temperature of the filtration removes a source of error, as the reds go into solution quite rapidly as the temperature rises, and *vice versâ*. Testing the non-tannin filtrate for soluble hide and for tannin are essentials of the method.

The test for soluble hide is precipitation by means of an excess of tannin solution: it is quite delicate, as the tannate of gelatin is insoluble in an excess of tannin. The reverse test—that for tannin by means of an excess of gelatin—is not sufficiently delicate, as the tannate of gelatin is soluble in excess of the gelatin solution. I have been using for this test a solution of gelatin in dilute alcohol—a saturated solution that does not gelatinise. This is made by adding 5 grms. of gelatin to 100 c.c. of water, warming, and, when dissolved, adding 40 c.c. of 90 per cent. alcohol, and filtering off the precipitate at a temperature a few degrees below that of the laboratory. In making this test a turbidity is to be expected in dilute solutions rather than a precipitate, which is best viewed in a Nessler tube.

The use of a uniform amount of hide in solutions of uniform density is a matter of such importance that I present the following data regarding it. Using a solution of a commercial tanning extract, 10 and 20 grms. to the

litre, and following the method otherwise for non-tannins, I obtained—

10 Grms.	20 Grms.
Grm.	Grm.
0·1778	0·3680
0·1790	0·3690
0·1796	0·3685
0·1775	0·3677
Average 0·1785	Average 0·3683

40 grms. of pressed washed hide, equivalent to 10·60 grms. dry hide, were used to 200 c.c. of the tannin solution, and in all cases the non-tannin filtrate was free from soluble hide and from tannin.

Using 200 c.c. of a solution of 20 grms. commercial extract to the litre, and varying the hide. 40 grms. of wet-pressed hide, equal to 10·60 grms. dry hide, gave 0·3683 grm. non-tannins from 100 c.c. of the non-tannin filtrate that was evaporated, while 50 grms. wet-pressed hide, equal to 13·25 grms. dry hide, gave 0·3493 grm. under like conditions. These are averages of four determinations, and in all cases the non-tannin filtrate was free from soluble hide and tannin. These figures show that the additional 2·65 grms. of dry hide took as a dye 0·0380 grm. of non-tannins, equal to 0·0142 grm. per grm. of dry hide, while 10·60 grms. of dry hide removed 1·2858 grms. from the extract solution, equal to 0·1212 grm. per grm. of dry hide. In connection with this, a solution of commercial glucose was made containing 8·3840 grms. dry material to the litre; a non-tannin determination was made following the method. The residue on the non-tannin weight from 100 c.c. was 0·7548 grm.

40 grms. of wet-pressed hide were used, equal to 10 grms. dry hide. 10 grms. of dry hide had taken 0·1672 grm. of glucose as a dye or 0·0167 grm. to a grm. of dry hide. Corrections for the dilution caused by the wet hide were made in each instance. These results show that variations in concentration of solution and amount of hide used are factors that must be regarded.

Variations in the amount of non-tannin material due to differing kinds of hide powder cannot be so easily regulated, and is the source of more discord in result than any other point in the method.

I tested seven different lots of hide powder by shaking 5 grms. of the air-dry hide with 200 c.c. of water containing 6 grms. of gallo-tannin, with the following result:—

1 removed	0·4094	grm. of gallo-tannin per grm. of hide.
2 „	0·8552	„ „
3 „	0·1884	„ „
4 „	0·1037	„ „
5 „	0·2531	„
6 „	0·1867	„
7 „	0·4540	„ „

Samples 3, 4, 5 and 6 did not meet the requirements of this method for hide powders, while 1, 2, and 7 did. Three chemists working with these hide powders got the following results on the same extract:—

	Per Cent.
A. Hide powder 1 gave non-tannins.........	17·07
B. „ 2 „ „	15·37
C. „ 7 „ „	17·83

While C obtained on same extract—

	Per Cent.
With hide 1 non-tannins	17·50
„ 2 „	16·55

It is therefore essential that, where tanning materials are being bought upon analysis, that the chemists be supplied with the same hide powder and observe the same method in its use.

The variations in hide powder are not due so much to the use of different kinds of hides as a source of supply, but to the means employed in preparing it.

A COMPARISON OF FUEL GAS PROCESSES.

BY F. L. SLOCUM.

The practical supplying of fuel gas to the public in sufficient quantities and of satisfactory quality has now been an established industry for little over a decade. Like many other industries, the beginning was made with supplies from nature, and are still largely obtained from this source. The successful prosecution of fuel gas supply in the last ten years has fully and accurately demonstrated the advantages derived in using gaseous fuel in every branch of industry and domestic economy, as well as developing very completely the most economical appliances for handling and burning the same; in other words, the business of distributing, supplying, selling, and burning fuel gas is well-developed and the public thoroughly educated to its use. This applies, of course, in its fullest sense to the natural gas belt, but is by no means confined to it, as the annual statements of nearly all gas companies throughout Europe and America show an annually increasing percentage of consumption of their total output for fuel and power purposes, and this at a price which is far above a comparative price with solid fuel in almost every instance.

As gas undertakings are divided into two parts—the manufacturing and distributing departments—and as the supply of natural gas is failing, we are confronted, in the natural gas region, with the serious necessity of developing the manufacturing processes so that they will be adaptable to the supplying of artificial fuel gas under all conditions. The last few years have seen processes and methods come and go almost out of number, yet through the numerous negative results as well as real advances there has been withal some permanent gain. The probable lines on which fuel gas will be produced have been clearly established, and a marked beginning made in each field. This is indeed something, for the field is a large one.

The methods of producing fuel gas can be divided into three classes—

First: *Coal gas* by distillation, including by-product ovens.

Second: *Bituminous coal water-gas producers*, or intermittent machines using bituminous coal, blasting or heating with air, and then decomposing steam.

Third: *Producers*, or continuous machines, of Mond and Siemens' type.

The above divisions are classified according to the heating value of a cubic foot of the gas which they respectively produce, and will be taken up in the same order.

Perhaps the greatest detriment to the more rapid development of successful fuel gas processes has been the so far unattainable ideal in heating value per unit of the natural product, *i.e.*, 980 B.T.U. per cubic foot. Numerous heat unit analyses of natural gas at Pittsburg made with the Junker's calorimeter show the average to be 978 to 980 B.T.U., the gas often analysing nearly pure CH_4. I therefore use for all calculations with Pennsylvania natural gas 980 B.T.U. as the proper average. This, however, does not hold true with other fields; for example, a recent examination of the natural gas supplied at Louisville, Ky., gave—

	Per Cent.
CH_4	87·75
H_2	1·31
Total combustibles	89·06
CO_2	6·60
N_2	4·34
Total non-combustibles.........	10·94
Grand total	100·00
The calculated British thermal units per cb. ft. .... =	936·8
Junker's calorimeter gave	939·0

Error very likely caused by hydrocarbon vapours, as Junker's calorimeter is an excellent check on analyses.

The highest attainable heating value in manufactured gas is from coal gas; this gives 600 to 625 B.T.U., which is approximately two-thirds the indicated efficiency of the

natural. This makes natural gas the ideal, and has led, of course, to unnumbered fruitless attempts to duplicate it. The only possible way has been by means of petroleum, and petroleum will not be considered here in any form, as its value in the liquid or natural form per million heat units is sufficient to debar further manipulation with the object of producing fuel gas to compete with coal. The fuel gas of the future will almost unquestionably be a very much lower gas in heating value per cb. ft. than is now generally sought. It is not going to be so much the question of the heating efficiency of a cubic foot of gas with the consumer as the cost of his gas per available million heat units. The question of delivery, therefore quantity, does not interest him.

First: *Coal Gas Process.*—There is little unsaid on this question. The present methods of manufacture are fully abreast of the times in the quality and quantity of gas obtained from a given coal. The tar is being disposed of to the best advantage and also the ammonia. A general change will be gradually effected by which the cyanogen will be saved—usually 12 to 16 oz. of $(CN)_2$ per ton of American coals—as well as the sulphur and benzole. This latter will average approximately 1·5 gals. per ton of coal carbonised. This will be accomplished more from necessity through larger demands for gas than from new processes. The one thing which needs a radical change is the quality of the coke. This is caused by the ever increasing demand for a metallurgical coke with the gradual diminishing beds of ideal coking coals for bee-hive ovens, also the diminishing of the anthracite deposits, whose substitute must be crushed coke of suitable hardness; added to this the fact that the increasing demand for gas requires a better coke in order to extend the market, which, with ordinary gas house coke, is very limited.

With the above conditions in existence it is no wonder that the by-product oven has lately received such prominence as to make it appear, erroneously, in a class by itself. This view has been strengthened by the great secrecy maintained by several of the by-product oven promoters as regards their machinery for, and methods of, handling the gas. It seems to the writer that the by-product oven is only to be an improvement in one of the departments of coal gas manufacture, *viz.*, the retort house.

Once the gas is made and delivered to the hydraulic main it has entered wholly into the domain of the coal gas manufacturer, and no one, I think, will successfully contradict this fact. If the by-product coke oven can replace the retort by making a better and more saleable coke, as good a gas and as great in quantity with as much tar and ammonia at a less cost of labour and repairs, then the retort must be largely replaced by the oven, and this one disadvantage in coal gas manufacture will have been brought abreast of the requirements of the times. This is now practically an accomplished fact. There are ovens now in operation which are as tight as the average gas retort, yielding a coke that is lower in volatile matter than bee-hive oven coke made from the same coal, and is very hard and thoroughly adapted to metallurgical purposes, while the gas is of excellent quality, analysing as follows:—

	Per Cent.
CO_2	3·0
Illuminants	2·6
O_2	0·2
CO_2	7·0
CH_4	30·0
H_2	52·6
N_2	4·6

The great drawback in the use of the by-product oven has been some means of economically heating them, as the ovens require gas firing to produce a good quality of coke. This, however, is most satisfactorily accomplished by producer gas (preferably the Mond type, which will be mentioned below). Only a limited amount of recuperation is required. One of the best types for recuperation is the one employed by Carvés. The system of regeneration does not seem to produce as good coke on account of regular fluctuations in the temperature of the oven during coking, producing a more unevenly porous coke, and forming a larger percentage of higher decomposing hydrocarbons, thus making a higher percentage of volatile matter in the coke.

Assuming the by-product oven in place of the retort, the products of one ton of coal, at a reasonable cost of manufacture, are—

	Lb.
Metallurgical coke	1,400
Crude tar	100
Ammonia (NH_3)	5
Cyanogen $(CN)_2$	0·75

10,000 cb. ft. gas, 600 B.T.U. = 6,000,000 B.T.U.

One ton of bituminous coal averages 30,000,000 B.T.U.

In assuming 600 B.T.U. per cb. ft. for coal gas, I have taken a very conservative average, and one that can always be maintained in practice, as the following analysis, made while making a test of Dominion coal with ordinary retorts, will show:—

The coal analysed—

	Per Cent.
Moisture	1·13
Combined volatile matter	40·25
Fixed carbon	53·76
Ash	4·86
Sulphur	1·50
Nitrogen	1·39

The coke analysed—

No. 1.

	Per Cent.
Moisture	0·44
Combined volatile matter	2·1
Fixed carbon	86·52
Ash	8·7
Sulphur	2·24

No. 2.

	Per Cent.
Moisture	1·31
Combined volatile matter	2·80
Fixed carbon	87·27
Ash	6·62
Sulphur	1·91

The analyses of the gas from this coal is a good average coal gas. The heat unit determinations were made with a Junker's calorimeter between the gas analyses and represent, not the same gas as that analysed, but give an average of the same gas and show the slight fluctuations during carbonisation.

——	1st.	2nd.	3rd.	Heat Unit Tests (Made between Analyses Gas per Cb. Ft.).
	Per Cent.	Per Cent.	Per Cent.	
CO_2	2·6	3·0	1·4	
Illuminants	5·2	5·0	6·2	1st 617·9 B.T.U.
O_2	0·8	0·4	0·6	2nd 644·9 "
CO	5·8	5·8	4·5	3rd 617·0 "
CH_4	33·7	35·1	39·2	
H_2	50·4	44·6	44·8	Avg. 626·6 "
N_2	1·5	6·1	3·3	

A sample of gas, taken when the same works were running on Westmoreland (Pennsylvania) coal, gave, on analysis, the following:—

	Per Cent.
CO_2	1·8
Illuminants	5·9
O_2	1·1
CH_4	42·8
CO	3·6
H_2	40·9
N_2	3·9

The candle power was 17·78.

Calorimeter gave 690 B.T.U. per cb. ft.

This, with the other analysis given above, shows that there is no difficulty in producing at least a 600 H.U. gas and maintaining it in daily manufacture.

Second: *Bituminous Coal Water Gas Producers.*—Under this type are classed such apparatus as manufacture gas from bituminous coals by means of first heating a large body and depth of fuel with an air-blast, burning the coal at bottom to coke, then admitting steam and producing water gas, the water gas formed passing up through the undecomposed coal, thus distilling off a part of the volatile matter as coal gas. The best developed machines of this type are the Hastings and Loomis. The regular water gas machines are not considered here, as they use only expensive fuel and produce a lower heating value gas.

An analysis of blue water gas made from gas house coke in the Granger machine gave—

	Per Cent.
CO_2	4·8
CO	41·5
H_2	52·41
CH_4	0·2
N_2	0·47

A heat unit determination of the same gas with Junker's calorimeter gave—

B.T.U. per cb. ft.	331·5

The ideal of this class of gas machine would be a *machine which would remove all of the volatile matter from the coal as coal gas and convert all carbon into water gas, i.e.,* H_2 + CO. This would be accomplished in part by either getting the necessary heat from outside sources to maintain incandescence, or when blasting up to allow the blast gases to pass only through the carbonised portion of the coal. Numerous patents have been taken out to accomplish this in the different ways mentioned, but as yet this field is open to very marked improvements.

The process which is the best developed in this class is the Hastings, or, as the European patents read, the "Hastings and Hauk process." The writer had occasion to examine this process, which is in operation as an auxiliary to the natural gas plant at Louisville, Ky. The test extended over a period of 10 days. Jellico coal was used, analysing as follows:—

	Per Cent.
Carbon	63·10
Volatile matter	32·53
Moisture	1·90
Ash	2·4
Sulphur	0·07

In this process they work with a mass of fuel from 10 ft. to 12 ft. deep. In heating up by means of the air-blast, the gases produced rise up through the whole mass of fuel, thus destroying a part of the volatile matter of the coal. An analysis of these blast gases taken at the outlet of the machine gave—

	Per Cent.
CO_2	13·15
O_2	2·00
CO	9·00
N_2	76·15

The volume of this gas produced per blast was 12,085 cb. ft. corrected to atmospheric pressure. It was found afterward that there was an extra air-blast over the coal, which destroyed all traces of CH_4 and H_2. The blast gas was then completely burned in an upright tubular boiler recuperating nearly 10 per cent. of the heat generated in the process of manufacture. It is, however, safe to assume that quite as much of the volatile matter is distilled with the blast gases as by the water gas, as the heat range is necessarily exactly the same in each. The coal gas in the coal should be in volume approximately one-fifth, or as one is to four, hence the following calculated mixture should represent the resultant gas in this process. The two actual analyses made from two different runs show that this deduction is practically accurate.

—	Water Gas (Typical).	Coal Gas (Typical).	Mixture (Calculated).	Analysis (No. 1 Found).	Analysis (No. 2 Found).
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
CO_2	2·2	2·0	2·16	8·2	5·4
N_2	3·4	2·0	3·12	5·2	9·2
O_2	0·4	1·0	0·52	0·6	1·1
H_2	57·5	49·0	55·8	43·8	41·9
CH_4	..	35·0	7·0	8·2	7·5
CO	36·5	6·0	30·4	32·8	34·0
Illuminants	..	5·0	1·0	1·2	0·9

B.T.U.		363·3	342
Specific gravity of gas		0·576	

The large percentage of non-combustibles is caused by poor construction of the machine used, there being leaky valves which allowed air to work in during gas making, as a constant air pressure was maintained against these valves.

The average yield of the 363 B.T.U. gas per ton of 2,000 lb. of coal was 51,250 cb. ft. (corrected).

There was some considerable tar produced, and 8·03 lb. of ammonia per ton of coal, calculated as sulphate.

The gas indicated five-candle power (approximately).

Taking the first analysis in the above table, which gave 363·3 B.T.U. per cb. ft., multiply by 51,250, the number of cubic feet of gas from a ton of coal gives an efficiency of 9,309 B.T.U. in gas from each lb. of coal used. 1 lb. of Jellico coal gives 15·105 (calculated). This, therefore, shows a fuel efficiency of 61·63 per cent. of the coal used, without considering the coal used to produce steam. It was impossible to obtain this last data, on account of the construction of the plant and other uses of the steam.

I have received from the company who operate the plant the following as the average of their winter's operation. They are making, I understand, about 2,500,000 cb. ft. per day.

Each 1,000 cb. ft. required to produce—

	Lb.
Coal	23
Coke	5
Coal under boiler	10
Total	38

The time of admitting the steam, or of gas making, was eight minutes. On one of these normal runs the gas was sampled every two minutes, and analysed, with the following results:—

—	End of First Two Minutes.	End of Fourth Minute.	End of Sixth Minute.	End of Eighth Minute.
	Per Cent.		Per Cent.	Per Cent.
CO_2	4·4	Lost	9·0	9·5
Illuminants	1·0	"	0·8	0·7
O_2	1·0	"	2·2	3·8
CO	33·0	"	28·8	23·5
H_2	44·2	"	45·24	45·1
CH_4	7·8	"	4·2	6·2
N_2	8·6	"	9·76	11·2

The analyses show that the hotter the gas the greater the amount of volatile matter carried off. This type of apparatus, with some few changes in construction, should produce a 400 B.T.U. gas. This has been corroborated in part by some recent analyses made on a new plant, in which some of the errors noted in the first had been corrected.

The coal used was ordinary slack, mined near Greensburg, Pa., and analysed as follows:—

	Per Cent.
Moisture	1·00
Volatile matter	31·93
Fixed carbon	59·99
Ash	7·08

No oil was used, and the sample of gas for first analysis was taken from the large holder. The second was taken from a second holderful and a different run.

——	First.	Second.
	Per Cent.	Per Cent.
CO_2	7·9	8·1
Illuminants	1·4	1·1
O_2	0·1	0·3
CO	28·5	28·1
H_2	48·4	49·65
CH_4	8·2	9·82
N_2	5·2	3·95

Calorimeter test gave 390 B.T.U. per cb. ft., which shows that by a more careful exclusion of nitrogen and a proper reduction of the CO_2 there will be no difficulty in making and maintaining a 400 B.T.U. gas.

With proper appliances for utilising the producer gas formed in blasting up and the saving of the waste heat in the gas made, the net actual efficiency should be raised to at least 70 per cent. of the fuel charged, while any class of fuel can be used.

The net results at a very moderate cost of manufacture per ton of coal would be (approximately)—

8·0 lb. ammonia sulphate.
52·500 cb. ft. of 400 B.T.U. gas = 21,000,000 B.T.U.
One (1) ton of coal = 30,000,000 B.T.U. (Apx.)

Third :—*Producers or Continuous Gas Machines.*—This type is represented by the Siemens and Mond producers. The inventions of these two gentlemen represent the two steps of real progress made in continuous gas machines, *i.e.*, the manufacture of gas by partial combustion with the recuperation of waste heat and saving of by-products. Dr. Siemens so constructed his producer as to get the very largest percentage of decomposition of CO_2, admixing eventually with the air a small percentage of steam in order to reduce his temperature, at the same time forming H_2 and CO from same. The following analysis by Ritchie is a typical one, and gives the practical working limit of this machine :

	Per Cent.
CO_2	5·2
CO	24·4
C_2H_4	..
CH_4	2·4
H_2	8·6
N_2	59·4

Caloric value in B.T.U. per cb. ft., 137·28.

The amount of gas produced usually represents, in heating efficiency, from 60 per cent. to 65 per cent. of the coal charged in the producer. This type of machine practically supplied the demands for producer-gas from 1861 to 1889. True, there were many modifications, but no great progress was made in the art during this time.

The advent of Dr. Mond's invention marked the second epoch in this branch of gas making. His apparatus produces a higher heat unit gas per cb. ft., gives approximately a 20 per cent. better yield per ton, or over 80 per cent. fuel efficiency of the coal charged in the producer. The gas is clean and cold, and can be piped any distance, burning readily cold, as it is very high in hydrogen, and is adaptable to all purposes where fuel gas can be used. Added to this, about 100 lb. of sulphate of ammonia are produced and recovered per ton of coal, or, putting it more exactly, at least 70 per cent. of the fixed nitrogen in the coal is recovered as NH_3. The machine is nearly automatic, and the composition of the gas changes very little. An average of many analyses from several months' operation gives—

	Per Cent.
CO_2	17·0
CO	11·0
C_2H_4	0·4
CH_4	1·8
H_2	27·2
N_2	42·5
	100·0

Calorific value in B.T.U. per cb. ft. = 156·9.

The coal used to produce this is a very poor quality of slack.

The first installation of these producers on this continent was at Halifax, Nova Scotia, and are being worked successfully with Dominion Coal Co.'s slack, the average analysis of the gas being—

	Per Cent.
CO_2	18·0
O_2	..
CO	9·9
H_2	27·0
CH_4	2·7

Taking the Mond producer as the type of this class, one ton of coal yields, with a very moderate expenditure in labour—

160,000 cb. ft. of 156·9 B.T.U.gas = 25,105,000 B.T.U.)
100 lb. of ammonium sulphate.)
(One ton of coal equalling, approximately, 30,000,000 B.T.U.

Tabulating the results obtained in the different classes we have, as the products of one ton of coal—

——	Cb. ft. of	B.T.U. of Total Gas.	Coke.
Coal gas	10,000	6,000,000	1,400
Bituminous water gas.	52,500	21,000,000	..
Producer gas (Mond).	160,000	25,105,000	..

——	Ammonia (NH_3).	Tar.	Benzol.	Cyanogen.
Coal gas	5 lb.	100	1·5 gal.	0·75
Bituminous water gas.	2 lb.	..	..	..
Producer gas (Mond).	100 lb. sulphate.	..	..	..

The cost of producing a thousand feet of gas by these different processes, not taking either the coal used or the products sold into consideration, using only the cost of labour. The interest on the capital investment and cost of materials in the operation is—

	Cost per 1,000 cb. ft.
Coal gas	5·0 c.
Bituminous water gas	0·9 c.
Producer gas (Mond)	0·62 c.

This gives only the cost value of producing a given volume of gas with its accompanying by-products, as given above. The cost of coal with the price obtainable for the different by-products all combine to change these values. These conditions, however, are different in each different locality and cannot be given here, but can be easily calculated for any locality from the above tables.

The last and one of the most important considerations is the real comparative value of the different gases, with each other, to the consumer; in other words, taking natural gas as a unit, its comparative value as coal is so thoroughly established in practice that it certainly makes a very accurate standard. Gas is found to be less economical in the generation of steam than for any other purpose. The average of a large number of tests show that it requires 80 per cent. of the heating value of the coal in natural gas in order to get the same results, labour, repairs, &c. considered. In one test of two months' duration, the heating efficiency of the gas used was 81·67 per cent. of that of the coal in order to obtain the same results under the same boilers. This is also true of Mond gas, as tests already made give the same evaporation from the gas produced from one ton of coal as burning direct under the same boiler. (It has been noted above that the fuel efficiency of the Mond producer is a little over 80 per cent.)

In all other branches of industry, especially iron, steel, and glass there is required only 40 per cent. to 45 per cent. of the actual heat units as gaseous fuel as compared with bituminous coal.

In domestic use, *i.e.*, cooking and the heating of houses, the economy is still greater. It is proven by long practice

that 15 per cent. to 20 per cent. of the actual heating efficiency of a ton of coal delivered as gas will compete successfully with coal. The above calculations are based on the prices obtained for natural gas and coal in Pittsburgh, and the difference in value unit for unit includes all advantages and savings connected with the burning of gas as compared with coal. These results tabulated, give—

—	1 Ton of Coal, including all Costs, Firing, Repairs, &c.	Gas Equivalent, including all Costs of Burning.	Percentage Equivalent of Gas necessary to equal Coal = 100 per Cent.
	B.T.U.	B.T.U.	Per Cent.
Generation of steam.	30,000,000	24,000,000	80
General mill use	..	12,000,000 to 13,500,000	40 to 45
Domestic use ...	,,	4,500,000 to 6,000,000	15 to 20

In supplying and consuming gas it is very advantageous from the manufacturers' standpoint, to have as high a heat unit gas as possible on account of capital investment in mains; while from the consumers point of view it does not make as much difference, as the higher heating value gases require more air to accomplish complete combustion. The different percentages of air are therefore given below.

In the calculations given below a definite composition of each of the different gases considered had to be assumed in order to find the exact amount of air necessary for combustion, as well as the temperature of combustion.

The following analyses have been selected as representative average analyses of each gas considered :—

Natural Gas (PGH) (Analysis made April 1897).		Coal Gas.		Bituminous Water Gas.		Mond Gas.	
	Per Cent.		Per Cent.		Per Cent.		Per Cent.
CH_4	96·9	CO_2 ...	2·2	CO_2...	6·83	CO_2....	17·0
C_nH_m ...	0·8	Illuminants.	5·2	Illuminants.	1·24	CO	11·0
CO_2.......	0·3	O_2	0·6	O_2	0·2	Illuminants.	0·4
N_2........	1·2	CO_2....	3·8	CO ...	29·46	CH_4 ...	1·9
		CH_4 ...	36·5	H_2....	48·95	H_2.....	27·2
		H_2	45·4	CH_4 ..	8·68	N_2.....	42·5
		N......	6·3	N_2....	4·64		
			100·00		100·00		100·00

Air necessary to burn completely 1 cb. ft. of each of the above gases is as follows:—

Natural gas	9·80 cb. ft. air for complete combustion.		
Coal gas	5·65	,,	,,
Bituminous water gas	2·97	..	,,
Mond gas..................	1·25	,,	,,

Before closing, I wish to acknowledge the able assistance rendered by C. S. Lomax, who has had charge, under my direction, of all the different examinations from which extracts have been made for this article.

Meeting held on Friday, March 19th, 1897.

PROF. C. F. CHANDLER IN THE CHAIR.

ON THE ANALYSIS OF ASPHALTUM.

BY S. F. AND H. E. PECKHAM.

Dr. H. Endemann, in the Journal of the Society of Chemical Industry for December 1896, has proposed a new scheme for the technical analysis of asphalts, which may be characterised as wholly radical. We shall not attempt to discuss all of the points that he makes. A few will be sufficient.

One of us has been at work on asphaltums of various sorts since 1865. We fully agree with the inference that may be fairly drawn from what Dr. Endemann says, that we can all plead considerable ignorance of the subject; still, something has been learned in the 60 years since Boussingault attempted to separate the maltha of Pechelbronn into its constitutional principles. We think that Dr. Endemann has overlooked some of the facts, of which it may be said we are pretty sure. Of these facts let us mention a few as pertinent to this discussion: that bitumens, from natural gas to asphaltum, include compounds and mixtures of compounds belonging to all of the known series of hydrocarbons; that of the more dense petroleums, malthas, and asphaltums these compounds have been very little investigated; and that no one has pretended to determine the series present in any asphaltum. Consequently, to talk about *petrolene* and *asphaltene* as entities, as chemical compounds, is no more rational than to speak of kerosene and naphtha as constituents of petroleum.

Dr. Endemann deals with refined asphalts as if they were quite independent of the crude materials from which they were derived. As a chemist we think he has made a great mistake at the outset. He says that asphalt is asphaltene dissolved in petroleum. He might, with equal propriety, say that petroleum is petroleum tar dissolved in kerosene. Neither the word *petrolene* nor *asphaltene* has any meaning as a name for a chemical compound.

While at Trinidad we were impressed with the large amount of strong mineral water that saturated the crude pitch, and the important rôle that it must play in the technology of that substance. The water itself is of no consequence, it is the salts that the water holds in solution. If the water is all evaporated the salts may act only with greater energy. We therefore determined to study the pitch with reference to this question. The pitch, when taken out of the lake is completely saturated with this water, some of it is dripping wet. When it is refined the water dries out, and the salts, particularly the sulphates, react with the bitumen. Every refiner knows that vast quantities of hydrogen sulphide escapes. Does Dr. Endemann think that this gas exists entangled in the pitch ready formed, and is simply driven off by heat? If so, he is greatly mistaken.

We have carefully examined pitch procured from both within and without the lake. Their action was identically the same. About 100 grms. were placed in an iron casserole and heated very carefully, with a thermometer as a stirrer. They softened at about 140° F., and continued to grow softer, until at 200° F. they were quite plastic, and began to give off little puffs of vapour. On testing these puffs with lead paper the reaction was strong for hydrogen sulphide. As the heat increased the reaction became more energetic, until at 300° F. the melted pitch boiled from escape of gas, a large part of which was hydrogen sulphide. The temperature was maintained at about 400° F. for about three hours, until the fused pitch became quiet and gave off very little gas. The gases given off from start to finish consisted largely of hydrogen sulphide.

In order to further test this matter, 100 grms. of crude commercial lake pitch, from near the tramway, was coarsely pulverised, suspended in distilled water in a flask, placed on a steam bath, and heated at from 140° to 150° F. At that temperature the pitch was not adhesive, and remained suspended in the water. It was digested in successive portions of water for about a week, until the water no longer gave a reaction for sulphuric acid, the whole amount of water being about 5 litres. The first portion of water used contained a notable quantity of ferrous sulphate, which, on standing, became ferric sulphate, with deposition of ferric oxide. The 5 litres were concentrated to one, and portions of 100 c.c. each were evaporated in duplicate on a water-bath for total solids. The amount obtained equalled an average of 1·355 per cent. of the pitch. In two other portions the sulphur existing as soluble sulphates was determined. It amounted to 0·1483 per cent. of the pitch. The residue from which the aqueous solution had been made, when dried, was without cohesion at ordinary temperatures, and when rubbed up in a mortar resembled in appearance a rich garden soil. Duplicate determinations of the sulphur in this extracted pitch gave an average of 3·3425 per cent. of the pitch originally taken. Duplicate

portions were then extracted with petroleum ether, with petroleum ether followed by boiling spirits of turpentine, and with the same followed by chloroform, and the sulphur determined in the several pairs of solutions. The values obtained were as follows :—

	Per Cent.
Sulphur in the aqueous solution	0·1483
" " petroleum ether solution	1·1450
" " spirits of turpentine solution	0·7818
" " chloroform solution	0·5313
" " residuum solution	0·8844
Total sulphur in pitch	3·4908

When Miss Linton analysed the 27 specimens for the Persia trial, the chloroform extract accumulated to the amount of about 2 galls. The excess of chloroform was distilled off on a water-bath, the residue evaporated nearly to dryness and allowed to become quite dry by exposure to the air of the laboratory. The dry residue was repeatedly washed in 95 per cent. ethylic alcohol until the alcohol was no longer coloured; when a very light, dry, brown, and incoherent powder remained, which contained 5·871 per cent. of sulphur. When carefully heated in a platinum crucible, it became black, intermixed, gave off, first, hydrogen sulphide, then dense vapours, which took fire and burned with a smoky flame, leaving a mass of coke that soon burned off, leaving a very light ash that retained the form of the intermixed mass. This ash consisted almost wholly of ferric oxide that dissolved in hydrochloric acid with difficulty. The amount was 5·016 per cent. of the powder =3·5112 per cent. of iron, which, with the 5·871 per cent. of sulphur amounted to 9·3822 per cent. that was neither carbon, hydrogen, nor oxygen. We believe, however, that the sulphur and iron are inorganic combination with the other three elements.

This chloroform-soluble part is the only constituent of Trinidad pitch that has been isolated in such form as to approach the requirements demanded of a chemical compound. Of course if it contains nearly 6 per cent. of sulphur, which Dr. Endemann did not know was there, his determination of the carbon and hydrogen are probably valueless. We have not made any combustions for several reasons, chief among which is the fact, that the material, while all obtained from Trinidad pitch, was not all from the same specimen. We have concluded that the brown powder is a sulpho-salt of iron, in which ferrous sulphide is linked by sulphur to an organic radical. It is properly to be considered a part of the bitumen. Before we know anything about the compound, if it be a compound, it has got to be treated as suggested by Miss Linton in her first paper—a suggestion that Dr. Endemann appears to have overlooked. She says "a review of the results here given suggests the query, have the methods heretofore employed for the technical analysis of asphaltum really been analytical at all. Would not a method and process suggested by the results of the qualitative analyses given in this paper, and based on the successive application of different solvents and yielding results similar to those of fractional distillation, really become analytical, especially if the separate portions dissolved by the different solvents were subjected to such treatment by oxidising agents as would enable us to determine to what groups of hydrocarbons the different substances dissolved respectively belong"?* The colossal magnitude of this problem deters any chemist who knows anything about it from attempting its solution.

There are few substances in nature more complex than crude Trinidad pitch. It consists of crude bitumen, mineral salts, sulphates, chlorides, &c., partially decayed wood, humus, humic acid, &c., sand and clay, ferric disulphide in such a minute state of division that it may almost be said to be molecular, and sulphur; some of it no doubt free, and the remainder in various forms of organic combination. No chemist has yet attempted to show what the varied reactions of these materials upon each other produces as a resultant in the crude pitch, nor has any attempt been made to show what the chemical effects of refining are. It is obvious that a part of the sulphur escapes as hydrogen sulphide. It is quite certain the amount so removed depends upon the temperature and the length of time the pitch is heated; but what becomes of the sulphur that remains, what new combinations follow; what part, if any, those remarkable humus compounds play in the new arrangement; how much, if any, of the compounds of iron and sulphur are removed, and many other problems, are those that the chemists of the future have got to solve.

We know already that refined Trinidad pitch may consist of a good quality of crude pitch carefully melted, containing, after being refined, about the same percentage of sulphur as the crude; but, as a large proportion of the pitch is removed in the process of refining, so a correspondingly large proportion of the sulphur has disappeared. There is nothing in the treatment by solvents to change the condition of the mineral matter. If, however, the pitch has been oiled in the still, as Mr. Richardson says in one of his reports is the custom of the Barber Co., the problem of comparison of results becomes infinitely complicated: for, as the pitch is made more fluid, more or less sulphur may burn out more or less hydrogen, and a variety of combinations and recombinations follow. Dr. Endemann seems to think that oiling simply adds to the petrolene and makes it capable of dissolving more asphaltene. If that is the case it destroys the validity of the comparison and increases the apparent value of the oiled specimen. It may do more: it may menace the volatility as well as solubility of the so-called asphaltene, and thus augment the apparent value of the specimen. It also makes a specimen apparently require less residuum oil to soften it, when in reality it does not, the oil having been added in two portions.

Again, the pitch may have been blown with air in refining. Blowing bitumen with air at 400° F. burns out the hydrogen. The Blyerite process proves it in the same manner that the Dubb's process proves the burning out of hydrogen by sulphur. A large amount of personal experience, which we are not at liberty to further explain, has proved to us years ago that both air and sulphur will at high temperatures remove hydrogen from asphaltum, making it hard and brittle.

If the pitch is oiled in the kettle, the increased fluidity of the refined pitch makes a barrel of pitch extremely irregular in its composition. Much of the organic matter, not bitumen, and a part of the chloroform soluble will rise to the top, while a part of the mineral matter sinks to the bottom, thus leaving the middle of the barrel much richer in desirable qualities than the average of the barrel. Those who are posted take their samples from the middle of the barrel.

Now Dr. Endemann has proposed to take a sample of one or the other of these refined pitches and heat the bitumen extracted by chloroform for 29 hours in a stream of carbonic acid gas, at a temperature of 200° C.—220° C. =392° F.—428° F., and weigh the residue as asphaltene. He says, "the loss should be called water and light petrolines." Why? It should be called exactly what is. If the chloroform is dry, as it should be, where does the water come from? At that temperature the distillates are not light unless they are cracked products of decomposition, as they might be in this case. If they are not decomposition products they are heavy oils. As long as any volatile matter is driven off, hydrogen sulphide will form a part of it, until nothing but coke is left; why, then, shall we name it until we know what it is?

He says that "asphalt is mainly asphaltene dissolved in petrolene, like turpentine." What kind of asphalt does he mean? Is it one containing 7½ per cent. of sulphur or one half of one per cent. of sulphur? Is it one with a large per cent. of pyridin bases, that on distillation will not yield a trace of paraffin, or is it one that contains only a trace of nitrogen and yields largely of paraffin? Is it one that forms substitution compounds with great readiness with nitric acid, or is it one that is simply oxidised into water and oxides of carbon, without a trace of nitro-substitution compounds? We satisfied ourselves 30 years ago that nothing but decomposition products can be obtained

* Journal of the Amer. Chem. Soc. 16, p. 809; Chem. News; Analyst; Chem. Centr. Blatt.

from the distillation of solid asphaltums. Why, then, should we attempt to go back to Boussingault's early research, which he himself admitted was unsatisfactory?

We have lately had two specimens of refined Trinidad pitch submitted to us by members. They were so nearly alike in colour that when powdered and placed in the sun in a window seat to dry, they were both black and coherent in 10 minutes, and if not carefully labelled could not have been distinguished.

The results of analysis were reported as given below:—

—	I.	II.
	Per Cent.	Per Cent.
Soluble in petroleum ether	39·340	38·485
„ turpentine, after	19·270	21·705
„ chloroform „	0·713	1·133
Total bitumen	59·250	61·323
Organic and volatile	8·507	7·460
Mineral matter	32·243	31·217
	100·000	100·000
Sulphur	3·639	3·372
Silica	19·182	20·005
Oxides of iron and alumina	21·168	19·377
Total bitumen—		
Soluble in petroleum ether	66·409	62·743
„ turpentine, after	32·417	35·394
„ chloroform „	1·174	1·863
	100·000	100·000
Proportion of total bitumen soluble in boiling turpentine.	98·854	98·137

Upon these results we pronounced these two specimens, samples of practically the same thing. Such an examination involves a considerable amount of careful analytical labour, but the results are not without value. If anyone has anything really better to offer, no one would be more glad than ourselves to hear the report.

Discussion.

Mr. Clifford Richardson, in reply to a question whether the silica and the oxides of iron and aluminium were given on the total or on the percentage in the ash, said that they were the percentages in the ash. Adding together the 19 and 21 made 32. The silica and oxide of iron made up the total. The silica was the much larger constituent of the ash.

Dr. Endemann said it seemed evident that the author looked upon the sulphur as a necessary constituent, not only of asphalt, but also of asphaltene, or of the substance which remained behind after distillation. Without intending to deny the presence of a certain amount of sulphur in bitumen prepared by the author's method, he (Dr. Endemann) denied the conclusions which the author had drawn from the work he has done. Sulphur was present in most asphalts, but the sulphur was not a consituent of asphaltene. His own investigation of asphalt was with a view of isolating those substances which really gave asphalt its peculiar properties, and the substances which he had isolated did not contain sulphur. He consequently left out all such substances which he knew did not belong to the bituminous material. He separated the sulphur on the same principle that he separated the ashes. If Texan asphalt, alluded to in his second paper (this Journal, 1897, 122), were distilled, sulphur compounds, as well as some sulphuretted hydrogen, were obtained in the distillate. The residue, dissolved in a little chloroform and evaporated, contained in the average about 0·4 per cent. of sulphur. Of this 0·4 per cent. of sulphur, however, he had been able to eliminate during the whole process described in the paper still more, leaving only a little over 0·1 per cent. of sulphur behind. Asphaltic oxide $C_{26}H_{26}O_4$ contained 16 per cent. of oxygen. One atom of oxygen therein would correspond to 4 per cent. of oxygen; one atom of sulphur in such compound would correspond to 8 per cent. of sulphur. Assuming the material to contain 0·4 per cent., the formula must be multiplied by $\frac{8}{0 \cdot 4} = 20$, in order to insert one atom of sulphur; that is, they would have a formula for asphaltic oxide like the following: $C_{520}H_{520}O_{79}S$, which was quite preposterous. But since more than half this sulphur could be removed by further treatment, it was clear that sulphur was simply a slight impurity, owing to an imperfect method of extraction. There were thus asphalts where sulphur played no part whatever. Admitting that Trinidad asphalts contain a very large amount of sulphur, he had not analysed for it, not wanting sulphur in his product; and he had done his best to exclude it during the purification described in his former papers. The author implied that the asphaltogen or asphaltene contained sulphur of constitution, and that it was this sulphur which linked the metals to the organic portion; that is, he looked upon the solution of the iron as due to the formation of a thio salt. Now, apart from the fact that the chloroform-soluble inorganic material consisted mainly of alumina with only small quantities of iron and lime, and that aluminium sulphide could never exist in a mass often containing 10 per cent. of water, if bitumen of refined Trinidad asphalt were extracted by means of a little chloroform, a considerable amount of sulphur dissolved. But if that solution were diluted with more chloroform, as stated in his previous paper, a precipitate was obtained, which, on being examined, gave far more sulphur than the pitch which remained in solution. Further dilution gave a further precipitate. If that were filtered off and the solution allowed to stand a day of two, the apparently clear liquid deposited a third precipitate, and thus within a few days the 10 or 6 per cent. of sulphur which the author found in his asphalt came down to about 1 per cent. Of course the removal afterwards of the petrolene would still more reduce the sulphur in the residue. Now, according to his theory, the sulphides were soluble in a chloroform solution of asphaltene and more soluble in concentrated solution than in dilute solution. This was nothing so very extraordinary. They could not dissolve resins in water, yet they knew that they could dissolve resins in water containing other substances in solution. Those who had had anything to do with the poorer grades of logwood extract would know that logwood extract diluted with water gave a heavy precipitate, insoluble on washing; but it was in perfectly clear solution before. Refined asphalt contained, like nearly all carboniferous minerals, pyrites, and later, after exposure, perhaps also sulphates; so it might contain arsenical pyrites. That these substances might have influence on the two asphalt constituents during application was possible, and certainly worth the study; but everybody must admit that this was a secondary matter.

Now as to the reaction accompanied by the formation of sulphuretted hydrogen. For many years his hydrogen sulphide apparatus consisted of a flask filled with paraffin, to which a certain amount of sulphur had been added. Heating alone produced sulphuretted hydrogen. But if iron pyrites instead of sulphur were taken and heated with paraffin they would find that, instead of all sulphur passing over, with formation of an unsaturated hydrocarbon, in the case of iron pyrites, FeS_2, only half the sulphur passed over, while half remained behind with the iron. The petrolenes contained paraffins. In Texas asphalt, at least, the petrolene contained about 25 per cent. of paraffins. The other substances or substance seemed to be a diterpene or perhaps a mixture of several diterpenes, as far as he had been able to determine. Boussingault made an analysis of petrolenes obtained from the asphalt of Pechelbron, and he found it had the composition of spirits of turpentine. However, it had a very much higher boiling point, and the vapour density was found to be about 9, which would indicate that he had not a terpene but a diterpene in his hands.

His reasons for believing that there was a diterpene present were: concentrated sulphuric acid acted upon the material very energetically, developing sulphurous acid, and producing, besides a sulpho acid, a solid black condensation compound, looking like asphaltene, but which was not asphaltene; also the boiling point of the sub-

stance, the peculiar fluorescence, and the artificial musk odour on treatment with nitric acid. Artificial musk is trinitrocymene. The products which he obtained could not be chemically pure. The nature of these substances did not permit it. He had stated repeatedly that his formulæ were only approximate, and to be used simply as guides in chemical analysis, and in this case they held good. By whichever of his methods the chemical analysis was carried out he generally obtained concordant results.

The author's references to his paper as to how asphalt should be treated, &c., were not always correct. He stated that Boussingault's process was unsatisfactory, and that he had said himself it was unsatisfactory. The reason he (Dr. Endemann) had never dwelt upon it before was this: Boussingault had found that when he made a combustion of his original asphalt, that he could find in it only 2 to 3 per cent. of oxygen. When afterwards he examined his so-called asphaltene he found it contained 15 per cent., and when he developed the relation between the two, then he found out he got a good deal more of asphaltic oxide than would correspond to that small quantity of oxygen which he found in the original asphalt. In this special case he had asphalt which contained very likely asphaltogen as well as asphaltene.

The Chairman said he understood from the paper that the author seemed at issue with Dr. Endemann, because he understood Prof. Endemann to intimate that asphaltum was a combination of two or three simple definite compounds, while his opinion was that asphaltum was a mixture of an indefinite number of definite compounds.

Dr. Endemann said, as the result of his last investigation, he started with an asphaltogen containing less than 0·2 per cent. of sulphur, and being, therefore, a practically pure hydrocarbon. This substance oxidised to asphaltene, then to asphaltic acid, and finally to asphaltulmic acid. Commencing with a pure hydrocarbon, he obtained all these substances. Of these substances the asphaltogen and asphaltene and asphaltic oxide might be found in one single specimen of asphaltum. The asphaltic oxide, however, was not a substance which possessed bitumen properties, so that the solid hydrocarbon or the solid bitumen consisted of two substances—asphaltogen and asphaltene; the volatile substances consisted of a series of hydrocarbons belonging to the paraffin series and also one or more hydrocarbons belonging apparently to the diterpene series. This investigation was not concluded, but at any rate there were a number of substances present. Mr. Peckham based his remarks on his first paper which treated simply of the hard asphalts used for paving. Those contained mainly asphaltene. At that time he did not yet know asphaltogen. He did not get asphaltogen until he commenced to work with the malthas. His first paper was on the hard paving asphalt after being refined, while the second paper treated of the malthas, which reached them also refined; that was, so far refined that the water was driven off before they were sent out, and probably the very lightest petrolenes. There were a great variety of asphalts, both hard and soft, but he had invariably been able to get asphaltic oxide from any and all asphalts which he had examined. That is, they contained a hydrocarbon or an oxygen compound which oxydised at a temperature of between 220° and 250° C., and might finally be converted into the substance asphaltic oxide.

Dr. Schweitzer asked if every asphalt contained asphaltogen.

Dr. Endemann said that every one which he had seen contained asphaltene; even the petroleum residuum oil used as a diluent in case of fluxing for cement contained about 25 per cent. of asphaltogen, which might be obtained as asphaltic oxide.

The Chairman asked what were the characteristic properties of asphaltene generally.

Dr. Endemann, in reply, said that asphaltene was, at ordinary temperature, solid. Its melting point was at about 210° C. When melted it soon became covered with a wrinkled skin, and this skin, when pushed aside, became quite elastic, and another skin formed, all of which skins finally were converted into asphaltic oxide of the formula $C_{26}H_{26}O_4$. If mixed with real petrolene, obtained by the distillation of asphalt, it gave a mixture with the properties of asphaltum.

In reply to further questions, Dr. Endemann went on to say that asphaltogen was not very good for varnish purposes; asphaltene was better. A certain quantity of asphaltogen would make an asphaltene more elastic. In the case of varnish gum, the petrolenes were nearly all driven out, and a small quantity of asphaltogen was of great value as taking the place of the petrolene, and producing elasticity, with the additional advantage that it was far more stable than the more volatile petrolene. The asphaltogen had a very high boiling point. Asphaltogen was separated from asphalt as follows:—In the first place, the amount of asphaltogen present was determined by the quantity of asphaltic oxide which can be obtained from it. Guided by this, the asphalt was distilled in a partial vacuum; it being necessary not to overheat the material, because the temperature for the distillation of petrolenes and the destruction of the asphaltene lay very near together. A member present had tried distillation in an absolute vacuum, and perhaps would give his experience.

A member stated that in his hands distillation in a partial vacuum was not a success; but a vacuum of about 711 mm. gave a very good result. By distilling in a half vacuum, he obtained a peculiar decomposition and a sulphurous odour, and on this account he decided to get a perfect vacuum with a pump working with about 42 ft. water pressure. With this pump he got a splendid result, but distilled about ½ oz. only. The first trial in a vacuum was not a success; his half-gallon retort collapsed. With a sand-bath it distilled very easily and very quickly. The liquid was perfectly transparent. He used Trinidad asphalt, but did not know to what extent it would be of use to Dr. Endemann.

Dr. Endemann said the Trinidad asphalt did not contain much petrolene. He had used an asphalt containing about 25 per cent. of petrolene, and with this he was able to obtain a sufficient yield of material.

Mr. S. F. Peckham wrote, in reply, that Dr. Endemann's paper in the December issue of the Society's Journal was the only paper of his relating to asphalts that he had seen. He did not recollect having said that sulphur was a necessary constituent of asphalt or asphaltene, of which latter substance he had no knowledge. The statements made were the result of careful research, extending over many months on his own part and that of his son. They were intended to refer to Trinidad pitch solely, and not to bitumens in general, asphaltene, asphaltogen, or any other substance. After having an opportunity to study the discussion in print, and the paper on the same subject promised by Mr. Richardson, he might possibly prepare some further notes on the subject.

Journal and Patent* Literature.

* Any of these specifications may be obtained by post by remitting 8*d*.—the price now fixed for all specifications, postage included—to Sir Henry Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

I.—PLANT, APPARATUS, AND MACHINERY.

Stone [Artificial], capable of Resisting Fused Alkalis, Alkali Sulphides, and Hydrochloric Acid. W. Hempel and W. Sezierski. Bayerisches Ind. u. Gewerbe Blatt, Thonwaaren Ind. **11**, 148.

See under IX., *page* 442.

New Fractional Distilling Apparatus for Laboratory and Industrial Uses. A. Tixier, Chenal-Ferron-Douilhet and Co. Bull. Soc. Chim. 1897, **17**, 392.

See under XXIII., *page* 465.

PATENTS.

Superheating Steam, Impts. in Apparatus for. G. Sinclair, Albion Boiler Works, Leith, N.B. Eng. Pat. 7819, April 14, 1896.

The "apparatus for superheating steam, comprises two vessels provided with steam inlet and outlet branches, and connected together by rows of tubes, the tubes being arranged in the main flue or equivalent part of a boiler or set of boilers," so that they are heated "by the outgoing gases."—R. A.

Highly Superheated Steam or Vapours, Improved Method and Apparatus for Producing. D. Adorján, Budapest, Hungary. Eng. Pat. 6377, March 23, 1896.

The heating gases are conducted between a system of continuous tubes, through which the steam or vapour is passed in a direction opposite to the direction of the gases. The tubes are tapered or reduced in diameter from the place at which the steam or vapour enters from the generator to the place at which it leaves the tubes, this construction giving a greater area of passage to the heating gases at the place where they enter the system of tubing. The heating gases are deflected by plates disposed between the tubes, to distribute the gases uniformly, and heat-conducting bodies may also be placed within the tubes to transmit the heat to the steam or vapour. A draft regulator, automatically adjusted by means of a "cataract," is employed to regulate the passage of the heating gases in exact proportion to the quantity of steam or vapour passing through the tubes.—R. A.

Oil-Presses, Filter-Presses, and the like; Impts. in or relating to Cloths for. A. J. Boult, 111, Hatton Garden, Middlesex. From H. W. J. Van Otterbeek Bastiaans, Cleve, Germany. Eng. Pat. 7718, April 11, 1896.

To avoid unequal wear, the cloths employed are in the form of folded or flattened tubes, which, after one or more operations in the press, are refolded so that the parts of the opposed sides occupy new relative positions. The cloths may be used as ordinary flat fabrics, or their edges may be thickened by folding them.—R. A.

Asbestos [Filtering, &c.] Fabric, Impts. in and relating to. J. Germain, L. Boisne, and H. Boisne, Condé-sur-Noireau, Calvados, France. Eng. Pat. 8078, April 16, 1896.

The warp or weft threads of the fabric, or both, are each formed of a core of one or more twisted asbestos threads, around which a number of strands or slubs of untwisted or slightly twisted asbestos are wound. After the weaving operation, the untwisted or slightly twisted fibres or strands adhere to one another, so that no interstitial spaces or openings are left through which liquids or gases can escape unfiltered.—R. A.

Fluid Extracts and Solutions, Impts. in the Method of and Means for Preparing. J. Longshaw, Burlington Street, Liverpool. Eng. Pat. 9085, April 30, 1896.

The material is placed in one or more tanks, in which it is treated with the extracting liquid or solvent. The liquid is withdrawn from the tanks through a steam heater, and again returned, this process being continued until the required degree of strength is obtained. A pump may be employed to effect the circulation of the liquid.—R. A.

Evaporating Apparatus, Impts. in and relating to, chiefly designed for obtaining Salt from Brine. W. Shedlock, Clapham, Surrey. Eng. Pat. 7997, April 15, 1896.

Apparatus is claimed of the kind described in the Specification of Eng. Pat. 7796 of 1896. The brine or other liquid is fed into one or more externally heated tubes or troughs, which extend through the steam space of a steam generator, and the salt or other deposited substance is automatically and continuously conveyed (by screw conveyors) from the said troughs into a chamber or casing connected therewith, from which it is elevated by an additional conveyor, through an inclined casing, and is discharged at a higher level than that of the liquid in the tubes or troughs.—R. A.

Metallic Surfaces, A Process for Protecting, against Chemical and Atmospherical Action. B. Politzer, Vienna. Eng. Pat. 10,864, May 19, 1896.

The metallic surface is coated with a thin layer of a drying oil or mixture of drying oils to which a siccative has been added, and when thus coated is exposed to a temperature of from 200° to 400° C. for from half an hour to one hour. The adhesion of the drying oil to the surface is increased by the addition of a little resin, before the heat is applied.
—C. A. M.

Furnaces, Impts. in. J. Wagner, Paris. Eng. Pat. 16,061, July 20, 1896.

Air is distributed to the under side of the furnace grate by a series of louvres, movable on a horizontal axis, and jointed at the ends of the rods. By this arrangement the rate of combustion is regulated by acting upon the admission of air. The furnace is provided with side combustion chambers above the grate and fuel bed. The walls are furnished internally with flues for the circulation of air, opening into the furnace through holes in a specially prepared perforated brick. The fireclay blocks also have angles, which cause the hot air to produce whirls, whereby a perfect mixture of the gases is obtained. The furnace is provided with two bridges at a distance apart, the first permitting the entrance of hot air by the side flues to the bed, the two bridges forming really a combustion chamber in the furnace. Mechanical raking is performed; it is based on the imparting of a series of shocks or movements to the grate. The mouth of a blower or ventilator, or the blast pipe of a steam injector, is arranged in the ash pit. To delay the flame and the gaseous current, and to change their direction, thereby allowing for the deposition of ashes, there is arranged a series of flame directors, fixed at a certain distance behind the bridge and distant from one another by a space proportional to the size of the furnace. Air is supplied by side flues in the fire blocks or the lateral masonry. The combustion chambers behind the bridge are closed by movable louvres working on an axis, to allow removal of cinders without interruption.—R. S.

Earthenware Still-Worm Pipes, Impts. in. E. Edwards, London. From L. Rohrmann, Krauschwitz, near Muskau, Germany. Eng. Pat. 29,345, Dec. 21, 1896.

In order to make framing or other supports unnecessary the separate pipes are provided with projecting parts, in which openings or passages are formed for the circulation of the cooling water, the pipes being laid helically, directly one above the other, the interior of the pipes being strengthened,

if necessary, by ribs or stays. A bottom stand or support, the inclination of which is made to correspond with that of the worm pipes, and provided with openings, serves to carry the entire apparatus.—E. S.

Millstones and Milling-Rollers, Impts. in Artificial. S. Theiner, Pilsen, Bohemia. Eng. Pat 3458, Feb. 9, 1897.

The inventor claims the use of carborundum along with material such as quartz, &c., as an ingredient of artificial millstones.—V. C.

Retorts for the Distillation of Shale and other Bituminous Substances, or for the Calcining of Ironstone, Lime, or other Substances; also applicable to Gas Producers; Impts. in or relating to. J. Bryson, Pumpherston, Midlothian. Eng. Pat. 4249, Feb. 17, 1897.

In retorts of the kind mentioned above, each retort or pair of retorts is provided with one lower hopper leading to a discharge door, the door being operated by one of four methods illustrated: (1) a chain and hydraulic or other ram direct; (2) a chain and hand-lever; (3) a chain, spur gearing, and hand-wheel; and (4) a chain and ram, and a horizontal shaft connecting the two.—R. S.

Compressed or Liquefied Gas [Carbonic Acid Gas, &c.], Impts. in Capsules for Containing, and Apparatus for Charging them. T. A. Calderwood, Canonbury, London. Eng. Pat. 5008, Feb. 24, 1897.

One end of the capsule is formed with a cylindrical mouth, which is closed by a plug, and the other end is formed with a hole provided with a check valve through which the capsule can be charged and emptied. In charging the capsule, it is pressed lengthwise between two dies, the lower die being formed with two valve-controlled ducts, one to supply the gas to the capsule, and the other to permit the escape of superfluous gas, &c. after the capsule is charged. —R. A.

II.—FUEL, GAS, AND LIGHT.

Gases from Coke Furnaces, Treatment of. Stahl u. Eisen, 1896, **17**, 90; Chem. Zeit. **21**, 38.

Separation of Cyanogen. — The gases, previously freed from ammonia, are passed through scrubbers containing a solution of soda or potash, in which is suspended carbonate or oxide of iron. A ferrocyanide is formed, which may be purified by crystallisation. The amount of cyanogen formed depends on the temperature of the furnace. Thus, at 800—900°, 1 cb. m. of gas gave 8 grms. of sodium ferrocyanide, whilst at 950°, and above this, 56—95 grms. were obtained.

The Use of the above Gas for Lighting Purposes.—The gas itself has only half the illuminating power of ordinary coal-gas, but it can be easily enriched with benzol. The lighting power of the gas may, by this means, be raised to that of 16 candles.

The Application of Benzol for Enriching Coal-Gas.—The benzol may be used for enriching inferior coal-gas. Judiciously admixed, it does not condense in the gas pipes, even in very cold weather. At present, about 5 per cent. of the coal-gas produced in Germany is thus enriched. —W. P. S.

Coal-Gas, The Use of "Manjak" [Barbados Bituminous Pitch] in the Manufacture of. J. H. Pye. J. Gas Lighting, 1897, **69**, 882.

"Manjak" is a natural bituminous pitch, and is found in Barbados, very near and sometimes upon the surface of the ground, in seams varying from 1 to 2 feet in thickness, running usually at an angle of about 45°, and in close proximity to rocks. Petroleum oil occurs in abundance in the same localities, and is often seen oozing out of the ground or floating down the streams. "Manjak" is black, has a high lustre and a bright conchoidal fracture, in appearance closely resembling newly broken pitch. It gave on analysis: moisture, 2·00 per cent.; volatile organic matter, 70·85 per cent.; non-volatile organic matter, 26·97 per cent.; and mineral matter, 0·18 per cent. "Manjak" has, under different names, been used in the insulation of electric wires, in the manufacture of varnishes, a[illegible] concrete in asphalt roads and pavements, and as patent fuel when mixed with peat or other organic matter.

The gas-producing power of "manjak" was compared with that of Wigan Arley coal, which latter, owing to the defective state of the plant, gave only 9,000 cb. ft. of 17 candle-power gas per ton. The "manjak" was carefully hand-picked so as to remove all stuff and dust, and preserve the stokers from the intense heat produced when charging with very fine material, and small charges of 80 lb. each were carbonised for 6 hours, before being completely burned off. The gas-holder was practically emptied before commencing the test, and six tests of the illuminating power of the gas at the outlet of the holder were made during the day, their average being taken. The coke was water-slaked and dried before weighing. The tar and liquor were not measured, but a larger quantity of tar was apparently formed than was the case with coal. The rapidity with which the gas was evolved during the first three hours of the charge, necessitated regulating the exhauster to draw $\frac{3}{4}$-in. vacuum, instead of working with a level gauge as usual, and this caused a slight suction of air through the leaky condensers, which somewhat reduced the illuminating power of the gas. One ton of "manjak" yielded 13,324 cb. ft. of 43·85 candle-power gas, and 9 cwt. of friable and porous coke.

The author considers that "manjak" is eminently suitable as an enricher of coal-gas, since, when well mixed in small quantities with the retort charges, it is very easily handled by the retort men.—A. S.

Monazite Sands, Examination of the Earths contained in. Schützenberger and Boudouard. Comptes Rend. 1897, **124**, 481—486.

By treating monazite sands with sulphuric acid, and precipitating the earths of the cerium group as double sulphates by the addition of potassium sulphate and further purification, the authors have obtained:—

(1.) A cerium metal having an atomic weight of about 138, the solution of which is not precipitated by copper oxide.

(2.) A substance which can be precipitated by copper oxide and by sodium sulphate, with an atomic weight of about 148.

(3.) A substance of atomic weight, 157, the sulphate of which is precipitated by copper oxide, but not by sodium sulphate.—J. S.

Thorium Nitrate, Crystallised. O. Fuhse. Zeits. angew. Chem. 1897, 115—117.

Thorium salts, as is well known, form the principal material for impregnating mantles for incandescent gas lighting. The nitrate is the most suitable salt for this purpose, because it is easily soluble, and is converted into oxide without producing a troublesome amount of smoke. As sent out from the chemical works it is known as "calcined" thorium, and forms white fragments, obtained by drying an aqueous solution of the nitrate. In this condition it contains 48 to 50 per cent. of oxide, the anhydrous nitrate, $Th(NO_3)_4$, representing 55 per cent. Thorium nitrate decomposes on evaporation, so that it is not concentrated beyond the point where a nitrate of the above oxide content results, and which still dissolves easily in water to a clear solution.

With regard to this behaviour the question arises, Why is not crystallised thorium nitrate produced, which, like every crystalline salt, would offer a certain guarantee of purity and uniformity in percentage of oxide? Earlier authorities considered thorium nitrate as uncrystallisable, or as crystallisable with extreme difficulty. Clève, in 1871, prepared large, well-formed crystals in the form of six-sided plates, containing 12 mols. of water of crystallisation—

$$ThO_2.2N_2O_5 + 12H_2O.$$

These, however, quickly lose 18 per cent. of water, or about 8 mols. of water of crystallisation, when dried over sulphuric acid. But if an aqueous solution of the nitrate be evaporated and allowed to crystallise hot, large, well-formed crystals are easily obtained, essentially different

from those of Clève. The salt contains 6 mols. of water of crystallisation, loses none of this over sulphuric acid, and, when well dried, is only slightly hygroscopic. Its formula is $ThO_2.2N_2O_5+6H_2O$, and the crystals belong to the quadratic system.—L. J. de W.

Free Nitrogen in Purified Coal-Gas, Apparatus for the Determination of the. G. Arth. Bull. Soc. Chim. 1897, **17**, 427.

See under XXIII., *page* 470.

Thoria, Behaviour of, with Oxalic Acid and Ammonium Oxalate, and the Estimation of. C. Glaser. Zeits. anal. Chem. **36**, 213.

See under XXIII., *page* 468.

PATENTS.

Illuminating Gas [Acetylene, &c.], Impts. in the Manufacture or Production of. V. B. Lewes, Greenwich. Eng. Pat. 6922, March 30, 1896.

To obtain illuminating gas containing acetylene, which can be used in ordinary burners without smoking, mixed carbides are manufactured in an electric furnace, using such compounds in their production as will by double decomposition give rise to a high temperature, whereby the carbides are produced with less expenditure of electrical energy than heretofore. Such metals are chosen that the carbides formed can be decomposed by the action of water, liberating a mixture of acetylene and other hydrocarbon gases. For example, 261 parts of manganese peroxide, 56 parts of lime, and 72 parts of carbon are mixed and heated in the electric furnace in the ordinary way. The oxygen liberated from the oxide of manganese burns violently with some of the carbon, the heat of the combustion, together with the heat of the electric arc, producing a fused mass of the carbides of calcium and manganese. On treating this substance with water, the gas evolved, consists of acetylene diluted with methane and hydrogen, which can be burnt with very great illuminating effect and without smoking.—H. B.

Acetylene Gas for Illuminating Purposes, Impts. in Apparatus for the Production and Storage of. E. Clausolles, Barcelona, Spain. Eng. Pat. 11,737, May 29, 1896. (Under International Convention, Jan. 24, 1896.)

The admission of water to a receiver containing carbide of calcium is controlled by the rise and fall of the bell of a gas-holder, which automatically moves a cock on the water-supply pipe. The gas-holder may be a storage reservoir, or may merely receive gas temporarily when the production exceeds the consumption. When the bell has fallen a certain distance, it closes the circuit of an electrical signalling apparatus, and thus the attendant is notified that recharging is necessary.—J. A. B.

Acetylene, Impts. in or relating to the Generation and Storage of. R. R. Gibbs, Egremont. Eng. Pat. 12,788, June 10, 1896.

Within a gasometer bell floating in a tank is a perforated diaphragm for the support of carbide of calcium. A thick layer of oil on the water in the tank is provided, with the object of preventing moisture acting on the carbide when the latter is drawn up by the rise of the bell from the tank. A pipe from the base of the tank telescopes within a wider pipe attached to the crown of the bell. The latter pipe is perforated near the gasometer crown, and thus the acetylene generated may be withdrawn from, or other gas for admixture admitted to, the gasometer by either pipe. Fresh supplies of carbide are admitted through a sliding chamber with mercurial seal attached to the crown of the gasometer, or by hand through a siphon filled with oil.—J. A. B.

Acetylene Gas Generators, Impts. in. D. Whalley and J. Hacking, Blackburn. Eng. Pat. 15,654, July 15, 1896.

The generator is preferably a cylindrical metal vessel with a detachable lid secured tightly thereto by a screw or clamp. Within the upper part of the generator is a perforated metal receptacle for carbide. Water-inlets with separate taps are provided at three different levels, and a gauge shows the height of the water in the generator. The gas passes from near the top of the latter through a cooler to the burners or a gasometer.—J. A. B.

Acetylene, Impts. in and in Apparatus for the Production of. G. S. T. Gastine, Marseilles. Eng. Pat. 20,529, Sept. 16, 1896.

The apparatus comprises a generator or generators and a bell gasometer. Each generator consists of a vertical tank, with discharge cock at its base, in which is supported a perforated basket containing calcium carbide. A bell-shaped cover, having a protruding spout near its rim, passes over the basket. Beneath the dome of the cover is the mouth of a pipe which rises vertically through the generator. When the tank is filled with water, the gas generated collects under the cover and normally passes by this pipe to the gasometer, whence the burners are fed. A vertical groove allows the passage of the spout when the cover is being affixed or removed; by turning the cover when it is in the lowermost position about its vertical axis, the spout is brought beneath an overflow chamber, and at the same time retains the cover in that position. The movement of the gasometer bell is made to operate the spindle of a tap on the pipe which conveys the gas from the generator to the gasometer in such a manner that the tap closes when the bell is charged to about one-fifth of its total capacity. The pressure of the gas then accumulating in the generator depresses the level of the water below the carbide, but gas continues to be slowly evolved through the action of the aqueous vapour, and further depresses the water until the spout becomes unsealed and the gas escapes into the overflow chamber. A pipe from the top of the latter conveys it to the gasometer.
—J. A. B.

Acetylene Gas, Impts. in Apparatus for Producing and Storing. G. Trouvé, Paris. Eng. Pat. 23,591, Oct. 23, 1896. (Under International Convention.)

This relates to improvements in the apparatus described in Eng. Pat. 23,521, 1895 (this Journal, 1896, 892). In the generator the stopper is dispensed with and the vessel permanently closed by a heavy lead top, and the wire cage is supported by a stand at a convenient height, instead of being suspended. The vessel is now provided with a removable bottom. In place of a condenser, for cooling and drying the gas prior to consumption, it is now stated that a more favourable result is obtained by coupling together two gas-holders which are supplied alternately by one or more generators. One holder delivers while the other is filling. Suitable taps and connections between the generators and holders are described. The whole apparatus is arranged so that the gas is under the pressure of a very small (10 to 30 cm.) head of water.—J. A. B.

Acetylene Gas, Improved Apparatus for Producing and Utilising. G. Trouvé, Paris. Eng. Pat. 23,592, Oct. 23, 1896. (Under International Convention.)

This, like the preceding, relates to improvements in the apparatus described in Eng. Pat. 23,521, 1895. They comprise a condenser, packed with glass beads or absorbent material, attached to the top of the generator, and a safety siphon tube, by which, when the pressure in the generator rises, the excess gas passes direct to the gas-outlet pipe, or to a point above a burner, instead of escaping into the atmosphere.—J. A. B.

[Acetylene.] Generation of Gas, Impts. in or relating to Apparatus for the. A. J. Boult, London. From H. F. Fuller, Chicago. Eng. Pat. 1440, Jan. 19, 1897.

Within an outer tank, containing water, is a bell, supported by counter-weights and guide-rods. In the lower part of the tank is a cylindrical chamber surrounded by the water and fitted internally with diaphragms so arranged that the gas entering at one point pursues a tortuous course before passing out at the opposite point, the object being to cool it and thus condense any water present. The gas passes from

the upper part of the bell by a pipe, which passes downwards and telescopes over a stand pipe which rises from the bottom of the tank. The stand pipe being in communication with the condensing chamber, the gas from the bell can always pass into the condensing chamber whatever the height of the bell. The calcium carbide holder is fixed in the upper part of the bell. Three forms of holder are described, the object of each being to bring a single isolated portion of the charge into contact with the water at a time. In each form, as the bell descends, fresh portions of carbide come into contact with the water as required. The generator is fitted with a safety vent.—H. B.

Acetylene Gas, Improved Apparatus for the Production and Storage of. R. F. Carter, Niagara Falls, Canada. Eng. Pat. 2284, Jan. 28, 1897.

The generator consists of a cylindrical bell, the base of which stands in a water lute. Within the bell is a cylindrical carbide receptacle centrally divided by a perforated diaphragm, preferably of hemispherical form. The carbide is put in the space above the diaphragm, and the lime formed falls through the latter. The carbide receptacle is provided with an agitator worked by a crank outside the generator. The water is sprayed on to the carbide from a pipe fed through a funnel and siphon. The gas passes from the generator to a bell gas-holder, whence the burners are supplied. A lever, attached at one end to the gas-holder bell and at the other to a cord connected to a valve spindle in a water receptacle, controls the supply of water thence to the funnel mentioned above, by the rise and fall of the bell. The water receptacle, of which the capacity is regulated by the position of a movable overflow pipe, is filled from a cistern through a cock actuated by a float within the receptacle. The water flows very slowly through this cock, but escapes rapidly through the exit valve when it is open, and thus the water receptacle serves as an automatic measuring and feeding apparatus, which limits the amount of water admitted each time to the generator.—J. A. B.

Carbides, Process for the Agglomeration of. G. de R. de Sales, Lyons. Eng. Pat. 1653, Jan. 21, 1897.

So-called "cartridges" are formed by filling carbides into cylindrical cases made of cardboard, paper, or metal, which are provided, if desired, with a longitudinal slot or slots. The interstices are filled up with tar, bitumen, or sand, according to the rapidity of action required when generating gas. The ends of the cartridges and the slits are covered, till required for use, by impervious paper or waterproof fabric.—R. S.

Fire-lighters, Impts. in. P. R. de F. d'Humy, Liverpool. Eng. Pat. 5795, March 16, 1896.

The fire-lighters are formed principally of fine shavings, or sawdust, which is first immersed in a caustic soda solution, and, after drying, is plunged into petroleum or other mineral oil. It is afterwards again dried, pressed into a mass, and then reduced to powder. A second powder is formed by grinding the product obtained by mixing and heating together powdered resin, fine shavings or sawdust, and an oxidiser, as saltpetre, sodium nitrate, or other nitrate or chlorate. The first powder is mixed with the second and heated, a little dust of charcoal and lime being added. The material is afterwards moulded under pressure.—R. S.

Chambers for the Combustion of Gases [Waste and Blast-Furnace Gases, &c.], Impts. in or relating to. J. Hartmann, Paris. Eng. Pat. 8679, April 24, 1896.

The apparatus, serving for heating steam-generators or other furnaces, by the combustion of waste gases from blast furnaces or the like, is composed of three, or sometimes of two, chambers so arranged as to obtain at least one reversal of the flame. To avoid loss of heat by radiation, all the walls which are externally in contact with the atmosphere, are entirely hollow or provided with flues in which the air supply for the combustion of the gas circulates. A pressure gauge is fixed at the point where the gas enters the chamber, indicating the pressure of the gas, and to this pressure correspond certain bars and numbers marked upon the valves of the air- and gas-supply pipes, in such a manner that when the valves are opened to these bars, the proper proportions of air and gas flow into the chamber.—H. B.

[Hydrocarbon Mist.] Gas for Illuminating and other Purposes, Impts. in and relating to the Treatment of. C. E. and C. F. Botley, Hastings. Eng. Pat. 9316, May 2, 1896.

To prevent the formation of naphthalene and other deposits from the gas and to increase its illuminating power, it is passed through a horizontal cylindrical metallic vessel, at one end of which are atomisers producing a mist of any suitable liquid hydrocarbon, from 0·680 to 0·980 sp. gr. The atomisers may be operated by either compressed air, gas, or superheated steam, and each consists of a tubular body provided with inlet branches, one of which communicates with a smaller tube arranged within, to convey the oil, whilst the other communicates with the annular space between the said tube and the outer body, and forms a passage for the compressed air, &c. The combined oil and air, &c., then enter an expansion chamber and finally pass through a nozzle covered with a perforated plate to complete the "atomisation" of the oil. "The gas will thus carry a certain amount of oil in mechanical suspension."—H. B.

Pure Hydrogen Gas, Impts. in the Production of. L. V. Pratis and P. Marengo, Turin, Italy. Eng. Pat. 16,277, July 22, 1896.

Hydrogen gas is obtained by acting upon iron filings and water by gradual additions of sulphuric acid of 50° B., equal parts by weight being taken of each. The hydrogen produced is conducted first through water, then through solution of a lead salt, then through a device containing diaphragms of wire gauze, to a gasometer, whence the gas traverses an insulating water valve, an elastic chamber, and a second device similar to the first, when it is taken by branch pipes to the place of utilisation. The arrangements described, permit of the gas being produced under considerable pressure.—E. S.

Combustible Gas, including Illuminating Gas; Impts. in and in connection with the Manufacture of, and in Apparatus or Plant for the Purpose. J. C. Mewburn, London. From F. G. Bates, Philadelphia. Eng. Pat. 26,261, Nov. 20, 1896.

The 15 pages of text, with 26 claims and 23 drawings, describe improvements upon Lorois's Eng. Pat. 16,413 of 1892. For producing combustible gas, heated air and superheated steam, either or both, carburetted with hydrocarbon vapours, are passed through a producer containing incandescent fuel, wherein combustion is maintained under a pressure of over 42 lb. per square inch. For producing illuminating gas the pressure is maintained at over 50 lb. per square inch, whereby the hydrocarbons are partly oxidised and partly fixed as illuminants; and for gas of high illuminating power the proportion of hydrocarbon is increased, and the pressure is maintained at about 100 lb. per square inch. In one modification for making combustible gas, the incandescent fuel in the producer is dispensed with. Means are described for utilising the heat of the waste and other gases for heating the boiler feed-water, injecting the same into the boiler, for heating the compressed air and for utilising the heat disengaged by the compression of the air, &c.—H. B.

Water-Gas, Impts. in the Production of, and in Apparatus therefor. C. Dellwik, Stockholm. Eng. Pat. 29,863, Dec. 28, 1896.

During the air-supply period, such a quantity of air is admitted to the fuel as to produce chiefly carbon dioxide instead of carbon monoxide, the combustion to carbon dioxide producing a greater amount of heat, and thus shortening the necessary air-supply period as compared with combustion to carbon monoxide, the patentee's object being to have as small a proportion as possible of producer gas in the gas finally obtained. In a suitable generator

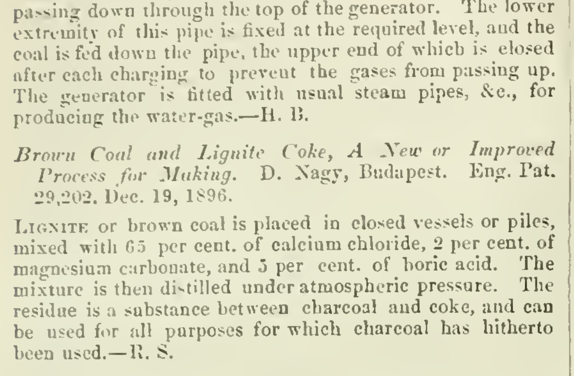
described and claimed, the air is supplied from an upright pipe into the fuel at various levels, and the fuel is kept at a definite level which a trial has shown to result in the production of the maximum of carbon dioxide. The height of the fuel is automatically kept at the required level by using a coal receptacle, consisting, say, of a cast-iron pipe passing down through the top of the generator. The lower extremity of this pipe is fixed at the required level, and the coal is fed down the pipe, the upper end of which is closed after each charging to prevent the gases from passing up. The generator is fitted with usual steam pipes, &c., for producing the water-gas.—H. B.

Brown Coal and Lignite Coke, A New or Improved Process for Making. D. Nagy, Budapest. Eng. Pat. 29,202, Dec. 19, 1896.

Lignite or brown coal is placed in closed vessels or piles, mixed with 65 per cent. of calcium chloride, 2 per cent. of magnesium carbonate, and 3 per cent. of boric acid. The mixture is then distilled under atmospheric pressure. The residue is a substance between charcoal and coke, and can be used for all purposes for which charcoal has hitherto been used.—R. S.

Combustion Products under Pressure, and utilising the same for Operating Heat Engines and for Propelling Ships; Impts. in and connected with Means for Generating. D. Cook, Queen Victoria Street, London. Eng. Pat. 6073, March 18, 1896.

The products are produced under pressure in a chamber A, leading to a receiver B, the whole of the chamber and receiver being lined with non-conducting material C, as a



thick lining of fireclay. Outside the plates is a space D, and then comes the outer shell. Air enters the space D at *a*, and passes to a ring of perforations L, leading to the combustion chamber. Fuel (as powdered coal) and air are forced in measured quantities into the chamber through an opening M, a special device for this purpose being illustrated. The fuel is ignited by an incandescent tube, or grid, or wire, or electric spark, or, as shown, by a tube E, capable of being rendered red hot by a blow-pipe flame, applied internally, and afterwards of being withdrawn. The hot products under pressure escape through an expansion nozzle lined with fireclay, and provided with a valve or valves.—R. S.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Benzene, Recovery from Coke-Oven Gas. C. Heinzerling. Chem. Rundschau, 1897, 82 and 102.

In the author's method of separating benzene from coke-oven gas by cooling the compressed gas by a counter current of that from the expansion cylinder, a temperature of $-70°$ to $-80°$ C. is obtained, the benzene being deposited in the condensing tubes, which are duplicated so that one set is always available while the other is being freed from the solid deposit. In an experimental installation in Upper Silesia, about 40,000 cb. m of gas were treated daily; they contained 20 grms. of benzene per cubic metre. For treating 60,000 cb. m. a pressure of about 240 H.P. is required, working day and night, to produce 1,200 kilos. of benzene. The working expenses may be reduced by utilising the excess of gas, over and above that required for heating the coke ovens, as a source of motive power in gas engines; and, since the excess from 60 Otto coke ovens is 24,000 cb. m. per diem, there is an ample margin for this purpose, only 3,600 cb. m. being needed daily for the amount of power in question. It is found more advantageous to dispense with an expansion cylinder in working the apparatus, and to allow the gas to expand in a chamber instead, the increased power requisite being obtained from the waste gas, without much expense.

Before the coke-oven gas is compressed, it is advisable to dry it by passing it through a Rohrmann-Lunge sulphuric acid tower, or by means of calcium chloride, to prevent the formation of ice in the tubes, economise power, and enable the residual ammonia to be recovered. Filtration for the removal of traces of coke-dust is also expedient.

For estimating the benzene content of the gas, a modification of Deville's method, by substituting liquid carbon dioxide for methyl chloride in the final cooling, is recommended. A simpler but less accurate method is to absorb the benzene by passing the gas through cumene in a cooling mixture, distilling off the fractions below 140°—145° C., and submitting the distillate to fractional distillation.—C. S.

Asphalt and Petroleum, The Crédo Deposit of. Boulangier. Comptes Rend. Mens. de la Soc. de l'Ind. Minérale, 1896, 14.

The deposit recently discovered at Crédo, which lies midway between two important asphalt mines—that of Seyssel, in France, and that of Val-de-Travers, in Switzerland,—contains beds of sand impregnated with a viscous petroleum, rich in paraffin, running from the sand as soon as the beds are cut and flowing at and from an altitude of 2,952 ft. Asphalt has also just been discovered.—A. S.

Paraffin, Estimation of, in Distillates of High Boiling Point from Brown Coal Tar and Crude Petroleum. D. Holde. Zeits. angew. Chem. 1897, 116.

See under XXIII., *page* 471.

IV.—COLOURING MATTERS AND DYES.

Indigo Salt, Action of Light on Kalle's. E. Kopp. Bull. Soc. Ind. Mulhouse, 1897, 87—89.

In some of the early dyeing experiments with Kalle's indigo salt, o-nitrophenyl-lactylmethylketone—

$$C_6H_4(NO_2).CH(OH).CH_2.CO.CH_3,$$

it was noticed that the goods had a patchy appearance after developing in caustic soda, which was found to be due to the action of light previous to development. The cotton was printed with the following mixture: 100 c.c. of gum-water, 1 grm. of sodium carbonate, 29 (4 + 25) c.c. of water, 7·5 grms. of indigo salt, and 10 c.c. of sodium bisulphite solution (40° B.), and then passed through a caustic soda bath (15° B.) at 62° C., washed, soaped, and dried. On exposing samples to sunlight it was noticed that after 2 minutes a distinct effect was produced, whilst in 5 minutes the shade was reduced to one-half. The yellow rays of the spectrum produced no effect, whilst the action gradually increased from the green, through red, to blue. This method has been applied for obtaining photographic prints by employing the solution as above with three times the quantity of gum-water, for coating a suitable medium and drying the plates in the dark. The exposure varies from 1 to 12 hours, the image appearing in yellow and brown shades, and is then developed. It would appear that light effects a partial decomposition of the indigo salt. Indigo salt was formerly sold in the form of its bisulphite compound, but owing to the readiness with which it decomposes, even in diffused light, it is now supplied as the free ketone.
—T. A. L.

Diphthalylethene, An Analogue of Indigo in the Indene Series. V. Kaufmann. Ber. **30**, 382—387.

In the course of a research on the cause of colour in organic substances, the author has prepared an analogue of indigo, in which the imido groups are replaced by carbonyl groups. The action of potash and hydrogen peroxide on diketohydrindene (indandion)—

$$C_6H_4 \left\langle \begin{matrix} CO \\ CO \end{matrix} \right\rangle CH_2$$

(obtained by the action of sodium on a mixture of phthalic acid diethyl ether and acetic ether, and decomposition of the product with dilute sulphuric acid. Wislicenus and Kötzle, Annalen, **252**, 72), gives, according to the conditions of the experiment, four well characterised substances:—

1. A green substance, which is the potassium salt of the analogue of indigo white, and having the following constitution:—

$$C_6H_4 \left\langle \begin{matrix} CO \\ CO \end{matrix} \right\rangle CK.CH \left\langle \begin{matrix} CO \\ CO \end{matrix} \right\rangle C_6H_4$$

2. A red substance, which is also obtained by the action of sodium or molecular silver on dibromodiketohydrindene in benzene or toluene, which is the analogue of indigo, has the formula—

$$C_6H_4 \left\langle \begin{matrix} CO \\ CO \end{matrix} \right\rangle C:C \left\langle \begin{matrix} CO \\ CO \end{matrix} \right\rangle C_6H_4$$

and is termed diphthalylethylene or indenigo.

3. A brownish-yellow substance, triketohydrindene (indantrion), probably—

$$C_6H_4 \left\langle \begin{matrix} CO \\ CO \end{matrix} \right\rangle CO$$

4. An acid, which is the final product of the oxidation, and apparently identical with—

$$1.2.\ C_6H_4(CO_2H)COCO_2H.$$

The first of these, termed diphthalylethane, is in many respects similar to indigo as regards its solubility in aniline and sulphuric acid, its stability, and faculty of sublimation. Since, however, its alkali salts are practically insoluble in water, it is, unlike indigo, unsuitable for employment in the reducing vat, and hitherto it has not been possible to obtain in quantity a soluble sulphonic acid of diphthalylethylene. From a boiling bath, the alkali salts of this latter, dye animal fibres a greenish shade. For the preparation of the potassium salt of diphthalylethane, 5 grms. of diketohydrindene and 2·25 grms. of potash are dissolved in 50 c.c. of 0·5 to 0·75 per cent. hydrogen peroxide, and heated for half an hour on the water-bath. The precipitate is washed with dilute potash and extracted with alcohol, which, on concentration, deposits the potassium salt as a greenish powder. On boiling with glacial acetic acid, diphthalylethane is obtained as an amorphous yellow powder, which crystallises from alcohol or aniline in microscopic needles, melting at over 200° C. The best yield of indenigo (about 10 per cent.) was obtained by dissolving 5 grms. of diketohydrindene with 2·2 grms. of potash in 50 c.c. of water, and gradually adding 11·25 grms. of 80 per cent. potassium persulphate, neutralised with ammonia. The sulphuric acid formed is gradually neutralised with the calculated amount of potash, and the precipitate is washed with hot dilute potash. The product is extracted with glacial acetic acid, and crystallised from aniline, forming red needles, which sublime with partial decomposition, and form a sparingly soluble sulphonic acid. Indantrion is obtained, together with diphthalylethylene, when the oxidation is effected with 3—3·5 per cent. hydrogen peroxide. It is dissolved out with glacial acetic acid, forms brownish-yellow plates melting at 190°—206° C., and is insoluble in dilute acids and alkalis.—T. A. L.

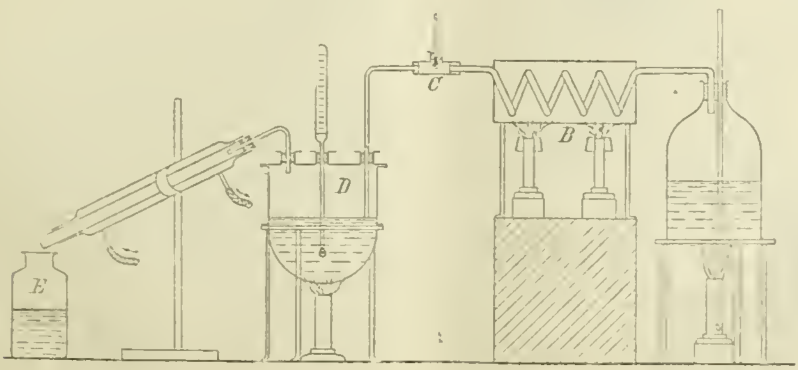
Nitronaphthalene, α-Naphthylamine, p-Phenylene Diamine, and Phenylene Blue, Preparation of. L. Paul. Zeits. f. angew. Chem. 1897, 145—152.

The author describes a number of experiments for the preparation of the various products mentioned, the following quantities giving the best results:—

Nitronaphthalene.— 1·5 kilos. of naphthalene are gradually stirred into a mixture of 1·1 kilos. of nitric acid (43° B.) and the same weight of sulphuric acid (66° B.) at a temperature below 45°—50° C. Towards the end of the operation the temperature is allowed to rise for about half an hour to 50° C. The nitronaphthalene is then allowed to solidify, separated from the acid, washed with hot water, and dried at 140° C. The yield was 1·91 kilos., and the product melted at 50°·25 C. Although no appreciable quantity of naphthalene was obtained on steaming, the solidifying point after this treatment rose to 50°·5 C. For further purification, 2·73 kilos. of the crude by-product were dissolved in 0·68 kilos. of solvent naphtha, filtered, and cooled to 30° C. About half the nitronaphthalene crystallises out and is filter-pressed, the press cakes being steamed to remove the last traces of naphtha. The nitronaphthalene, after drying at 130°—140° C., solidifies at 54°·25 C. and weighs 1·17 kilos. If required still purer, 100 grms. of this product are recrystallised from 500—750 c.c. of alcohol, when the solidifying point rises to 55°·75 C.

α-Naphthylamine.—A cast-iron vessel which can be closed, provided with an agitator, and containing 800 grms. of ground iron borings, 40 grms. of hydrochloric acid (20°·5 B.), and 400 c.c. of water, is heated in an oil-bath to 70° C., when 656 grms. of nitronaphthalene (91·5 per cent., m.p. 50°·5 C.) are gradually added in portions of 20—25 grms. every 5 minutes, and stirred for 7—8 hours. The melt weighs 1·84 kilos.; half of it is neutralised with 50 grms. of slaked lime in an iron vessel and distilled with steam heated to 150°—265° C.

Fig. 1.



In Fig. 1, B is the superheater, and D represents the still containing the neutralised melt. The flame under D is so adjusted that the temperature of the thermometer is approximately the same as that in C. At the commencement, about 250 c.c. of water pass over: the temperature in D is then raised to 150° C., when α-naphthylamine begins to come over. The temperature gradually rises to 260°—265° C., the whole operation lasting 5 hours, and about 4 litres of water passing over, the quantity of α-naphthylamine varying from 406—418 grms. The product, after drying, is distilled *in vacuô* at a temperature from 240°—290° C., the largest fraction distilling at 248°—252° C. 389 grms. were obtained from 400 grms. of crude base, but no details are given as to the pressure.

p-Phenylene Diamine.—A mixture of 4·67 kilos. of amido-azobenzene hydrochloride (= 6·7 kilos. of a 90 per cent. paste), 13 litres of water, and 2 kilos. of spirit, is placed in a wrought-iron vessel provided with a water jacket and an agitator. The temperature is raised to about 60° C., and 3—5 kilos. of zinc dust in quantities of 75 grms. are added every 5 minutes. The melt is finally heated to 90° C., and maintained at this temperature for half an hour. One kilo. of sodium carbonate is then added, and by means of a current of steam, first the alcohol and then the aniline is blown off. The alcoholic distillate measures about 3 litres, has a sp. gr. 40° Tralles, and is used for the next operation. In order to recover the aniline, 25 litres are distilled off, and the aniline is salted out. The residue remaining in the reducing still consists of an aqueous solution of *p*-phenylene diamine (about 40—50 litres), which, after filtering from the zinc hydrate, is evaporated under reduced pressure in a current of carbonic acid to about 6 litres. The *p*-phenylene diamine crystallises out and is hydro-extracted. The yield is 2·0 kilos., equal to about 90 per cent. of the theoretical. The addition of 0·5 to 0·75 kilo. of hydrochloric acid to the mother-liquor gives about 150 grms. of *p*-phenylene diamine hydrochloride more. The base so obtained is sufficiently pure for the preparation of water-soluble indulines, but for further purification it is extracted with benzene. A diagrammatic representation of an apparatus for this purpose is given in the accompanying Fig. 2.

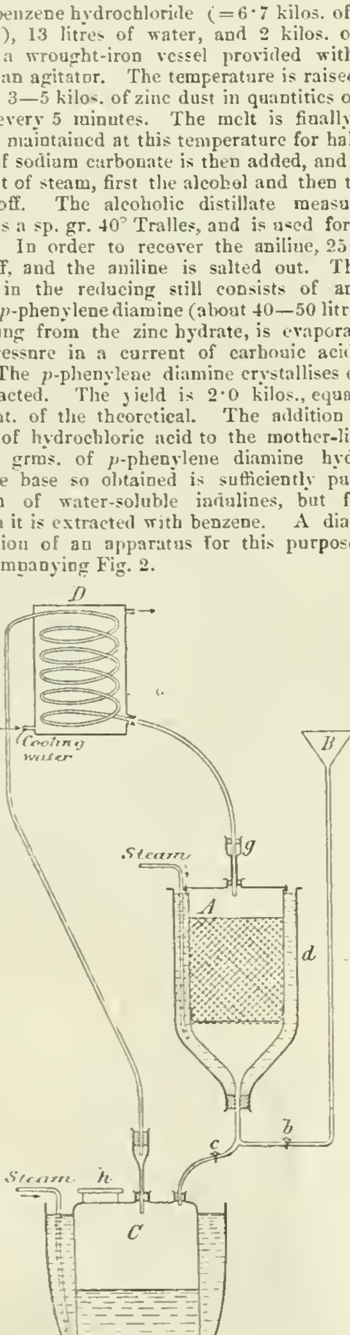


Fig 2

A is a jacketed, leaden, cylindrical vessel provided with a sieve plate covered with cloth. The vessel is filled about two-thirds with crude base and is then closed with a well-fitting cover by means of a red-lead and glycerin joint. The cock *c* being closed and *b* open, benzene is poured into the apparatus through B. After closing *b* the water in *d* is heated by means of the steam-pipe in order to bring the benzene to gentle ebullition. When the extraction has been carried on for a sufficient time, *c* is opened, and the benzene charged with *p*-phenylene diamine is allowed to flow into C, whence it is distilled through the condenser D into A, and the operation is again repeated. After several extractions, A is charged with fresh base, and in the end C contains pure *p*-phenylene diamine, which is removed through the man-hole *h*.

Phenylene Blue.—100 grms. of crude *p*-phenylene diamine and 35 grms. of its hydrochloride are heated to 150° C., and 36 grms. of α-nitronaphthalene are slowly added, the temperature during this operation being kept below 170° C. The melt is then heated to 180°—190° C. for three hours, when, after cooling, it is ground, dissolved in 4 litres of water and 200 grms. of hydrochloric acid, and precipitated with 5 kilos. of salt. The dyestuff so obtained weighs 150 grms., and is in no way different from commercial Paraphenylene blue. The mother-liquor contains about 15—18 grms. of a redder dyestuff, which can be separated by the addition of more salt or by caustic soda, but it is also contaminated with α-naphthylamine, due to reduction of the α-nitronaphthalene.—T. A. L.

Yellow Dyestuff derived from Dinitrofluoresceïn.
F. Reverdin. Bull. Soc. Chim. 1897, **17**, 416—419.

By the action of ammonia on dinitrofluoresceïn at the ordinary temperature, a yellow dyestuff is obtained which dyes wool in an acid bath a yellow shade, similar to that obtained with tartrazine. The following is the method of preparation:—To a paste of 100 grms. of dinitrofluoresceïn and 100 grms. of water, in a flask kept in ice-cold water, 75 c.c. of 21 per cent. ammonia are added. The mixture dissolves, and, after some hours, is changed into a solid mass. This is triturated with 125 c.c. of brine and filtered, and after well draining, the ammonium salt is converted into the acid by the addition of dilute hydrochloric acid. The acid is filtered, and digested with 10 parts of acetone to remove impurities; it is then converted for use into the easily soluble sodium salt.

On leading a current of hydrochloric acid gas into the boiling alcoholic solution of the acid, a yellow crystalline ether is formed, the sodium salt of which crystallises well.

The composition of the yellow dyestuff is probably represented by the formula—

$$C \begin{cases} C_6H_2(NO_2)OH \\ \quad > NH \\ C_6H_2(NO_2)OH \end{cases}$$
$$C_6H_4.CO.O$$

an oxygen atom of dinitrofluoresceïn being replaced by the imido group.

Caustic soda in excess does not change the yellowish-red solution of the new dyestuff, but turns the pure yellow solution of dinitrofluoresceïn to a deep violet. The effects obtained on wool with the new colour are distinguished from those given by dinitrofluoresceïn by their level shade and by their much greater fastness to light, which is, however, not quite equal to that of tartrazine.

A similar dyestuff is obtained by the action of aniline on dinitrofluoresceïn at 120° C., removal of excess of aniline by hydrochloric acid, solution in sodium carbonate, and salting out; this colour dyes wool reddish-yellow. The product of the action of ethylamine on dinitrofluoresceïn colours wool orange; the *p*-toluidine compound produces a brownish-yellow.

Similar dyestuffs have been obtained by the action of ammonia on other nitrofluoresceïns, such as the products of the nitration of dichloro- and tetrachloro-fluoresceïn,

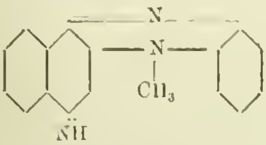
and the substance produced by fusing resorcinol with the nitrophthalic acid—

$C_6H_3(COOH)_2NO_2$ [COOH : COOH : NO_2 = 1:2:3]

—A. C. W.

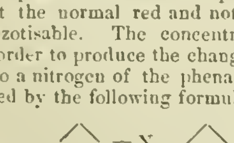
Azonium Bases, Relations of the, to the Safranines. O. Fischer and E. Hepp. Ber. **30**, 391—402.

THE ammonium compounds obtained by the action of methyl iodide on phenazine, naphthazine, or naphthophenazine (Ber. **26**, 179), are easily converted by the action of potash into other bases. Thus the iodomethylate of naphthophenazine yields *ms*-methyl rosindone. By the action of ammonia instead of potash, these addition products yield rosindulines, as Kehrmann (Ber. **29**, 2316; this Journal, 1896, 802) and Fischer and Hepp (Ber. **29**, 2752; this Journal, 1897, 132) have shown. The iodide of *ms*-methyl naphthophenazonium is obtained by heating naphthophenazine with an excess of methyl iodide to 100—120° C. The product is extracted with benzene, and is sparingly soluble in water. The chloride obtained by treating a warm alcoholic solution of the iodide with silver chloride is easily soluble, but can be salted out. When dissolved in 20 parts of 70 per cent. alcohol and treated with an excess of ammonia, it yields, after standing about two days and separation from some tar formed, dark red needles of *ms*-methyl rosinduline hydrochloride.

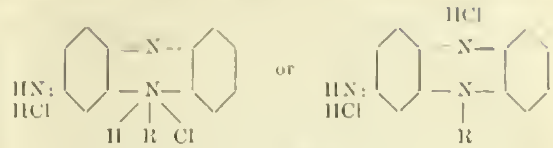


The aqueous solution is a fiery orange-red, and does not fluoresce, whilst the alcoholic solution exhibits a fine yellow fluorescence. The base is very similar to rosinduline, and dissolves with a greenish colour in sulphuric acid, which becomes yellowish-red on dilution. When heated with hydrochloric acid and acetic acid to 180° C. under pressure, or else with dilute sulphuric acid at 170° C., the product is converted into the corresponding indone, the imido group being replaced by oxygen. As already mentioned, the action of potash on an alcoholic solution of a salt of naphthophenazonium yields the same substance. It crystallises from dilute alcohol in golden yellow matted needles, and dissolves in concentrated sulphuric acid with a cherry-red colour and slight greenish-yellow dichroism, the solution on dilution becoming yellow. Continuing the discussion on the relations between the azonium bases and the safranines, and in reply to Kehrmann and Nietzki (Ber. **29**, 2316 and 2771; this Journal, 1896, 802, and 1897, 132), the authors point out that Kehrmann adopts the paraquinone formula for the safranine bases, and that whereas the anhydride of *m*-diazobenzoic acid is formed in an acid solution, Nietzki's proposed 5-ring anhydride is decomposed by acids and re-formed by alkalis.

With regard to the fact of the diazotisability of aposafranine, &c., the authors maintain that this does not affect their previous views, and they again refer to their remarks on the conversion of different nitroso bases, such as nitrosophenol, *p*-nitroso-aniline, and *p*-nitrosodiphenylamine, into diazophenol, diazo-aniline, and diazodiphenylamine. All these quinone imides are converted by the action of nitrous acid into another configuration, and a similar change would appear to take place in the case of aposafranine salts, that is to say, that the normal red and not the abnormal green salts are diazotisable. The concentrated sulphuric acid necessary in order to produce the change, causes the acid to attach itself to a nitrogen of the phenazine ring. The salts are represented by the following formulæ:—

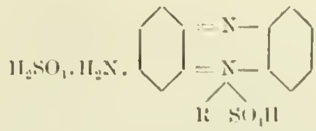


Red Salt.

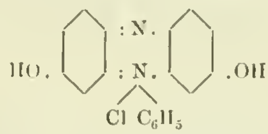


Green Salt.

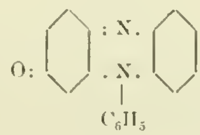
By the action of nitrous acid the substance assumes the *o*-quinonoid form—



and diazotisation follows. It appears more probable that a transformation such as this, should take place under the action of concentrated sulphuric acid and nitrous acid, rather than that this change should be brought about by cold water, which is in effect Kehrmann's view, the bases according to him being paraquinones and the salts *o*-quinones. Safranol hydrochloride, for instance, according to Kehrmann and Nietzki, has the formula—

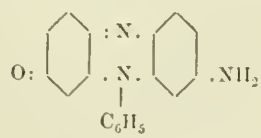


By washing with cold water it yields safranol, and it more probably has the constitution of a *p*-quinone like the base—



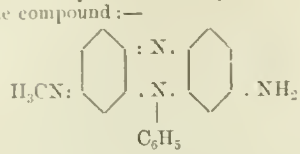
since, on heating with acetic anhydride under pressure, only a mono-acetyl derivative is obtained, which is also the case with safraninone. Further proofs are afforded by the behaviour of methylphenosafranine (from methyl-*p*-phenylene diamine + 2 anilines).

Safraninone.—This substance, having the formula—



has been obtained by the action of alcoholic potash on phenosafranine (Annalen, **286**, 211; this Journal, 1895, 797), and synthetically by Jaubert (Ber. **28**, 274; this Journal, 1895, 564). Neither method is satisfactory, but one giving better results, consists in heating 1 part of phenosafranine hydrochloride, 1 part of crystallised sodium acetate, and 100 parts of water under pressure to 150° C. The product forms bluish-green prisms, which dissolve to a red solution fluorescing brownish-yellow. The solubility of the base in water is noteworthy, as also its relatively high tinctorial value. The substance contains one primary amido group, which can be diazotised, and it also unites with aldehydes with liberation of water. When heated with dilute alkalis under pressure, it is converted, like phenosafranine, into safranol. Methylphenosafranine, obtained by oxidising *p*-methylphenylene diamine, together with two molecules of aniline, yields a hydrochloride crystallising in green prisms. The solutions are somewhat bluer than those of phenosafranine. Mineral acids cause a change to bluish-violet or blue. The solution in concentrated sulphuric acid is bluish-green, whilst the solutions fluoresce similarly to those of phenosafranine. The substance contains one primary amido group and can be diazotised. From reactions described, the authors conclude

that methyl phenosafranine only contains one nitrogen group capable of acetylation, and they ascribe the following formula to the compound :—



—T. A. L.

Azo Dyestuffs. Dynamical Investigations on the Formation of. H. Goldschmidt and A. Merz. Ber. **30**, 671—687.

With a view of studying the course of the reaction in the formation of azo dyestuffs, the authors have investigated the rate of formation of certain amido-azo and hydroxyazo dyestuffs under various conditions by combining diazobenzene sulphonic acid with dimethylaniline hydrochloride and with phenol, and have arrived at the following conclusions :—1. On combining the hydrochloride of a tertiary aromatic amine with diazobenzene sulphonic acid, the base, itself liberated by hydrolysis, combines with this latter. 2. An excess of hydrochloric acid diminishes the rate of combination. 3. The rate of combination is unaffected by the concentration of the solutions. 4. In the formation of hydroxyazo derivatives from phenols and diazo compounds, the reacting substances are the free phenol (liberated by hydrolysis) and the syn-diazo compound. 5. An excess of alkali diminishes the rate. 6. The time requisite for the combination increases with the concentration of the solutions.—T. A. L.

The "Shinia" Leaves of Cyprus. Imp. Inst. J., May, 155.

See under XXIV., *page* 474.

PATENTS.

New Colouring Matter [Violet-Blue] from Dinitronaphthalene, The Manufacture and Production of. J. Y. Johnson, London. From "The Badische Anilin and Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 7766, April 13, 1896.

According to Eng. Pat. 10,996 of 1893 (this Journal, 1894, 630), a dyestuff termed A, was obtained by reducing dinitronaphthalene with grape sugar. It had, however, the disadvantage of being very insoluble, and could only be used for dyeing purposes by producing it on the fibre. If, however, the dinitronaphthalene be reduced, in presence of a sulphite or a bisulphite, with grape or milk sugar, sodium sulphide or stannite, or with zinc dust, a dyestuff is obtained which is completely soluble in water and dyes unmordanted wool deep violet-blue shades. About 10 kilos. of finely powdered 1.1'-dinitronaphthalene, 10 kilos. of grape sugar, 20 kilos. of a sodium bisulphite solution (40 per cent. $NaHSO_3$), 20 kilos. of caustic soda lye (24 per cent. NaOH), and 800 litres of water, are heated for about 2 hours to 80 C. until no unaltered dinitronaphthalene can be detected. The solution, which is blue, is mixed with 30 kilos. of hydrochloric acid (32 per cent. HCl) and boiled so long as sulphurous acid gas is given off. The dyestuff is then precipitated with salt or potassium chloride, and after drying has the appearance of a violet-black powder with a metallic lustre, and is readily soluble in hot water.

—T. A. L.

Dyestuffs and Tanning Extracts, Impts. in and relating to the Manufacture of. H. H. Lake, London. From Lepetit, Dollfus, and Gansser, Milan and Susa (Piedmont), Italy. Eng. Pat. 8582, April 23, 1896.

The action of bisulphite or neutral sulphite of soda on certain vegetable colouring or tanning extracts gives new products having valuable properties. Thus, 300 kilos. of old fustic extract (28° B.) and 100 kilos. of sodium bisulphite solution (35° B.) are mixed and heated for 8 hours in an autoclave to 110°—115° C. A magma of microscopic crystals of so-called sulphomorine is thus obtained, which is sparingly soluble even in hot water. This dyestuff gives bright yellow shades on alum, chrome, and tin mordants, quite different from those given by ordinary fustic extract. Young fustic treated in the same way, but heated to 140°—150° C. gives a product dyeing very pure yellow shades. By heating 300 kilos. of quercitron bark extract (28° B.) with 100 kilos. of sodium bisulphite for 8 hours in a closed vessel to 140°—145° C., a product is obtained of much greater solubility than the original extract and giving much stronger shades on mordanted cotton. The products obtained from quebracho and hemlock extracts are perfectly soluble in cold water, and give a pink colour to hides similar to that produced by mimosa bark. These tanning extracts keep the leather perfectly soft, and act more quickly than the quebracho extracts generally used.—T. A. L.

Colouring Matters of the Rosinduline Series, Impts. in the Manufacture of. C. D. Abel, London. From "The Actien Gesellschaft für Anilin Fabrikation," Berlin, Germany. Eng. Pat. 8857, April 27, 1896.

Phenyl- or tolyl-rosinduline sulphonic acids are obtained by the action of aniline or *o*- or *p*-toluidine on the rosinduline derivatives described in Eng. Pat. 16,217 of 1895 (this Journal, 1896, 587). By the further sulphonation of these products, trisulphonic acids are formed. The following working quantities are given :—10 kilos. of the rosinduline sulphonic acid of the aforementioned specification, 30 kilos. of aniline, and 5 kilos. of aniline hydrochloride are heated for two hours to 165°—170° C. The phenyl rosinduline sulphonic acid separates as a greenish crystalline powder, the excess of aniline being removed in the usual manner. The new acid is almost insoluble in water, whilst its sodium salt is sparingly soluble. It dyes wool bluish-red shades from an acid bath, and when treated in the following manner yields a trisulphonic acid. 10 kilos. of the product are stirred into 30 kilos. of fuming sulphuric acid (32 per cent. SO_3) at 30°—35° C., after which the temperature is raised to 65°—70° C. for four hours. The melt, when cold, is poured on to 3—4 times its weight of ice, the trisulphonic acid filtered off, converted into its sodium salt, and the dyestuff salted out. It dyes very level bluish-red shades on wool, which are distinguished for their fastness to light and alkalis. The following characteristics distinguish it from the phenyl rosinduline sulphonic acids of Eng. Pat. 19,167 of 1890 (this Journal, 1891, 831). The free acid is readily soluble in water and forms well-characterised salts. Its disodium salt crystallises in brown lamellæ, is insoluble in alcohol, sparingly soluble in cold but easily in hot water, and can be completely precipitated by salt. The acid barium and calcium salts are nearly insoluble in water.

—T. A. L.

Basic Disazo Dyestuffs, Manufacture of. O. Imray, London. From "The Farbwerke vorm. Meister, Lucius, and Brüning," Hoechst a/M., Germany. Eng. Pat. 9343, May 2, 1896.

The amido ammonium bases produced according to Eng. Pat. 14,494 of 1895 (this Journal, 1896, 587), when utilised as dyestuff components, yield basic disazo dyestuffs, which dye cotton not only when mordanted with tannin, but also unmordanted cotton from an acid bath. The dyestuffs may be obtained according to three methods: either by diazotising amido dyestuffs produced by the action of diazotised amido ammonium bases on primary amines and combining them with other components, such as resorcinol, naphthol, *m*-phenylene diamine or β-naphthylamine, or by combining monazo dyestuffs with diazotised amido ammonium bases, or by treating azo dyestuffs capable of being combined, and containing an amido ammonium base as component, with diazo compounds. The following method gives the quantities employed in one of the processes :—3·77 kilos. of *m*-trimethyl ammonium phenyl-azo-α-naphthylamine, obtained by the action of diazotised *m*-amidophenyltrimethyl ammonium, on α-naphthylamine, are dissolved in 150 litres of water and diazotised by means of 2·4 kilos. of hydrochloric acid, and 0·69 kilo. of sodium nitrite. The diazo solution is run into a solution containing 11 kilos. of resorcinol and 3 kilos. of sodium acetate. After standing some time it is boiled and salted out, when it forms a blackish-brown powder.—T. A. L.

Cinnamic Aldehyde, Impts. in the Manufacture or Production of. C. F. Boehringer and Söhne, Waldhof, Germany. J. Y. Johnson, London. Eng. Pat. 10,003, May 11, 1896.

See under XX., *page* 463.

Discharging Para Nitraniline Red Fabrics White and Coloured, and Manufacture of a Discharging Colour therefor: Process for. O. Imray, London. Eng. Pat. 8860, April 27, 1896.

See under VI., *page* 438.

New Red Colouring Matters of the Triphenylmethane Group, and Method of Producing the Same. J. J. M. Ville, Paris, France. Eng. Pat. 16,039, July 20, 1896.

These dyestuffs, which the patentee terms rhodazines, are obtained by heating under an inverted condenser for about 12 hours, one, two, or three molecular proportions of phenylhydrazine with one molecular proportion of rosolic acid, with or without the presence of alcohol. The melt forms a reddish-brown mass, and for use it is dissolved in concentrated acetic acid. The solution is added directly to the dye-bath, which is afterwards neutralised with sodium aluminate until only slightly acid.—T. A. L.

Sulphuretted Colouring Matters [Black, Bluckish-Brown], Impts. in the Manufacture of. H. R. Vidal, Paris, France. Eng. Pat. 18,489, Aug. 20, 1896.

Certain meta derivatives of benzene, when heated with sulphur or with sulphur and ammonia, either alone or together with alkaline sulphides, yield dyestuffs which give black or blackish-brown shades on vegetable fibres. The derivatives suitable are dihydroxyl, amidohydroxyl, diamido, triamido, and certain sulphur derivatives, such as sulphaminol (*m*-oxythiodiphenylamine) or thioresorcinol. Thus, 30 kilos. of resorcinol saturated with ammonia and 6·4 kilos. of sulphur are heated in a closed vessel to 260 C. for eight hours, when a brittle blackish mass is produced. According to another method, 25 kilos. of Chrysoïdine or Bismarck brown are heated to 240° C. with 30 kilos. of sodium sulphide. When the reduction is finished, 6·4 kilos. of sulphur are added, and the reaction is complete when the mass has become too stiff to agitate.
—T. A. L.

New Colouring Matters [Blue-, Green-, and Brown-Blacks] from Benzidine Sulpho Acids, Manufacture of. H. Imray, London. From F. Petersen and Co., Schweizerhalle, Bâle, Switzerland. Eng. Pat. 1145, Jan. 15, 1897.

According to Eng. Pat. 13,742 of 1896 (this Journal, 1896, 647), certain intermediate products are obtained by combining the benzidine sulphonic acid of Griess with two molecular proportions of γ-amidonaphthol sulphonic acid. In the present specification, one or both of these latter is replaced by one or two molecular proportions of amidonaphthol disulphonic acid II, or by one molecular proportion of acid II and one of a phenol or amine. Polyazoic dyestuffs are thus obtained which show peculiar bluish shades on wool or half-wool and similar fibres. On further diazotising and combining with an amine or a phenol, or one of their derivatives, dyestuffs are produced which give bluish-black to brown-black shades on unmordanted vegetable fibres, fast to light and washing. A quantity of benzidine sulphonic acid corresponding to 10 kilos. of sodium nitrite is mixed in an alkaline solution with 17 kilos. of γ-amidonaphthol sulphonic acid, and, immediately afterwards, with 25 kilos. of amidonaphthol disulphonic acid II. The mass is then further diazotised and combined with 23 kilos. of *m*-phenylenediamine. After making the solution alkaline, the dyestuff separates as a black precipitate, which dyes cotton or wool a deep blue-black, fast to light and washing.
—T. A. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

Mercerised Cotton, Scrooping. H. Werner. Leipziger Färber- u. Zeugdrucker Zeit. **46**, [5], 179—181.

To cotton which has been lustred by mercerisation whilst in a stretched condition (see this Journal, 1896, 804 and 898) a silk-like scroop may be imparted by the following process.

Work the goods for 15 minutes at 40° C. in a solution of calcium acetate of 5—6° B. Squeeze lightly, and, without rinsing, work for 15 minutes at 50°—60° C. in a bath containing 40—60 per cent. of soap. Again squeeze lightly and pass through a cold bath consisting of 1 part of acetic acid to 10 parts of water, and dry without rinsing.

This process is generally applied after dyeing. Should the shade have undergone a slight change, this may be rectified by adding a suitable basic dyestuff to the acetic acid bath. For light shades it is preferable to pass the goods in the grey or bleached state through the calcium acetate, and to dye them by adding some substantive dyestuffs to the soap-bath, and to shade, if necessary, with basic colouring matters in the acetic acid bath.—I. S.

Raw Fibres, A New Method for the Quantitative Estimation of. Lebbin. Arch. Hyg. 1897, **28**, 212.

See under XXIII., *page* 471.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Acetic Acid as an Assistant in Dyeing. A. H. Wainright. (N. Dyers' Journal.) Leipziger Färber Zeit. **46**, [1], 149—150.

The author advocates the substitution of acetic acid for the stronger mineral acids in dyeing, on the ground that the dyes obtained with it are more level, the penetration of the dyestuff, in the case of heavy cloths or felts, much better, whilst yarns are less liable to felting than with the more active mineral acids.—I. S.

Logwood Substitutes in Woollen Dyeing. Leipziger Färber-u. Zeugdrucker Zeit. **46**, [4], 155—157.

This is a review of the various artificial colouring matters which have been put on the market in competition with logwood for blacks on woollen cloth. The dyestuffs which the author considers as most suitable substitutes for logwood, are Diamond Black F, Naphthylamine Black R, and Naphthyl Blue-black N. The following methods of application are given. Diamond Black is dyed with 10 per cent. of Glauber's salt and 4 per cent. of acetic acid. Enter at "hand-heat," raise slowly to boiling, keep the bath at this temperature for half an hour, then add 1 per cent. of sulphuric acid, and boil again for half an hour. Add now 1 per cent. of potassium bichromate and boil half an hour longer.

Naphthylamine Black is dyed with 10 per cent. of Glauber's salt and 5 per cent. of acetic acid. Enter at 65° C., raise to boiling point, and maintain this temperature for half an hour; then add 3 per cent. of copper sulphate and 4 per cent. of acetic acid, and keep boiling until the bath becomes exhausted.

Both these colouring matters possess the advantage over logwood of being much faster to light and acids; whilst Naphthylamine Black R has the additional advantage that its tint is not altered in artificial light.—I. S.

Pink on Cotton with p-Nitraniline. M. Goldovsky. Färber Zeit. **8**, [6], 89.

The author aimed at finding a substitute for alizarin pinks that would not involve so many tedious manipulations as the latter, and he recommends the use of *p*-nitraniline for the purpose.

Equal molecules of *p*-nitraniline salt and hydrochloric acid, together with 15 per cent. of common salt (of the weight of material taken), are dissolved in boiling water. The yarn is impregnated with this solution, squeezed, passed through a nitrite bath, then through a solution of β-naphthol, and soaped. The resulting colour is yellowish rather than pink. But a good pink may be obtained if 2 mols. of sodium acetate are substituted for the 1 mol. of acid in the above formula.

Subsequently the author has modified this process by passing the impregnated yarn through a solution of a "lead salt" before entering the nitrite bath. Another method, which the author believes to be the most promising, is first to fix on the fibre a metallic oxide (preferably that of lead), and, after rinsing, to pass the goods through the diazo solution of *p*-nitraniline, and develop in β-naphthol. This process is said to give beautiful light pinks. For fuller and deeper shades the order of treatment is reversed. In either case the goods are subsequently to be soaped.—I. S.

Discharging Para Nitraniline Red Fabrics White and Coloured, and Manufacture of a Discharging Colour therefor; Process for. O. Imray, London. From "The Farbwerke vorm. Meister, Lucius, and Brüning," Hoechst a/M., Germany. Eng. Pat. 8860, April 27, 1896.

In discharging paranitraniline red it has been hitherto difficult to obtain pure whites, and the same objection also applies to the use of resists. The patentees have, however, discovered that satisfactory results are produced by employing in the resist, substances in which paranitraniline red is soluble. The products of reduction (*p*-phenylenediamine and amidonaphthol) are then removed by washing. A tin-salt discharge is printed on *p*-nitraniline red, containing 50—150 grms. per litre of a solvent such as glycerin, acetin, chlorhydrin, &c., together with the acetates and citrates of ammonium and a thickening such as wheat starch or gum. After printing and dyeing, the fabric is steamed for 10 minutes, passed through hot water, well washed, and then passed through a very dilute slightly acid solution of bleaching powder. By the addition of dyestuffs which are not affected by the treatment (such as Persian berries, with or without the addition of aniline colours, &c.), coloured discharges can be obtained.—T. A. L.

Paranitraniline Red Discharge on Indigo, The Production of. C. Kurz and F. Kunert. Färber Zeit. **8**, 49—50.

The only discharge suitable for this purpose is found to be the lead chromate and chlorine discharge, which depends on the fact that lead chromate precipitated on the fibre causes liberation of chlorine on passing through a bath of hydrochloric and oxalic acids, thereby discharging the indigo. Paranitraniline red is totally unaffected by chlorine. The method for producing a red on a dark vat-blue ground is as follows:—Pad the dyed cotton with 1 litre of water, 30 grms. of β-naphthol, 32 c.c. of caustic soda of 70° Tw., 60 grms. of Turkey-red oil, 2 grms. of tartar emetic, and 300 grms. of neutral potassium chromate. Dry in the hot-flue and print with 250 c.c. of starch-tragacanth thickening, 100 grms. of lead acetate, and 250 c.c. of a solution of diazotised paranitraniline containing 13 grms. of paranitraniline, and 30 grms. of sodium acetate in addition to sodium nitrite and hydrochloric acid. After printing, dry, pass through dilute ammonia, wash well, and pass rapidly through the discharging bath, which contains 40 c.c. of hydrochloric acid of 36° Tw., and 40 grms. of oxalic acid per litre; finally, wash thoroughly and dry.

If a white discharge be desired in addition to the red, a thickened lead acetate solution is also printed on the fabric before discharging in the acid bath.—R. B. B.

Orange and Red Shades by Means of Nitrotoluidines on a β-Naphthol Ground, Production of. T. Skawinski. Bull. Soc. Ind. Mulhouse, 1897, 90—94.

The author has examined the shades obtained by combining the diazo compounds of 8 of the 10 possible nitrotoluidines with β-naphthol padded on a fabric, and arrives at the results given below. To prepare the diazo bath, dissolve 3·5 grms. of a nitraniline or 3·8 grms. of a nitrotoluidine in 50 c.c. of boiling water and 6 c.c. of hydrochloric acid (22° B.), cool, add 100 grms. of ice, diazotise with 25 c.c. of normal nitrite, allow to stand 15 minutes, then add 7·5 grms. of crystallised sodium acetate and make up to 250 c.c. The cotton fabric is prepared with β-naphthol in the usual manner.

Base.	M.P.	Shade produced.
o-Nitraniline	..	Reddish-orange.
m-Nitraniline..........	..	Yellowish-orange.
p-Nitraniline	..	Bluish-red.
Nitrotoluidine, $CH_3.NO_2.NH_2$—		
1.3.4.............	114° C.	Yellowish-red.
1.4.5.............	109° C.	Orange, yellower and duller than *o*-nitraniline.
1.5.6.............	97° C.	Orange, redder than *o*-nitraniline.
1.2.6.............	92° C.	Orange, yellower than *m*-nitraniline, and brighter than 1.4.5.
1.2.4.............	78° C.	Orange, similar to the preceding, but duller.
1.4.6.............	107° C.	Orange, much redder than *m*-nitraniline.
1.3.6.............	128° C.	Reddish-orange.
1.2.5.............	138° C.	Orange, similar to *o*-nitraniline.
Nitroxylidine, $CH_3.CH_3.NO_2.NH_2$.		
1.4.2.5..........	142° C.	Red, much yellower than *p*-nitraniline.

—T. A. L.

Resists and Coloured Designs on an Aniline Black by Printing on Resists with a Black containing Substantive Colours. P. W. Pluzanski. Bull. Soc. Ind. Mulhouse, 1897, 98—103. Sealed Communication deposited May 10, 1885; opened Oct. 28, 1896.

The method consists in printing an alkaline resist, either white or coloured, and printing on this with an aniline black containing a direct dyestuff. The black does not develop on the resist, whilst the dyestuff contained in it, does, thus giving its own colour if the resist used be white, and a different shade if it be coloured. A second communication gives details of the quantities employed, of which the following is an example:—On a diamine blue ground the following resist is printed: 218 grms. of tragacanth, 100 grms. of crystallised tartaric acid, 46 grms. of calcined potassium carbonate, 66 grms. of caustic soda lye (45° B.), and 100 c.c. of water; this must react neutral, and is mixed with 170 grms. of tragacanth and 120 grms. of tin salt, after which 60 grms. of sodium carbonate are added, and finally 100 grms. of crystallised sodium acetate, 10 grms. of olein, and 10 grms. of terebenthene. The black consists of 1 kilo. of black thickening, 520 grms. of Brilliant Green, 80 grms. of glycerin, 90 grms. of 97 per cent. aniline, and 90 grms. of hydrochloric acid (19° B.). The goods are then steamed for three minutes in a Mather-Platt steaming chamber well soaped (at 44° C.), dried, and finished. For obtaining discharges on coloured goods dyed with chromium acetate or with basic colours on tannin and tartar emetic, the fabric is printed with a mixture containing sodium chlorate, citrate, and acetate, citric acid, potassium ferricyanide, and chromium acetate, then steamed, grounded with Aniline Black containing Rhodamine, Methylene Blue, &c., passed through the Mather-Platt steaming chamber, and finished as above.—T. A. L.

Finishing Operations, The Influence of, on the Colour of Wool-Dyed Goods. O. Walther. Färber Zeit. **8**, 50—51.

In the selection of colouring matters for the dyeing of loose wool, too much attention cannot be paid to the influence of the various finishing operations upon the colour of the cloth. Washing, milling, carbonising, raising, shearing, drying, steaming, and hot-pressing may each have an important influence upon the shade, these influences being partly chemical and partly mechanical. The operations of raising and shearing may considerably affect the depth of colour. It is usual in raising, to commence with already used and comparatively blunt teazles, and

gradually proceed to sharper ones; if this change is not sufficiently gradual there is a tendency to tear out fibres from the fabric; the resulting covering of hairs is then less thick than when the progression has been less rapid, and the colour appears paler. Shearing too close has also the effect of making the shade appear paler, especially when raising has been badly carried out. Too vigorous a steaming may readily change the tone of colour, and too strong a pressure forces the surface hairs down upon the body of the fabric, and in this case also, the colour will appear paler.—R. B. B.

Leather Dyeing with Artificial Dyestuffs. Leipziger Färber- u. Zeugdrucker Zeit. 46, [4], 162—165.

Steeping Method.—The dyeing is most conveniently carried out in a wooden trough, with sloping sides, about 1 m. long, 0·5 m. wide, and 0·25 metre deep. Iron should be avoided in the interior of the trough. The water to be used should be free from lime and iron. When such water is not available, about 10 c.c. of acetic acid should be added to a bath of 5—6 litres.

Two skins about equal in size are laid with their flesh sides together, and are then pressed together on a plate of glass or zinc, by passing over them a blunt brass blade, which at the same time removes any adhering foreign matter. By this means only the grain side of the skin is dyed, leaving the inside white.

From 5—6 litres of water, at about 40° C., are poured into the trough, and the requisite dyestuff is added in solution. In order to obtain level dyeings, however, the dyestuff is distributed over three baths of varying strengths, the first being the weakest, and the last the strongest. The second pair of skins is first passed through the second, and then through the third old baths, and finally through a freshly-prepared third bath. For very delicate shades, two baths, one old and one new, are sufficient.

For basic colours, the leather is first mordanted with potassium bichromate for light, with iron acetate for medium, and with pyrolignite of iron ("Iron liquor") for dark shades. When very small quantities of potassium bichromate are sufficient, this may be added to the dye-bath direct; but when larger quantities are to be used, the bichrome is applied in a separate bath, in which the skins are worked for about 10 minutes. Iron mordants should be used of a strength of 15° B. for the pyrolignite, and $1\frac{1}{2}$° B. for the acetate.

After dyeing, the skins are well rinsed, and the surplus water is removed by laying them flat on an inclined plate of glass or zinc, and passing over them a blunt blade of brass or steel. Skins that are to be glazed receive now a coating of raw linseed oil, and are then dried in the air, or in a moderately heated room. Too slow drying may cause unevenness of shade. During the drying, creases are to be avoided.

Brushing Method.—To dye leather by this method, the dry skins are laid on a table and the tepid dye solution is applied with a long-haired brush. As it is difficult in this way to get an even colour free from streakiness, in one operation, the dye solution is used very weak and the skins receive several successive coats, allowing each one to dry before the next one is applied. When acid colours are used, some acetic acid, or a correspondingly smaller quantity of sulphuric acid, is added to the dye-bath.

Finally, the skins receive a dressing of milk, or of a mucilage of Flea-wort (Flohsamen), *Plantago Psyllium-L.*, or linseed, in order to impart to the leather, softness and a dull finish, not otherwise obtainable. The first of these dressings is used for light, the second for medium, and the third for dark shades. The two last are prepared by boiling the Flea-wort or linseed with water and straining off the mucilage, which is then diluted to the proper strength. These dressings are applied to the dry skins with a sponge, and after allowing to dry, the skins are rubbed with a hair brush or coarse flannel, which produces the required dull finish.—I. S.

PATENTS.

Bleaching, Dyeing, Washing, and similarly Treating of Spun Fibres and Stuffs of every kind; Process of and Apparatus for. B. Thies, Oehtrup, Germany. Eng. Pat. 8491, April 22, 1896.

A NUMBER of vessels, which serve as receptacles for the materials to be dyed, &c., are mounted near one another and connected with liquor-supply and -collecting vessels, the latter of which receive the liquors after use, for conveyance back to the supply vessels. All the vessels are closed when in use, and by means of arrangements of pumps, exhaust vessels, valves, and two- and four-way cocks, the flow of the liquors through the materials under treatment, is caused to take place alternately in opposite directions. The materials may also be treated with gases, air, and steam, if desired.

Numerous modifications in the construction and arrangement of the apparatus, and in the methods of working it, are described in the original specification, which is illustrated with eight sheets of drawings.—E. B.

Colouring Photographs, Improved Means for, suitable also for Dyeing and Printing. J. A. Michel-Dansac and L. Chassagne, Paris. Eng. Pat. 18,131, Aug. 15, 1896.

See under XXI., *page* 464.

Bleaching Cotton and other Vegetable Textile Materials, An Improved Method or Process of. J. Kent, London. Eng. Pat. 22,823, Oct. 14, 1896.

COTTON and other vegetable textile fibres are "bleached" or "half-bleached" by steeping them in a solution prepared by treating a finely ground mixture of lime and soda with water. The fibres may afterwards be treated with bleaching powder and acid in the usual way.—E. B.

VII.—ACIDS, ALKALIS, AND SALTS.

Sulphur, The Extraction of, in Sicily. Eng. and Mining J., April 24, 1897.

IN a recent paper in the *Rassegna Mineraria*, Sr. Aichino says that by far the greater portion of the sulphur raised from the mines of Sicily is extracted from the accompanying limestones by liquation in kilns, the necessary heat being produced by the combustion of part of the sulphur in the rock. In other cases the fuel used is coal or wood, or superheated steam, or the liquation is effected by a solution of calcium chloride, whose boiling point is slightly higher than the melting point of sulphur. The preponderating method is still that of using large kilns, originally introduced in 1860. The only difference now noticeable is the increased dimensions of the kilns. The kiln consists merely of a circular pit, surrounded by a wall. The bed slopes at an angle of 10° to 15° toward a rectangular aperture in front, 4 ft. high and 9 in. wide. The burning lasts, for kilns holding 250 to 2,500 cb. m., from a month to three months. The consumption of sulphur as fuel amounts to one-third or even more of the sulphur contained in the rock, a proportion notably greater that that theoretically necessary. In practice it is not found expedient to treat rock containing less than 10 per cent. The cost of treatment averages 18 c. per ton. In order to obviate the great loss of sulphur numerous modifications have been proposed. In 1880 Robert Gill patented a regenerative furnace for the purpose, which has been largely adopted. The chambers have a capacity of 10 to 30 cb. m., and a kiln with two chambers can be used for 45 to 90 operations in the year. The steam extractor, which was introduced 25 years ago, has gradually come into more general use. It consists of an iron vessel filled with mineral into which steam is conducted. The cylinder holds 5 to $5\frac{1}{2}$ tons, and the operation lasts 24 hours. In a recent installation with a horizontal cylinder holding $4\frac{1}{2}$ tons it has been found possible to complete 15 to 16 operations in 24 hours.

The relative importance of the three types of apparatus is shown by the statement that of the sulphur produced in Sicily in 1891 about 74·5 per cent. was from the old kilns, 17 per cent. from regenerative furnaces, and 8·5 per cent. by steam extractors. In 1895 the proportions were 56·3 per cent., 31 per cent., and 12.7 per cent. respectively.

Chlorine by Mond's Process, Preparation of. G. Lunge and E. Marmier. Zeits. f. angew. Chem. 1897, 137—138.

Although in this method the loss of nickel is said to be considerable and the chlorine obtained not pure, investigation has been undertaken with the view of a comparison with the Deacon process, with which it has certain points in common. The process consists in passing air and hydrochloric acid gas over pumice soaked in nickel chloride, and heated to a temperature of about 650° C. In the first experiments, the gases were first mixed, whilst subsequently they were passed over the hot pumice in succession. When employing the former method, the best result, *viz.*, 54 per cent. of the theoretical amount of chlorine, was obtained by passing 25 volumes of hydrochloric acid and 75 volumes of air over the pumice heated to 650° C. At this temperature, however, the sublimation of the nickel chloride was very considerable, and resulted in the exit tube soon becoming choked. According to the alternative method the pumice soaked in nickel chloride was heated in a current of air at 400° C., the temperature being gradually raised to 650° C. in order to convert the nickel chloride into nickel oxide. A known quantity of hydrochloric acid gas was then passed through the apparatus, that unabsorbed by the nickel oxide being retained by caustic soda and titrated, so that an approximate idea was obtained of the amount remaining in the apparatus. Air was then passed through the apparatus, the temperature being raised, and the chlorine and hydrochloric acid were estimated in the issuing gas. The following conclusions were arrived at from the experiments :—1. The best temperature for the absorption of hydrochloric acid by nickel oxide is between 450° and 500° C. 2. The freshly prepared catalyte absorbs hydrochloric acid well, but soon loses its absorptive power, so that frequent renewal is necessary. 3. The best temperature for the formation of chlorine by passing air over, appears to be between 600° and 650° C. The ratio of Cl : HCl during the whole process is, on an average, 80 : 20, but at certain periods the proportion of chlorine rises to 91 per cent. The decomposition proceeds best when the pumice, after treatment with hydrochloric acid gas, is quickly and not gradually raised to the higher temperature. It is to be observed with regard to the purity of the chlorine, that this method was patented with a view to utilising it in the Ammonia-soda process. Even for this, however, the method, it is said, has been abandoned, since volatilisation of the nickel soon chokes the tubes. (See also this Journal, 1893, 146.)—T. A. L.

Chlorine and Steam, Action of a Mixture of, on Glowing Charcoal. A. Naumann and F. G. Mudford. Ber. 1897, **30**, 347—354.

The authors have investigated the action of a mixture of chlorine and steam on glowing charcoal, and, in contradistinction to the results of Lorenz (Zeits. anorg. Chem. 1895, **10**, 74—77), have arrived at the following conclusions.

(1.) The action of a mixture of steam and chlorine on glowing charcoal takes place in accordance with the equation—

$$2Cl_2 + 2H_2O + C = 4HCl + CO_2$$

at the temperatures attained in an ordinary combustion furnace, and not quantitatively, according to the equation—

$$Cl_2 + H_2O + C = 2HCl + CO,$$

as maintained by Lorenz.

In proof of this, they point out that the ratio of carbon monoxide to carbon dioxide increases with increased length of the layer of glowing charcoal, which indicates that the carbon dioxide first formed is afterwards reduced in passing over the glowing carbon.

(2.) The increase of carbon monoxide is, in presence of a sufficient quantity of chlorine, limited by the following process, which takes place even at low temperatures and which may be of technical value :—

$$Cl_2 + H_2O + CO = 2HCl + CO_2.$$

(3.) The formation of hydrochloric acid as the result of the combined processes takes place with an evolution of heat, so that if the proper conditions are fulfilled, and especially if an excess of steam be avoided, the continued application of heat on the large scale would be unnecessary. —J. S.

Chlorate, On the Conversion of Hypochlorite into. J. Bhaduri. Zeits. anorg. Chem. 1897, **13**, 385.

The author has carried out a series of experiments to find the exact conditions under which the above conversion takes place. Sodium hypochlorite was prepared by conducting washed chlorine gas through a caustic soda solution made from metallic sodium. The content of the solutions varied in the different experiments, from 1·5 to 25 per cent. The temperatures at which the absorption took place, lay between 25°—33° C., and the gas current was continued in some experiments for 4 or 5 hours. It was found that the amount of chlorate formed was inappreciable in solutions below 7 per cent. strength, and that in such solutions the reaction proceeded according to the equation—

$$2NaOH + Cl_2 = NaOCl + NaCl + H_2O.$$

In solutions of 10 per cent. and upwards, the secondary reaction—that is, the conversion into chlorate—became more marked, and when the percentage strength rose above 20, it was found that time played an important part in the decomposition.

In one series of experiments a solution kept in the dark, showed a decomposition of only 6 per cent. after 21 hours. By conducting chlorine through another portion of the solution for 1 hour, decomposition proceeded up to 1·5 per cent.; free alkali hindered the change. Further experiment showed that a solution of sodium hypochlorite underwent change even in the dark, and that the amount of decomposition increased with the refrangibility of the light rays; the yellow rays were far more active than the red. The decomposition gave rise to chlorate and oxygen, and proceeded more rapidly under diminished pressure.

The author has also studied the change occurring in the dark at 100°, and he finds that with dilution, the decomposition diminishes to a certain point, where it possesses a minimum value. With further decrease in the concentration, the decomposition again increases. The most stable aqueous solution is one containing approximately 0·4 per cent. of salt molecules.—J. T. C.

Salts, Action of Free Bases on. A. Colson. Comptes Rend. 1897, **124**, 502—504.

The author has proved that the displacement of ammonia in ammonium chloride by the free bases di-isobutylamine and piperidine is a case of heterogeneous dissociation similar to the action of a fixed acid on the salt of a volatile acid, *e.g.*, sulphuric acid on lead chloride.—J. S.

Ammonium Cyanide, Note on the Formation and on the Manufacture of. D. Lance. Comptes Rend. **124**, 819—821.

According to Langlois and Kuhlmann, ammonia gas, when passed over carbon, always yields ammonium cyanide; on the other hand, Bueb and Bergmann assert that at 1,000° C. and temperatures above this, only hydrocyanic acid is formed, and the residual ammonia is all dissociated into nitrogen and hydrogen. The author has caused dry ammonia gas to pass through a column of wood charcoal, 0·2 m. high by 0·02 m. in diameter, contained in a porcelain tube. The gas was passed at the rate of 4 litres per hour, and the temperature of the charcoal, as determined by the Le Chatelier pyrometer, in a series of experiments, varied from 1,000° C. to 1,100° C. The products of the reaction were collected in a series of washing vessels containing distilled water, and the liquors obtained were titrated—1st, by Buignet's method; 2nd, by Liebig's method. Liebig's process being only applicable in the presence of an alkaline cyanide, the difference or the identity of the results obtained by the two methods at once showed whether a liquid containing free hydrocyanic acid or ammonium cyanide only, had been produced. In every case the tests by Liebig's method were identical with those by Buignet's, showing that under the conditions above described, ammonium cyanide was always formed. The author found that the temperature employed (1,000°—1,100° C.) was that which yielded a maximum amount of cyanogen, and that the nitrogen converted into cyanogen was equal to 25 per cent. of the nitrogen contained in the ammonia used.

When, however, a mixture of ammonia, hydrogen, and nitrogen was passed through the charcoal heated to the same temperature, a much larger yield of cyanogen was obtained. A mixture in the proportion of 1 litre of ammonia gas, 1 litre of nitrogen, and 8 litres of hydrogen raised the percentage of nitrogen converted into cyanogen to 30·6 per cent. of the nitrogen of the ammonia, and by decreasing the proportion of ammonia relatively to the two other gases, and also increasing the proportion of hydrogen relatively to the nitrogen, the yield of cyanogen was still further increased. Thus, a mixture in the proportion of 200 c.c. of ammonia gas, with 5 litres of hydrogen and 500 c.c. of nitrogen, yielded an amount of cyanogen containing nitrogen equal to an average of 85 per cent. of the nitrogen contained in the ammonia used. As this amount of cyanogen was combined with an equivalent of ammonia containing the same percentage of nitrogen, it was evident that at least 70 per cent. of the nitrogen in the recovered ammonium cyanide had been derived from the free nitrogen in the mixture.—H. S. P.

Sodium Bicarbonate, Analysis of. G. Lunge. Zeits. f. angew. Chem. 1897, 169.

See under XXIII., *page* 468.

Liquid Ammonia, Analysis of Commercial. A. Lange and J. Hertz. Zeits. f. angew. Chem. 1897, 224.

See under XXIII., *page* 467.

Monazite Sands, Examination of the Earths contained in. Schützenberger and Boudouard. Comptes Rend., **124**, 481.

See under II., *page* 429.

Thorium Nitrate, Crystallised. O. Fuhse. Zeits. angew. Chem. 1897, 115.

See under II., *page* 429.

Thoria, Behaviour of, with Oxalic acid and Ammonium Oxalate, and the Estimation of. C. Glaser. Zeits. Anal. Chem. **36**, 213.

See under XXIII., *page* 468.

PATENTS.

Salt from Brine, Impts. in and relating to Apparatus for obtaining, and for similar Purposes. W. Shedlock, Clapham, Surrey. Eng. Pat. 7796, April 13, 1896.

A PAN, with a circular brim and flat bottom, is continuously fed with brine, led into a circular vessel mounted upon a vertical central revolving shaft, which also carries a series of scrapers. One or more perforated pipes, extending from the receiving vessel, distribute the brine as they revolve. Flat chambers or pipes, having the form of incomplete rings, to admit of passage downwards of the salt scraped off their upper surfaces, are provided as heaters, and the heat of the water of condensation (when steam is used as the heating agent) is utilised for raising the temperature of the brine before it enters the pan. The pan is preferably steam-jacketed at the bottom. A trough or channel is arranged below for the reception of the salt, which, by the action of the scrapers, is continuously driven towards an aperture in the bottom of the pan; and the salt is carried by a screw-conveyor in the trough to an inclined casing, from which it is delivered by a suitable elevator, in a drained condition, into moulds or other receptacles. Alternative modifications of the apparatus are described.
—E. S.

Evaporating Apparatus, Impts. in and relating to, chiefly designed for obtaining Salt from Brine. W. Shedlock, Clapham, Surrey. Eng. Pat. 7997, April 15, 1896.

See under I., *page* 428.

VIII.—GLASS, POTTERY, ENAMELS.

Glass coloured by Copper. P. Zulkowski. Chem. Ind. **20**, [6], 134—139.

THREE kinds of glass coloured by means of copper were examined by the author, *viz.*:—*Copper-ruby*—an alkali-lead glass containing 64·8 per cent. of silica, 1·63 of cuprous oxide, 2·16 of stannous oxide, 10·71 of lead oxide, 9·11 of potassium oxide, and 5·13 per cent. of sodium oxide; "*Aventurin*"—an alkali-lime glass with 4·9 per cent. of copper, 3·07 of lead oxide, 8·74 of calcium oxide, 4·46 of potassium oxide, and 10·22 per cent. of sodium oxide; and "*Porpora*"—an alkali-lime-lead glass with only 35·8 per cent. of silica, but 39·06 of lead oxide, 7·89 of cuprous oxide, 4·31 of calcium oxide, 2·6 of potassium oxide, and 6·31 per cent. of sodium oxide, this latter glass resembling the ancient *hæmatinone*.

The *Aventurin* exhibited, under the microscope, innumerable crystals resembling those of metallic copper, the presence of which in the free state was proved by the decoloration, effected by the pulverised glass, of an ammoniacal solution of cupric oxide when heated in a sealed tube, thus confirming Wöhler's view that the peculiar appearance of this glass is due to metallic copper in suspension.

On the other hand, neither the *Copper-ruby* nor *Porpora* glass exerted any reducing action on cupric oxide solution, even after three years' exposure in sealed tubes, and this circumstance, coupled with their appearance, lends the author to conclude that their colour is due to cuprous oxide, in solution in the first-named, and as dark red crystalline ramifications, branching in all directions, in the *Porpora*.

Experiments on the devitrification of slags that have been forced to dissolve or combine loosely with extraneous substances, showed that though the mass remains homogeneous when cooled suddenly, yet, when the internal friction is diminished by reheating, molecular rearrangements impairing the homogeneity of the mass are easily occasioned, and, as this phenomenon is on a parallel with the behaviour of glass coloured by copper, it is assumed as probable that the cuprous oxide forms an unstable compound, which, on reheating, is broken up, the oxide remaining in solution (*Copper-ruby*), or, finally, after prolonged heating, separating out as crystals (*Porpora*) in a manner analogous to devitrification. This view is also held to apply to glazes, glasses and enamels coloured by metallic oxides, Seger (Jahresber. f. Chem. Tech. 1896, 762) having disproved the "dissolved metal" theory in the case of copper-red glazes by showing the necessity for an alternate reducing and oxidising atmosphere in the kiln during their preparation, whereby glaze containing cupric oxide and fritted at 400° to 500° C. in an atmosphere of hydrogen, assumes a red colour, which it loses at a higher temperature (1,000° C.), and only regains when fused again with a glaze free from copper, containing some oxidising agent. This reaction explains the previously unknown *rôle* of the tin and iron oxide always used in making these glazes, their function being to at first reduce the cupric oxide, and subsequently oxidise the metal. An excess of tin may reduce, not only the copper oxide, but also any sulphates present, as was found by Witt in a glass slag composed almost entirely of cuprous sulphide, and containing, in the centre, a regulus of tin and copper.

With regard to the production of gold colouring, which should be on a par with copper if the foregoing assumptions be correct, the author points out that the non-existence in a free state of a fire-resisting red oxide of the former metal, does not disprove the hypothesis; and, from the dyeing properties of the ammoniacal solution of "gold purple," he considers it not impossible that this metal may form in molten glass an unknown compound, which, in subsequent stages of decomposition, produces gold-red, and also gold *Aventurin* glass.—C. S.

PATENTS.

Coloured Glass Lights for Lenses, Impts. in the Methods of Producing; also applicable for other suitable Purposes. J. Davis and A. J. Davis, West Bromwich, Stafford. Eng. Pat. 15,804, July 17, 1896.

THE invention relates to the manufacture of lenses and dioptric lights of plain and coloured glass combined, as well as to coloured glass lights for use on ships, lighthouses, railways, &c., and may also be applied in the production of table glasses of various kinds. To produce

a plano-convex lens, of ruby colour for instance, a flat piece of ruby glass, cut to the required size, is heated and bent to fit a thin concave heated iron mould. The formed glass is transferred to the concave bed of a massive mould, and a mass of white molten glass is pressed upon it by a flat plate or cover. The mould, with its contents, is then placed in a heated kiln, and the lens thus formed is afterwards ground on the plane surface. In forming a dioptric lens, the piece of coloured glass is bent to the convex surface of a plano-convex-shaped mould, called the "combiner and compressor," and is retained in its place by clips; white molten glass "metal" is then placed in a mould of the form required for the outside face of the lens, and the compressor, with its adhering plate of coloured glass, is applied. The lens is then heated and annealed. The methods of producing other forms of combined coloured and white glass are similar.—E. S.

Stoneware and the like, Improved Method of Manufacturing. F. S. Winser, London. Eng. Pat. 2791, Feb. 2, 1897.

Selected fireclays are incorporated with flint, Cornwall stone, or china clay, finely ground and sifted, and "mellowed" in the clay shed. The material is then worked on plaster slabs, before being placed in plaster of Paris moulds, in which it is left for 24 hours. The moulded articles are then dried, finished off, and glazed with a compost of 4 parts of Cornwall stone, 2 parts of whiting, and 1 part of calcium sulphate; and, after again drying, are fired at a slowly increasing temperature. The name of "Siennow" is given to the stoneware thus made.—E. S.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Stone [Artificial] capable of resisting Fused Alkalis, Alkali Sulphides, and Hydrochloric Acid. W. Hempel and W. Sezierski. Bayerisches Ind. u. Gewerbe Blatt; Thonwaaren Ind. **11**, 148.

The natural soap-stone (steatite) occurring in Norway, the Tyrol, and North America is mainly composed of SiO_2 (44·3 per cent.), MgO (20·7), CaO (3·4), and Al_2O_3 (4·3 per cent.), and when fired, resists the action of fused alkalis and alkali sulphides, the loss in weight not exceeding 0·4 per cent., whereas in the case of ordinary fire-brick it amounts to about 54 per cent.

In an attempt to imitate this stone for lining alkali furnaces, the authors selected a brick-earth containing SiO_2, 72·82 per cent; Al_2O_3, 10·18; MgO, 0·31; and CaO, 0·63 per cent., differing from the natural stone chiefly in the inverted ratio of silica and alumina. To approximate to the constitution of the latter, they employed the calcined magnesia, formed as a by-product in the hydrochloric acid works at Stassfurt, and containing 83·76 per cent. of MgO, and 3·42 per cent. of SiO_2; also a dolomite from Ostrau, with 16·84 per cent. of MgO, 27·86 of CaO, and 5·13 per cent. of SiO_2. Two classes of mixtures (which follow) were made, the one forming a product agreeing in silica-content and the other in magnesia-content with the natural stone; these gave, on examination, the following results:—

Mixture I. (44·2 per cent. SiO_2).—Clay, 142 parts; magnesia, 100 parts. *Fusing point* same as steel. Loss of weight on exposure to fused alkali and alkali sulphides, 0·02 and 0·12 per cent. respectively.

Mixture II. (20·7 per cent. MgO).—Clay, 300 parts; magnesia, 100 parts. Fused concurrently with I.; lost 0·18 per cent. under the action of fused alkali sulphide, but decomposed in presence of fused alkali (soda).

Mixture III. (44·3 per cent. SiO_2).—Clay, 57·83 per cent.; dolomite, 42·17 per cent. This mixture forms a hard stone corroded by fused alkali to the extent of 0·5, and 0·01 per cent. by sulphides; it also resists concentrated hydrochloric acid, and, being more refractory than the natural stone, forms the best substitute for the latter. Both the component materials are plentiful in nature, and it is therefore advisable to employ the product for furnace linings, those parts not in contact with the fused alkalis being constructed of fireclay, with an intermediate zone of pure magnesia when high temperatures are in question. —C. S.

Cement, New Quick-Setting Resistent. Monit. Ceramique, **28**, [4], 39.

The Société métallurgique de Champigneulles employs hot blast-furnace slag—containing 32 to 36 per cent. of silica, 39 to 45 per cent. of lime, 15 to 22 per cent. of alumina, 0·5 to 1·5 per cent. of iron oxide, and 0 to 8·5 per cent. of manganese oxide — quenched in cold water, and thoroughly mixed with slaked lime in the following proportions:—

(1.) Strong lime, 25 to 30 parts; slag sand, 75 to 70 parts (dry).

(2.) Hydraulic lime, 35 to 50 parts; slag, 65 to 50 parts; together with sufficient water to form a stiff mortar. On leaving the mixer, the mass is formed into slabs, and exposed to the air until dry enough to break into large pieces, and it is then roasted in furnaces at a temperature ranging from dull to bright red heat, according to the degree of quick setting to be produced. It is finally ground to fine powder.

To prepare less rapid setting cements, the calcined mass is ground with dry, quenched furnace slag (as above) and slaked lime in suitable proportions.—C. S.

PATENTS.

Insulating Material for Underground Electric Wires, Cables, or the like; New or Improved Manufacture of. P. R. de Faucheux d'Humy, Liverpool. Eng. Pat. 5794, March 16, 1896.

The substance of this invention is the use of cellulose material, such as cotton disintegrated by heating in petroleum or other hydrocarbon. Shale oil and refined petroleum are mixed, and ozokerite and fine sawdust or like vegetable material introduced. A certain quantity of paraffin wax is then added, together with waste cotton or refuse fibre, and grease—in preference, linseed or cotton-seed oil. After the addition of water the whole is boiled, and when the cotton is entirely amalgamated and the water evaporated, fresh waste cotton or cotton wool is added, with a small quantity of ozokerite. The cotton wool is allowed to soak, and when cool, the material is pressed between cylinders and converted into sheets. Or it can be applied direct in a hot state, to the wire to be insulated.—C. A. M.

Bricks and the like, Impts. in Insulating. S. Pollak, Klausenburg, Hungary, and N. Zwillinger, Egeres, Hungary. Eng. Pat. 8281, April 20, 1896.

The inventors claim the method of making insulating bricks by impregnation, immediately on leaving the kiln, with a hot solution of asphalt, tar, paraffin, or pitch, and the use of such bricks for building purposes, electric insulators, and for rendering floors and walls damp-proof. —V. C.

Wood [Waterproofing for Carvings], Impts. in the Method of Preparing or Treating, for the Purpose of Waterproofing the same, or for making it more applicable for the Formation of Permanent Pressed Designs. W. P. Thompson, Liverpool. Eng. Pat. 11,182, May 22, 1896.

The wood is impregnated with a solution of cellulose, and, it is stated that it can be moulded under heavy pressure without disintegration, so that it is applicable for the preparation of "pressed wood carvings."—V. C.

[Plaster.] Process for the Production of a Material for making Floors, Plastering Ceilings, Walls, Roofs, and the like without Joints or Chinks. O. Kraner, Einsiedel, near Chemnitz, Saxony. Eng. Pat. 29,654, Dec. 24, 1896.

A solution of magnesium chloride is mixed with paper pulp "residue," filling matter is added, then carbonate of

magnesia, and lastly water. The usual proportion is 60 per cent. of magnesium chloride, 10 per cent. of "paper or asbestos leavings or residue," and 30 per cent. of water.

—V. C.

Wood, An Improved Process for Increasing the Density of. C. A. Allison, London. From F. P. Stone, Chicago. Eng. Pat. 2085, Jan. 26, 1897.

"A HARD, compressed, waterproof, wooden block, consisting of an integral structure composed of solidified cotton-seed oil, and the naturally united fibres of the wood transversely flattened and compacted, and having their natural length and direction, the flattened fibres and their intermixed cotton-seed oil being substantially as set forth."—V. C.

Glazed Bricks, and Manufacture of the same; Impts. in. W. D. Cliff, Leeds. Eng. Pat. 2402, Jan. 29, 1897.

IN addition to the glaze on the face, a glaze about ½ to ¾ in. wide is formed on the edges or sides of the beds and ends adjoining the coloured and glazed face.

The face of the brick is first dipped in a colouring body, the body being then scraped "off the adjoining edges of the beds and ends." Afterwards the face of the brick is dipped "from about ½ to ¾ in. below the surface of the glaze bath, so as to leave a coating of glaze of this width on the adjoining sides of the beds and ends."—V. C.

Cement and Concrete, Impts. in. W. S. Robinson, Hants, Nova Scotia, and S. M. Brookfield, Halifax, Nova Scotia. Eng. Pat. 4048, Feb. 15, 1897.

To earth, gravel, broken rock, or the like, are added metallic iron or ferrous salt, alum, Epsom salts, and silicate of soda. A hydraulic cement is thus said to be formed, capable of setting under either salt or fresh water.—V. C.

Bricks, Tiles, Earthenware Pipes, Pottery-Ware, Cement, Lime, Gypsum, and Similar Materials; Impts. in Kilns for Burning. John West, Didsbury, and W. J. Jenkins, Manchester. Eng. Pat. 7270, April 23, 1897.

THE inventors claim an arrangement (described in the specification with reference to drawings) "of flues and dampers, whereby fires are maintained and regulated along both sides of the kiln," and "conveying the gases from the top of the chambers to the chimney."—V. C.

X.—METALLURGY.

Gold Mining in Burma, Notes upon. A. H. Bromly. Trans. North of England Inst. of Mining and Mech. Engineers, 1897, **46**, [2], 122—129.

THE principal gold-mining property is that held by the Choukpazat Gold-mining Company, in the Wuntho district, Upper Burma. The lode is a bedded lenticular body in clay-slate, varying from 8 ft. in thickness downwards, the average being about 3 ft.; it is about 200 ft. deep. The country rock of clay-slate is much impregnated with chlorite, and also, in places, the quartz itself. The quartz is often heavily mineralised, containing iron and copper pyrites, arsenical pyrites, galena, blende, and a tin-white mineral, stated by Saville Shaw to be tellurium or a telluride. This latter is always associated with gold, and consequently enriches the stone. Schistose filling sometimes replaces the quartz.

A ten-stamp, 800-lb., high mill is used, with bins at the back, grizzly, spalling-floor, &c., and the power is furnished by a 40 horse portable Robey engine. The drop is 7 in.; depth of discharge, 8 to 10 in.; number of drops, 90; mesh 30, "or hole 0·021 in. square." The output is about 2·2 tons per stamp per 24 hours, and duty of stamps about 1·5 cwt. per effective horse-power per hour. These conditions of milling gave the best results with a decidedly refractory ore. From the plates, the pulp passes over two 6-ft. corrugated belt Frue-vanners. The concentrates consist mainly of iron pyrites, containing about 0·7 per cent of copper, a little antimony, and about 2 to 2½ oz. each of gold and silver. Milling returns give an average of 5 per cent. of sulphurets, value as above. Some coated non-amalgamable gold also escapes the battery. Returns over about 3,000 tons milled, shows the value of the ore to be about 10 dwt. of fine gold, or a little over 11 dwt. of bullion per ton. The tailings vary from 2 to 1½ dwt., and may be averaged at 3 dwt.; they are amenable to cyanidation. The average percentage recovered by amalgamation is 51 per cent.; locked up in concentrates, 20 per cent., and in tailings, 29 per cent. It is probable that concentrating may be dispensed with and direct cyaniding of tailings adopted.

The total working costs are: mining, 7·15 rupees; transport, 0·75; milling, 4·00 rupees per ton. Total, about 12 rupees or 14s. per ton.

The output from this mine is at present only 300 tons per month, but it is hoped that an output of 900 tons per month will shortly be attained.—A. S.

Gold-Gravel Washing in Eastern Siberia. E. D. Levat. Eng. and Mining J. 1897, **63**, [14], 329.

THE introduction of the sluice for washing gold-bearing gravels is gradually proceeding in Eastern Siberia. In the Trans-Baikal it is little employed, but in the Amour province it is quite common, and in the placers worked by the Zeya Company, it is used to the exclusion of other appliances.

In the placers worked by the Zeya Company, the sluices are 29·82 m. in length, of which 21·3 m. are enclosed in a kind of cage to prevent the stealing of gold, nearly all the metal being obtained from the riffles of the upper part. The bottom of the sluice is furnished with riffles of iron, where the gravel is coarse; when the alluvium is of fine gravel or sand, the bottom is of iron, perforated with holes 15 to 20 mm. in diameter. The average fall is 6°. A sluice of this kind, 0·71 m. in width, washes about 600 cb. m. in 10 hours, and the water required is about eight times the bulk of the gravel washed. If much clay be mixed with the gravel, lumps not broken up and dissolved are carried away with the stones and pebbles, and the gold contained in them is lost. The author suggests as improvements: an increase of the length of the sluice, use of a larger quantity of water, and the introduction of mercury in the riffles or boxes near the tail of the sluice. A washer used in the Amour region is also described. It is driven by a water-wheel, which imparts a rotary motion to the washer itself. This washer is used to treat the gravel directly from the placer and also for a second treatment of the tailings. It treats about 160 c.m. in 10 hours. The author suggests that the washer would be improved by the addition of riffles and mercury to the sluice, to catch the fine gold.—A. S.

Uranium, Preparation and Properties of. H. Moissan. Bull. Soc. Chim. 1897, **17**, 266—271.

URANIUM may be obtained readily from the double chloride of uranium and sodium, either by electrolysis or by reaction with sodium; but it is best prepared by the reduction of the oxide with carbon in the electric furnace. In the pure state its properties resemble those of iron: it may be filed, it combines with carbon, and may be hardened by quenching. It is, however, more oxidisable than iron, finely powdered uranium slowly decomposing water even in the cold. It may be obtained crystalline, and, if pure, it is quite white. A yellowish shade indicates the presence of nitrogen, with which it combines so readily that special precautions must be taken in preparing the metal in order to avoid the action of this element. Uranium, when free from iron, exerts no influence upon the magnetic needle, and as a metal it is more volatile than is iron in the electric furnace.—W. G. M.

Metals, Precipitation of. J. B. Senderens. Bull. Soc. Chim. 1897, **17**, 271—286.

THIS is a continuation of the author's previous work with nitrate solutions (Bull. Soc. Chim. **15**, 208, 691, 991; this Journal, 1893, 764, and 1895, 370), the action of metals upon solutions of sulphates, chlorides, and acetates of silver, copper, and lead being now examined. It has hitherto been stated in general terms that a certain metal is capable of precipitating certain other metals from their solutions; but it is now shown that such generalisations are unsafe, as the possibility of precipitation depends upon the

acid of the dissolved salt. Thus, neither tin nor iron will reduce the acetates of silver or copper; bismuth will act upon the acetate, but not upon the sulphate, of silver; aluminium precipitates silver sulphate completely, but is almost without action upon the acetate; and again, it precipitates cupric chloride very rapidly, whilst it attacks the sulphate very slowly, and the acetate even more slowly. The degree of dilution also causes similar variations in the behaviour of the metals: a metal which precipitates another very rapidly from a solution of a certain strength may react slowly, or even be quite inactive, with another of different strength; lead in cupric chloride, and zinc and aluminium in lead acetate, are cases in point. It has been previously shown that when metals were precipitated from their nitrates a variable amount of nitric acid was decomposed, and, as a result, Richter's law was never found to be verified. With the salts now tried, the acid remains unchanged, but the water is in some cases split up, hydrogen being evolved and a portion of the precipitating metal being oxidised and forming basic salts. Zinc and aluminium afford good examples of this action. Occasionally (*e.g.*, in the case of tin and bismuth) an acid salt is formed and remains in solution, whilst a basic salt or an oxide is precipitated.

It would seem that if this action of the acid on the water were eliminated, Richter's law should hold good; but account must be taken of the tendency, in some cases, to form alloys. Cadmium, placed in solutions of silver or copper, gives precipitates which absorb a considerable proportion of the precipitating metal. Other metals also form alloys, but in very small proportions. Zinc, alone, acting upon lead acetate, gives a precipitate of pure lead. It is found that out of 90 cases examined, Richter's law is only approximately true in about 10 cases, and true in one. In the other 80 cases either the weight of deposit was incorrect, or else no deposition took place at all, or not after the first instant. Richter's law is, therefore, not generally applicable, being affected by more or less complex reactions.

It may be specially noted that the author, confirming and extending his earlier work, shows that whilst annealed iron and steel readily attack silver nitrate solution, cold-worked iron and quenched steel are without action, which tends to indicate the existence of allotropic modifications of iron. This is observed with all specimens, whether roughly cleaned or smooth polished with rouge. Slight action is sometimes observed in the case of worked specimens, if the cut ends of the bar be exposed to the solution, as though the inner portions of the metal were different from the exterior; but if the ends of such bars be coated with vaseline no action will be apparent. The action of the annealed iron upon the solution varies with the temperature and duration of the annealing. With silver sulphate both kinds of iron can react, but the annealed bars act more vigorously. With silver acetate and cupric chloride or sulphate both classes of iron behave alike.—W. G. M.

Aluminium Bronze, The Use of. Imp. Inst. J., May, 155.

UNTIL quite recently aluminium bronze was used chiefly in the manufacture of cheap jewellery, for which purposes its capacity for taking a good polish and of retaining its colour, render it peculiarly suitable. In a paper communicated to the American Institute of Mechanical Engineers (*Engineering*, January 29th, 1897), Dr. Waldo points out that it is a very suitable material for the manufacture of the seamless tubes for water-tube boilers, for which brass and steel are now commonly used. The author has examined a series of alloys of aluminium and copper, and he has obtained evidence of the existence of the following definite chemical compounds:—

Cu_2Al.	Cu_5Al.	$Cu_{10}Al$.	$Cu_{16}Al$.
Cu_3Al.	Cu_6Al.	$Cu_{12}Al$.	$Cu_{18}Al$.
Cu_4Al.	Cu_8Al.	$Cu_{14}Al$.	$Cu_{32}Al$.

The evidence which Dr. Waldo adduces as proof of this may be summarised as follows:—

I. A rise of temperature occurs when the molten metals are mixed.

II. The alloys are crystalline when carefully made.

III. Chemical analysis and microscopical examination show them to be homogeneous.

IV. There is no "sweating out" of either metal when the alloys are worked at a red heat.

Of these alloys he recommends those having the composition Cu_8Al or $Cu_{10}Al$ as most suitable for industrial use.

A comparison of the tensile strengths of aluminium bronze with those of Swedish open-hearth steel and brass, at different temperatures, gave the following results:—

Temperature of Experiment.	Aluminium Bronze.	Brass.	Swedish Open-Hearth Steel.
15° C.	96,000	81,900	79,300
980° C. to 1,010° C.	6,100	2,770	17,500
1,300° C. to 1,400° C.	4,100	250	1,950

From this table it will be seen that the tensile strength of aluminium bronze is very high, and this, taken with the fact that it stands great strains without undergoing molecular rearrangement, ought to gain for it an extensive application in the arts.

Tungsten-Iron, Alloy of. T. H. Norton. J. Amer. Chem. Soc. 1897, **19**, 110.

THE author describes an alloy which, when analysed according to Ziegler's method (Dingler's Polyt. J. **274**, 513) was found to contain 93·4 per cent. of tungsten and 6·18 per cent. of iron. The specimen had a lustrous appearance, a hardness of 7, and, when powdered, a specific gravity of 14·55.—J. L. B.

Nickel, Estimation of, in Nickel Steels. J. Spüller. Chem. Zeit. 1897, **21**, 243.

See under XXIII., *page* 470.

Pig Iron, Determination of Sulphur in. A. A. Blair. J. Amer. Chem. Soc. 1897, **19**, 114.

See under XXIII., *page* 469.

Silicon, A Rapid Method for the Determination of, in Silico-Spiegel and Ferro-Silicon. C. B. Murray and G. P. Maury. J. Amer. Chem. Soc. 1897, **19**, 138.

See under XXIII., *page* 469.

PATENTS.

Steel or Alloys of Iron, Impts. in the Manufacture of. R. A. Hadfield, Sheffield. Eng. Pat. 7422, April 7, 1896. (Under International Convention.)

NON-MAGNETIC alloys of iron, nickel, manganese, and carbon, of high electrical and thermal resistance, tough, and of great tensile strength, which can be forged, rolled, or cast, are prepared by adding to pig-iron, cast or wrought iron, steel or iron scrap, or a mixture of these, either direct or by means of previously prepared alloys or mixtures, from 3 to 11 per cent. of manganese, 8 to 25 per cent. of nickel, and carbon up to $1\frac{1}{2}$ per cent.—J. H. C.

Gold, Silver, and other Precious Metals, Impts. in Apparatus for the Recovery of. S. Vivian, Southall. Eng. Pat. 8967, April 28, 1896.

REVOLVING drums or barrels of hexagonal or polygonal section, and furnished upon their inner circumference with radial partitions lined with flat or corrugated plates, preferably of copper, are used as amalgamators instead of the usual drums with curved plates.—J. H. C.

Zinc, Impts. in the Extraction of, from Ferruginous Liquors, specially applicable to the Treatment of the Spent Liquors of Copper Works. J. R. Wylde, J. W. Kynaston, and J. Brock, Liverpool. Eng. Pat. 10,494, May 15, 1896.

THE ferruginous spent liquor is taken hot from the copper precipitating tanks into a series of cisterns similar to Weldon chlorine stills, and calcium carbonate is added sufficient to

neutralise the free acid, and then in further proportion equivalent to the zinc present in the liquor. Hydrogen sulphide is passed through the series of cisterns (which are provided with agitators) in such a manner as to make the process continuous. When a batch of the liquor has been saturated with the gas, sufficient spent liquor is added to react with the excess of hydrogen sulphide, and, after subsidence, the precipitate of zinc sulphide and calcium sulphate is drained and washed free from iron salts, and is then heated in a Weldon still with hydrochloric acid, the hydrogen sulphide evolved being utilised in the first-described process. The contents of the still are then filtered from the undissolved calcium sulphate, and heated with milk of lime to obtain zinc hydroxide, which may be treated in any desired way. If iron be present in the zinc chloride solution, bleaching-powder solution may be added to peroxidise the iron, and then calcium carbonate in slight excess. The solution, after filtering off the ferric hydroxide, may then be precipitated as before.—E. S.

Sulphide of Aluminium, which may or may not be combined with other Sulphides (or Sulphurets), A Process of Manufacture of. [*Obtaining Aluminium.*] D. A. Peniakoff, Huy, Belgium. Eng. Pat. 11,555, May 27, 1896.

A SINGLE or double anhydrous aluminium salt, such as a fluoride or chloride, fused with the sulphide of an alkali or alkaline earth, together with a chloride or fluoride as a flux, yields a mixture containing aluminium sulphide, from which, in the same crucible, aluminium may be reduced, "either by the action of an electric current or by means of reducing gases (such as hydrocarbons, for example)."

The following formulæ present examples of the preparation of aluminium sulphide :—

$$(1.)\ Al_2F_6 + 3Na_2S = Al_2S_3 + 6NaF.$$

$$(2.)\ Al_2F_6, 6NaF + 3Na_2S = Al_2S_3 + 12NaF.$$

$$(3.)\ Al_2Cl_6, 6NaCl + 3Na_2S = Al_2S_3 + 12NaCl.$$

When the bath is constituted according to the reactions (1) or (2), if aluminium be reduced from the fused mass, the supply of aluminium sulphide is kept up by additions of aluminium sulphate and carbon; aluminium fluoride and sodium sulphate are first formed, according to the equation $Al_23SO_4 + 6NaF = Al_2F_6 + 3Na_2SO_4$, and the sodium sulphate is reduced to sulphide by the carbon, with subsequent production of aluminium sulphide according to equations (1) and (2). It is stated that "even where the sulphide of aluminium produced in the bath is subjected to electrolysis, the sulphide alone may undergo the electrolytic action, and not the fluoride, as the latter requires for its electrolytic decomposition, a far greater electro-motive force than that which suffices to bring about the decomposition of the sulphide of aluminium. Besides, where electrolysis is resorted to, the sulphur separated from the aluminium serves, for the most part, to reduce the alkaline sulphate to the condition of alkaline sulphide, thus adding its effect to the action of the carbon."—E. S.

Aluminium and Aluminium Alloys, Impts. in the Manufacture of Rods and Tubes mainly from. A. G. Brown, Manchester. Eng. Pat. 14,330A, June 29, 1896.

THE molten metal is poured into the chamber of a press and allowed to cool until it has attained a temperature of about 650° C., when it is forced through a die of the required form by means of a plunger. This system of treatment is claimed.—J. H. C.

Blast Furnaces, Impts. in, and in the Mode and Means for Charging the same. T. Lewis, Shifnal. Eng. Pat. 10,061, May 12, 1896.

THIS invention is intended to lessen the escape of gases or heat while charging, and to prevent the entrance of cold air; also to reduce the chances of explosion, and to prevent injury to the furnace lining, bell, and ring, and irregular charging, by the swaying of the bell.—J. H. C.

Sheet Iron and Steel, Impts. in the Treatment of, in the Manufacture of Polished Sheets. J. D. Grey, Bridgend, and T. Gwynne, Briton Ferry. Eng. Pat. 94, Jan. 1, 1897.

THE sheets, after leaving the pickling and washing tanks, are dipped into lime water and passed through a drying stove; they are then cold-rolled and annealed, and finally passed slowly through a furnace heated to dark cherry-redness to produce a thin film of oxide, which prevents rust.—J. H. C.

Ore-Briquettes, Process of Treating Granular or Pulverulent Iron Ores and Slag or other Residues containing Iron to facilitate the Extraction of the same. S. Neffgen, Mühlheim-on-the-Rhine. Eng. Pat. 4309, Feb. 17, 1897.

THE materials are moistened with calcium oxide or mixed dry with calcium hydroxide, pressed in moulds, and heated in a moist atmosphere for 4 to 6 days to a temperature of 90° to 100° C.—J. H. C.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(A.)—ELECTRO-CHEMISTRY.

PATENTS.

Carbon [Electro-Deposition of], Impts. relating to the Use and Treatment of, in Electro-Chemical or Electrolytic Processes, and to Apparatus therefor. Dr. A. Coehn, Göttingen, Germany. Eng. Pat. 5584, March 12, 1896.

ADVANTAGE is taken of the discovery that carbon dissolves when used as an anode in strong hot solutions of sulphuric and other acids which give off oxygen at the anode during electrolysis, to employ such solutions as baths for the electro-deposition of carbon on suitable cathodes. Under these conditions also, carbon may be employed as a soluble electrode in a battery, peroxide of lead or other suitable substance forming the negative element, and such battery is reversible and capable of being recharged.—G. H. R.

Secondary Voltaic Batteries, Active Material for [Lead-Antimony Backing], An Impt. in. G. A. Grindle, Prestwich. Eng. Pat. 10,472, May 15, 1896.

THE claim is for "active material for secondary voltaic batteries in form of strips, sheets, rods, or other pieces of composite metal, each consisting of a layer of lead and a layer or core of alloy of lead and antimony.—J. C. R.

Galvanic Batteries, Dry Cells for, Impts. in. T. Clark, Greenwich. Eng. Pat. 11,212, May 22, 1896.

THE inventor claims the employment in galvanic batteries (Leclanché type described) of a mixture of gum tragacanth (3 parts) and (1 part) sawdust or the like.—J. C. R.

Electrical Batteries [Leclanché], Impts. in. D. Mathieu, Buckfastleigh. Eng. Pat. 20,035, Sept. 10, 1896.

IN lieu of the ordinary porous pot in the Leclanché cell, the inventor illustrates and claims "a carbon pole composed of a number of carbon pencils clamped between recessed carbon discs [forming a "cage"] and containing manganese and gas carbon in the approximately cylindrical space between such pencils and discs."—J. C. R.

Primary Electric Battery, An Improved. O. Rothmund and E. von Burgwall, Vienna, and L. Ofenschüssl, Prag, Austria. Eng. Pat. 27,262, Dec. 1, 1896.

THE electrodes are peroxide of lead, of any form, and amalgamated zinc, aluminium, magnesium, or iron . . . "to carry the element dry to the place of use, and not to carry corrosive liquids or salts, the positive electrode is saturated in a liquid (like concentrated sulphuric acid, potash, soda, or chromic acid), so that the electrolyte is produced by the positive plate (electrode) by the application of pure water." To protect these plates against moisture of the air, they are

wrapped in gutta-percha paper or lead foil. The plates being regenerated electrolytically or in a chemical way, they are freed from moisture in drying chambers by chloride of calcium or other hygroscopic substance before being saturated with the aforementioned concentrated solutions. —J. C. R.

Batteries, Secondary: Impts. in. A. Heil, Fränkisch Crumbach, Germany. Eng. Pat. 29,306, Dec. 21, 1896.

The claim is for "A secondary battery cell in which a reduced and chlorinated peroxide of manganese and carbon electrode is placed in a chlorine-containing electrolyte opposite to a lead electrode that is covered with powdered lead, the arrangement being such that the two electrodes alternately give out and take up electrical energy by their chlorination and reduction, according as they are discharged or charged."—J. C. R.

Batteries, Galvanic; Impts. in. W. S. Doe, Brooklyn, U.S.A. Eng. Pat. 995, Jan. 13, 1897.

This zinc-carbon battery is designed mainly for lighting bicycle lamps, being readily rechargeable. "The invention consists principally of a perforated tube [hard-rubber], adapted to extend into the exciting fluid [sulphuric acid], and adapted to receive a depolariser" [fused sodium nitrate "cartridges"], and "of certain parts, and details and combinations of the same," fully described.—J. C. R.

Electric Accumulators, Impts. in the Manufacture of Positive Lead Electrodes for. [Improved Planté.] H. O. Tudor, Luxemburg. Eng. Pat. 10,718, May 18, 1896.

The plates are placed as positives in very weak sulphuric acid of 2° B., and charged at first with intervals of rest, and then of discharge, until they are coated with a sufficiently thick and hard layer of mixed sulphate and peroxide of lead, the process occupying from 15 to 20 days. The plates are then arranged as negatives, and the coating reduced to spongy lead, after which they are placed as positives in strong acid and formed in the usual way.—G. H. R.

Chlorine, Impts. relating to the Electrolysis of Soluble Salts, especially applicable for the Production of. [Reduction of Oxychlorides.] A. J. Outhenin-Chalandre, Paris, France. Eng. Pat. 15,834, July 17, 1896.

In order to prevent the formation of oxygen compounds, such as oxychlorides, at the anode during the electrolysis of salt solutions, hydrogen or hydrochloric acid is added continuously to the bath, so that the hydrogen combines with the oxygen, with the formation of water and the evolution of pure chlorine.—G. H. R.

Electric Accumulators [Planté Type], Impts. in or connected with. J. Julien, Brussels, Belgium. Eng. Pat. 21,956, Oct. 3, 1896.

The object is to produce accumulators of large surface and capacity. The claims are :—

"1. In a Planté accumulator, a positive electrode formed by the horizontal superposition of an indefinite series of finely grooved or corrugated rolled sheets of lead or lead alloys, having any suitable contours, with or without a fine coating of active material [waste from worn-out accumulators may be utilised], applied to assist the formation, and electrically connected together and with the terminal by bands of lead enclosing the block of superposed plates."

"2. In a Planté accumulator, a negative electrode consisting of cylinders or moulded prisms of litharge or other suitable plumbiferous compound perforated through their axis and strung on conducting rods so as to form unalterable electrodes for accumulators of great power."

3. Further, "the assembling of the electrodes in a single body by providing the positive electrode with a series of vertical channels or passages, and inserting in the latter a corresponding number of pencils of negative material connected in a group, with the object of reducing to a minimum the space occupied by the battery and the quantity of electrolyte in which it is immersed."—J. C. R.

(B.)—ELECTRO-METALLURGY.

PATENTS.

Wire, Impts. in the Method of Electrically Coating, and Apparatus therefor. [Continuous Deposition.] W. S. Rawson, London. Eng. Pat. 6069, March 18, 1896.

The apparatus consists of a roller formed of two end frames resting on insulated bearings, having centres for a number of longitudinal spindles carrying grooved pulleys made of porcelain or other insulating material, and placed side by side. In the upper parts of the frames are bearings for two grooved rollers, the lower one of which is metallic, and suitably connected to the negative terminal of a dynamo. The wire, which forms the cathode, is wound round the pulleys and the grooves of the metallic roller, against which it is pressed by caoutchouc rings on the upper roller, and as the rollers revolve, the wire is fed forward. This apparatus is immersed so that nearly the whole surface of the wire is covered by the electrolyte, and two anodes are employed, one of which is external and stationary, while the other, which is inside the pulleys, revolves, and may be formed with blades, or be a metallic basket containing scraps of the metal to be deposited. The anodes are so arranged that equal quantities of metal are deposited on both sides of the wire.—G. H. R.

Copper, Impts. in Apparatus for Depositing on Printing Rollers and the like. H. Wylie and J. Grant, Renton, Dunbarton. Eng. Pat. 8326, April 21, 1896.

A vertical rotating anode of copper is placed centrally in the electrolytic vat, and surrounding it, and suspended from an annular frame, are the rollers which are to receive the deposit. By means of gearing, the rollers are caused to revolve on their own axes, and the annular frame may also rotate around the anode, and preferably in an opposite direction.—G. H. R.

Metals, Impts. relating to the Coating, Covering, or Amalgamating of, with Metals, by the Aid of Electricity, and to Apparatus therefor. [Plating Ships' Hulls.] F. H. Snyder, Westcombe Park, Kent. Eng. Pat. 25,360, Nov. 11, 1896.

The method is applicable to the coating of the interior and exterior of iron and steel ships, bridges, and other metallic structures with metals or alloys to protect them from oxidation and corrosion. It consists in connecting the object to be protected to the negative terminal of a dynamo, while applying to its surface, as in painting, a brush about 6 ins. wide and $1\frac{1}{2}$ ins. deep, formed of asbestos or other suitable material, and provided with an insulating handle, through which connection is made to the positive terminal of the dynamo. The contact surface must be small, and the brush contains an anode of copper or the alloy which is to be deposited, and is saturated with an electrolyte of the same composition as would be used in ordinary electro-plating to deposit the metal or alloy. The electro-motive force employed is as high as practicable, and the brush must be kept constantly moving, or the deposit will be burnt and pulverulent. In the deposition of copper, an E.M.F. of 25 to 30 volts is employed, and a current of 10 to 15 ampères. The process can also be used for coating plates, electrodes, or cells for electric batteries with platinum or gold, or other suitable metal or alloy. —G. H. R.

Metallic Alloys [Ferromanganese, &c.] and Pure Metals, Process for the Extraction of, by Electric Heat. [Sulphide of Antimony Flux.] H. Aschermann, Cassel, Germany. Eng. Pat. 859, Jan. 12, 1897.

This process consists in the extraction of metallic alloys and pure metals by electric heat from a mixture of the oxide of a metal and the sulphide of another metal or of a metalloid, or of the oxide of a metalloid and the sulphide of a metal, the object being to accelerate the reduction, and eliminate the carbon and other impurities. The metal is obtained in the form of an alloy, from which the more fusible metal can be separated by distillation if desired. Sulphide of antimony is preferably employed as the flux;

for instance, in the preparation of chromium, 10 parts of chromium oxide are mixed with 23 of sulphide of antimony and placed as cathode in an electric fusion furnace, and treated by a current of 20 to 25 ampères, and the antimony is driven from the resulting alloy by reheating. Similarly, aluminium may be obtained from a mixture of 10 parts of its oxide with 37 of sulphide of antimony; and directions are also given for the preparation of ferrochromium, ferromanganese, and ferrocerium.—G. H. R.

Metals and Alloys, An Impt. in the Electro-Deposition of. [*Phosphor Bronze.*] G. A. Webb and W. A. Thoms, London. Eng. Pat. 1657, Jan. 21, 1897.

According to this process, phosphorus is deposited electrolytically in combination with platinum, antimony, or their alloys, on metals or alloys of metals, or other suitable materials. The anode is formed of the phosphor metal or compound which it is desired to deposit, and the baths are composed of solutions of one or more of the metallic salts required to furnish the electro-deposit. Instructions for the preparation of these baths are given.—G. H. R.

XII.—FATS, OILS, AND SOAP.

Wool-fat, The Action of Sulphuric Acid on some Alcohols occurring in. E. v. Cochenhausen. Dingler's Polyt. J. 1897, **78**, [303], 283—285.

The action of concentrated sulphuric acid on cetyl alcohol, ceryl alcohol, and cholesterol was investigated. The cetyl alcohol was prepared from spermaceti, and had a melting point of 49·3° C.; the ceryl alcohol was prepared from Chinese wax, and melted at 79° C., whilst the cholesterol melted at 143 C. The alcohols were dissolved in petroleum spirit, and about five times the quantity of concentrated sulphuric acid added, with constant shaking, the mixture being kept cool by cold water. If the mixture be kept thoroughly cool, the reaction takes place without development of sulphur dioxide. After standing for 24 hours, the mass was poured into a large quantity of cold water, more petroleum spirit added, and the whole well mixed. The petroleum spirit solution of the alkyl sulphates, separated from the dilute acid, was neutralised with alcoholic soda, the petroleum spirit distilled off, and the residue dried in the water-bath and extracted with absolute alcohol to remove the sodium sulphate. The extract, after distilling off the alcohol, yielded by crystallisation from 50 per cent. alcohol, the pure sodium salts of the alkyl sulphuric acids. The calcium and barium salts were prepared by treating a solution of the sodium salt with calcium chloride or barium chloride. They separated in flocks, and were obtained pure by crystallisation from 50 per cent. alcohol.

Cetyl Sulphuric Acid and its Salts.—The acid, which is with difficulty soluble in water and dilute sulphuric acid, but more easily in dilute alcohol, was not prepared in the free state. On evaporation of its solution in the water-bath, decomposition occurs, a black wax like residue being left behind. The sodium, calcium, and barium salts form, on crystallisation from dilute alcohol, white needle-shaped crystals without water of crystallisation, which blacken at 100° C. with partial decomposition. The sodium salt is soluble in acetone, the calcium and barium salts insoluble. Analysis of the barium salt showed 17·42 per cent. of barium, corresponding to the formula $C_{32}H_{66}S_2O_8Ba$, which requires 17·58 per cent. of barium.

Ceryl Sulphuric Acid and its Salts.—The free acid behaves towards solvents, and on heating, like free cetyl sulphuric acid. The sodium salt forms a thick paste with boiling water, which, on cooling, becomes thin. From absolute alcohol, the salt separates in the form of a crystalline powder: from dilute alcohol, in a flocculent mass, consisting of fine needles. The sodium salt is also soluble in acetone. The calcium salt is but slightly soluble in water, but equally soluble in hot and cold absolute alcohol; from 50 per cent. alcohol, it separates in flocks, which consist of fine needle-shaped crystals. The barium salt is insoluble in cold and hot water, and is only slightly soluble in hot and cold absolute alcohol. The calcium and barium salts are not soluble in acetone; they are converted by ether and petroleum spirit into pasty masses. Analysis of the sodium salt gave an average of 6·255 per cent. of Na_2O, corresponding to the formula $C_{27}H_{55}SO_4Na$, which requires 6·23 per cent. of Na_2O.

Separation of the Cetyl and Ceryl Alcohols from the Sulphuric Acid Compounds.—The alcohols were separated from the sulphuric acid compounds by boiling with dilute hydrochloric acid, and, after recrystallising, showed the correct melting points: cetyl alcohol, 49·3° C., and ceryl alcohol, 79° C. The decomposition takes place in a few minutes, if the acid compound be moistened with acetone before adding the hydrochloric acid.

Action of Concentrated Sulphuric Acid on Cholesterol.—Cholesterol does not form an acid compound by the action of sulphuric acid, but is probably converted into cholesterones (Zwenger, Beilstein's Handbuch, III. Aufl.; and Mauthner and Suida, Monatsh. für Chemie, **17**, 29). These are easily soluble in alcohol, ether, petroleum spirit, and acetone.

The treatment of the alcohols of wool fat with concentrated sulphuric acid has not yet led to any definite results, except that saturated alcohols were obtained after removal of the cholesterones. The author is of the opinion that the treatment with sulphuric acid may be used as a means of separating the saturated alcohols from the unsaturated ones.—A. S.

Wool Fat, Purification of. [*Lanolin Manufacture.*] C. Baron. Rev. Chim. Ind. 1897, **8**, 111.

800 kilos. of the crude grease are placed in an autoclave with 100 litres of caustic soda of 3·25° B., and boiled with live steam for three hours at a pressure of 2 atmospheres. 200 kilos. of water and 300 of acetic ether are next introduced, and the agitation is continued for eight hours at a pressure of 4 atmospheres. The whole is then allowed to rest, but until the liquids have separated, the temperature is maintained. The ethereal layer is drawn off, and washed with water, which is at first made slightly alkaline and warmed to 60°. It is finally filtered, the acetic ether distilled off, and the residue sold as "lanolin." The aqueous liquid is decomposed with sulphuric acid, the fatty acids washed and made into soap.—F. H. L.

Olive Oils, Partial Solidification. A. Goldberg. Chem. Zeit. 1897, **21**, 263.

The following series of iodine numbers were obtained from the liquid (α) and semi-solid (β) portions of olive oils taken from barrels, which had for some time been stored in cold cellars. A, B, C, D, E were genuine salad oils; F, H, and I were officially denatured with orange oil, clove oil, and rosemary oil respectively. G was also denatured with rosemary oil and gave evidence by Becchi's test that it contained cottonseed oil; also 100 c.c. which had been frozen hard melted at the ordinary temperature in a much shorter time than pure olive oil under the same conditions.

Iodine Numbers.

Aα	80·9; 81·0; 81·0.	Eβ	82·8; 82·6.
Aβ	80·2; 80·2; 80·3.	Fα	84·6; 84·5.
Bα	82·3; 82·5; 82·3.	Fβ	84·2; 84·3.
Bβ	81·6; 81·5; 81·7.	Gα	88·1; 87·9.
Cα	81·9; 82·0; 82·0.	Gβ	88·4; 88·2.
Cβ	81·2; 81·4; 81·2.	Hα	.. 84·5.
Dα	84·0; 83·9.	Hβ	85·6; 85·3.
Dβ	82·2; 82·4.	Iα	83·9; 83·8.
Eα	83·4; 83·6.	Iβ	83·2; 83·0.

All the liquid fractions (α) were kept during the winter at a moderate temperature, and for the most part they set hard. On separating the liquid from the solid portions, the iodine numbers of the two fractions were about the same. The author considers it remarkable that the above iodine numbers of the fractions show such slight differences; and, as will be seen in the cases of G and H, higher values were obtained in the solid portions, although neither were pure products.—J. L. B.

F

Olive Oil and Cotton-Seed Oil, On the Behaviour of Mixtures of, in the Cold. A. Goldberg. Chem. Zeit. 1897, **21**, [31], 304—305.

Olive oil, when exposed to low temperatures, becomes turbid at + 2° C., and at − 6° C. deposits 28 per cent. of solid matter. Cotton-seed oil similarly treated becomes turbid at about 12° C., and completely solid at 0° to − 1° C. In order to completely melt the frozen oils, the temperature must rise a little above the respective turbidity points. Mixtures of olive oil and cotton-seed oil, however, after having been exposed to temperatures of from − 15° to − 20° C. require a much shorter time to become fluid again, and do so at a lower temperature than the individual oils. Thus pure olive oil exposed for 2¾ hours at − 16° to − 20° C., and then allowed to stand at the ordinary temperature, began to melt after 55 minutes, and was completely liquid after 185 minutes, when the temperature had risen to + 11°·5 C. A mixture of 95 per cent. of olive oil with 5 per cent. of cotton-seed oil began to melt after 50 minutes, and was completely fluid after 162 minutes (temperature + 12°·5). A mixture of 50 per cent. of each oil showed signs of melting after 20—35 minutes, and was fluid in 95 minutes, the temperature being + 8·5° C. This behaviour is illustrated by long tables of results, and the time and temperature of melting are shown to stand in fairly constant ratio to the proportion of cotton-seed oil in the mixture. It is further shown that the results are practically the same whether the oils be exposed to the low temperature for one hour or longer.

Several determinations were made of the iodine numbers of the original mixtures, of the portions first becoming liquid, and of the more solid residue remaining in the tube, but the three results were in every case practically identical. Nor did a further fractionation by cold show any increase in the iodine number of the portion first becoming liquid.
—C. A. M.

"Orsin Oil," A New Lubricant from Herring Fat. J. Landin. Chem. Zeit. 1897, **21**, 265—266.

Up to the present, the experiments carried out to determine the value of herring oil as a lubricating agent have not been satisfactory. The author has investigated the so-called "orsin oil," which is derived from the fat of the herring. This oil has a specific gravity of 0·924 to 0·928 at 15° C., an acidity of 0·326 to 0·42 per cent. of oleic acid, and contains from a trace to 0·026 per cent. of mineral matter. Unsaponifiable oils and water were not present. The flashing point was 262° to 265° C. The new oil is satisfactory on account of its low acidity, and compares favourably in this respect with commercial olive oils, which sometimes contain as much as 30 per cent. of oleic acid. The author has further examined the so-called "refined animal oil," also said to be a lubricant, which is obtained from purified herring oil. It is said to be free from foreign matter and to have an acidity of 0·35 per cent. of oleic acid. Both these oils, it is stated, are rich in the glycerides of oleic and stearic acid and their homologues, whilst the glycerides of physetoleic and jecoric acid were present only in small quantities. No iodine was detected in the purified oils, but traces were found in such as were unrefined. Crystals of neutral fats were deposited at 15° C., the oils resembling, in this respect, neatsfoot oil. For the purpose of comparison, the viscosity numbers of these two oils were determined together with some authentic specimens of other oils.

The following values were obtained, it is said, with an Engler's viscosimeter:—Refined rape-seed oil, 14·5; "orsin oil," 11·4; olive oil from Malaga, 11·9 and 10·8; olive oil from Messina, 11·7; and "animal oil," 17·1. The determination of lubricating values was carried out with the apparatus of Ingram and Stapfer, in which a weight of 20 kilos. was used, and a speed of 1,260 revolutions per minute maintained until the temperature of the oil had risen from 20° to 70° C. The following numbers of revolutions were recorded: — Malaga Olive oil, 17,600 and 16,600; "Orsin oil," 16,000; Messina Olive oil, 15,100; "Animal oil," 15,200; Refined rape-seed oil, 15,000; and rape-seed oil, 13,000.—J. L. B.

Egg Oil. M. Kitt. Chem. Zeit. 1897, **21**, [31], 303—304.

By extracting with ether the yolks of 40 eggs, 130 grms. of oil were obtained—a yield of 19 per cent. on the yolks taken. The oil, when freed by filtration from some foreign suspended matters, chiefly lecithin, and dried, was of a clear orange colour, partially solidified at the ordinary temperature, and solidified in the elaidin test.

The following analytical data were obtained:—

Specific gravity at 15° C.	0·9144
Acidity value	1·2
Saponification number	{ 190·3 / 190·0
Ether value	189·0
Iodine value	{ 72·2 / 72·0
Hehner value	95·16
Melting point of fatty acids	36°—39° C.
Saponification number of fatty acids	{ 194·0 / 195·8
Iodine value of fatty acids	{ 72·9 / 74·6
Acetyl acid value	189·7
„ saponification value	201·6
„ value	11·9
Reichert-Meissl value	0·4
Glycerin (per cent.)	10·4
Lecithin „	0·2
Cholesterol „	1·5
Mean molecular weight of fatty acids	285·0

By calculation from the formulæ given by Benedikt, Anal. der Fette, pp. 136, 139, and 141, the author came to the conclusion that the percentage composition of the "total fatty acids" of the oil was:—Oleic acid, 81·8; palmitic acid, 9·6; oxy acids, 6·4; stearic acid, 0·6; and cholesterol, 1·6.—C. A. M.

Linseed Oil, Differences between Boiled and Raw. G. Morpurgo. Giorn. di Farm. di Trieste, 1896, **1**, 361; Chem. Zeit. **21**, 37.

See under XXIII., *page* 470.

Linseed-Cake Fat, On the Iodine Number of. B. A. van Ketel and A. C. Antusch. Zeits. angew. Chem. 1897, 210.

See under XXIII., *page* 471.

PATENTS.

Detergent, Manufacture of. S. Wohle, London, and A. C. Irwin, Highgate, Middlesex. Eng. Pat. 1978, Jan. 28, 1896.

An infusion of the leaves of *Albizzia amara* in hot water is strained and mixed with a little glycerin, olein, paraffin, spirits of turpentine, and citric or tartaric acid, in the proportion of from ½ to 1 per cent. of each. This mixture is digested, at about 160° F., with the bruised fruits of *sapindaria*, or with the beans of the *Acacia concinna*, or with dried and shelled horse-chestnuts. The extract is filtered through a press, and ammonia added, but not in sufficient quantity to make the mixture alkaline, and finally mixed with an antiseptic to prevent fermentation. It is said to be specially applicable for cleansing wool and other animal fibres.—C. A. M.

Lubricants, Impts. in or relating to the Production of. P. R. de Faucheux d'Humy, Liverpool. Eng. Pat. 5791, March 16, 1896.

About 5 parts of spermaceti and 15 parts of paraffin wax are dissolved in about 48 parts of crude petroleum heated to 250° F., and when the liquid is clear, 20 parts of pitch-pinewood sawdust and 12 parts of soda are added. The whole is then boiled until 10 per cent. of the total mass has evaporated, when 10 per cent. of water is added and the mixture heated gently until a soft paste is formed.
—C. A. M.

Soap, A New or Improved Manufacture of. P. R. de Faucheaux d'Humy, Liverpool. Eng. Pat. 5792, March 16, 1896.

The use is claimed, in the manufacture of soap, "of cellulose substances, and more especially of pitch-pine dissolved in alkali and petroleum."—C. A. M.

Wax for Making Comb Foundation for Bee-Hives, Impt. in the Manufacture of Sheets of. J. H. Howard, Holme, Hunts. From E. B. Weed, Medina, Ohio, U.S.A. Eng. Pat. 6297, March 21, 1896.

By subjecting the material to a high pressure after having first reduced it to a very plastic condition, the patentee claims that the sheet wax is materially altered in physical condition, becoming transparent and not getting dry and hard. The beeswax or other substance is heated to about 150° F., then cooled until plastic, and afterwards pressed. Special plant is described in which the process is carried out.—C. A. M.

Oils and the like, Impts. in or relating to the Liberation or Recovery of. The Fish Utilisation Syndicate, Ltd., and J. C. W. Stanley, London. Eng. Pat. 7269, April 2, 1896.

This invention consists in rendering animal or vegetable fats by boiling with water together with a suitable proportion of sodium silicate or other soluble silicate. The oil is subsequently well washed, to remove all traces of alkali. It is claimed that this treatment breaks open the fat-containing cells and also effects some deodorisation or purification.—C. A. M.

Lubricant, An Improved. C. S. Gilbert, Millersburgh, Penn., U.S.A., and J. F. Thompson, Liverpool, Penn., U.S.A. Eng. Pat. 10,221, May 13, 1896.

Mineral oil, lime water, resin oil, sodium sulphate, black antimony, and magnesium carbonate are mixed together in prescribed proportions. It is claimed that the lubricant will not settle to the bottom of the barrel or become solidified.
—C. A. M.

Soap, Improved, for Carpets. W. P. Thomson, London. From J. van de Bücken and S. Gattfeld, Germany. Eng. Pat. 1208, Jan. 16, 1897.

The soap is prepared by intimately mixing 60 parts of a hot saturated solution of common soap with 12 parts of sal ammoniac and 4 parts of turpentine. To these are added 16 parts of fresh ox-gall, and afterwards 8 parts of acetic ether, 10 parts of sugar, and 3 parts of honey. Finally, the soap is scented. It is claimed that this soap, whilst cleansing the carpets, also brightens and restores colours.—C. A. M.

XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

(A.)—PIGMENTS, PAINTS, &c.

Verona Earth. Ch. of Comm. J., April 1897, 89.

See under Trade Rep., *page* 477.

(B.)—RESINS, VARNISHES.

Resinates, Metallic [and Coloured], Manufacture of [for Varnish Making]. P. Truchot. Rev. Chim. Ind. 1897, 8, 107.

The resinates of the alkali metals are soluble in water, but the others are insoluble. Although the corresponding compounds of the heavy metals are less soluble in such solvents as enter into the composition of varnishes than those of the alkaline earths, yet they have the advantage of possessing the same colour as their respective hydrates. For very cheap varnish, common dark rosin may be used, but for higher-class products only the palest varieties can be employed; and it is often advisable—especially when coloured resinates are required—to first filter and bleach the colophony.

To effect this latter object the material may be either (1) distilled with superheated steam; (2) treated with chlorine, sulphuric acid, water, and finally weak nitric acid; (3) warmed with common salt and chromic acid, or a mixture of a bichromate and sulphuric acid, and washed in weak ammonia; (4) dissolved in alkali, the solution treated with chlorine, and the resin precipitated with an acid or a metallic salt; (5) heated with zinc dust, with or without the addition of sodium bisulphite or oxide of manganese and a bichromate, followed by filtration through sand; or (6) heated with sulphuric acid under a pressure of about 65 lb. for an hour, then cooled and washed in water. One of the best, but rather expensive, methods is (7) to filter the rosin, and then heat it for one or two hours with 5 per cent. of zinc chloride, afterwards adding 12 per cent. of potassium bichromate, cooling to 100° C., and filtering once more.

The hydrates of certain metals, after being dried at low temperatures, are dissolved direct on agitating them with a solution of rosin in coal-tar naphtha; but in other cases the desired resinates are obtained from the sodium compound by double decomposition. In the preparation of the latter, care must be taken only to employ the theoretical amount of alkali, and also to see that it is sufficiently free from carbonate, sulphate, silicate, &c., to prevent any contamination of the desired resinate with inorganic salts of the metal, insoluble in the volatile varnish solvent. The resinate of copper must be precipitated and dried in the cold; but in many other instances slightly warm liquids may be used, and the temperature of the drying room may be raised somewhat, except as mentioned below. The two solutions are mixed together in molecular proportions, and it is occasionally convenient to employ, as the precipitant, salts of two different metals (*e.g.*, zinc and copper) in order to obtain double resinates at one operation. The precipitates are very bulky, and take a long time to settle; but they must be well washed in cold water, especially if they are liable to contain a deliquescent body, such as chloride of calcium or magnesium, since these would materially hinder the drying of the varnish.

The metallic resinates dissolve more readily in the light coal-tar hydrocarbons than in the corresponding petroleum products, although their solutions in the former may be diluted to the proper consistency with light petroleum spirit, as this helps them to dry more quickly. Solution is also assisted by previously dissolving in the naphtha a quantity of rosin equal in amount to that existing in the resinate—a plan which has the advantage of saturating any free hydrate or carbonate of the metallic base.

If the solution of sodium resinate is mixed with a suitable aniline dye before the addition of the other metal, on precipitation a coloured resinate is formed after the fashion of ordinary lakes. In such cases both precipitation and drying must be carried out in the cold; and it is very important to remove as much moisture as possible by means of the filter press before placing the substance in the drying chamber. The coloured resinates of magnesium dissolve very readily in methyl alcohol, while those of the other metals are preferably dissolved in naphtha as before. Generally speaking, they are soluble in spirit varnishes and in oils, &c. which contain free fatty acids.

Excepting the aluminium compound, they keep well in solution, but are decomposed above 100° C. As varnishes they yield a good film, but the latter is brittle and apt to blister unless special precautions are taken to remedy these defects.—F. H. L.

(C.)—INDIA-RUBBER, &c.

PATENTS.

Substitute for India-Rubber, Celluloid, Leather, Oil-Cloth, Linoleum, Mother-of-Pearl, Tortoise-Shell, Amber, Ivory, and the like; An Improved Plastic and Incombustible Material to be used as a, and capable of being moulded, drawn, or made into Threads, and in the Form of Plates, Tubes, Cylinders, or Soft and Silky Threads, Resembling Silk in Appearance. Eug. Cadoret, Paris. Eng. Pat. 12,451, June 6, 1896.

The base of the "textiloid" is an oxidation product of oils, termed by the patentee "résinoline." This is prepared by

saponifying one part of any suitable oil with three-fourths part of a metallic carbonate, and treating the soap with one part of nitric acid (36°). After a contact of 30 – 35 minutes the liquid is decanted and the fatty body saponified by one part of caustic alkali. The soap is treated with acid and the liberated resinoid substance purified by solution in ether or alcohol, and evaporation of the solvent. The "résinolines" thus prepared show a great analogy in physical properties to ordinary resins. When combined with oxides or earths, natural resins, cellulose, nitro-cellulose, organic matter such as gelatin, or a mixture of these, they form the "textiloid."

For example, 100 parts of "résinoline" are mixed with 20 to 30 parts of zinc oxide, magnesia, kaolin, or the like, 60 parts of methyl alcohol are added, and the whole left for three or four hours, after which the mass is divided and passed through a rolling machine with horizontal cylinders slightly warmed (30°—40°). After one or two hours' working, the paste, from which the alcohol has now evaporated off, is submitted to a pressure of from 200 to 300 atmospheres in a hydraulic press heated to 80°—90°. After five or six hours the necessary consistency is attained, and the mass can be cut up, moulded, drawn, or made into threads.

In the case of organic substances such as protein, gelatin, and albumin, a preliminary treatment with glycerin and nitric acid is required before they can be incorporated with the "résinoline." In addition to the uses enumerated in the title, it is claimed that the "textiloid" can be applied to the manufacture of incandescent gas mantles.—C. A. M.

Gutta-Percha and Caoutchouc-like Substances, Impts. in the Production of Artificial. F. Fenton, London. Eng. Pat. 26,548, Nov. 24, 1896.

Easily oxidisable vegetable oils, such as linseed, hempseed, nut oils, or cotton-seed, are mixed with tar or "other form of pyroligneous acid," macerated in a bath of dilute nitric acid, and, when formed into a semi-magma, roasted in hot pans or plates. The elastic mass produced, can be vulcanised and applied to the same uses as india-rubber.

For artificial gutta-percha, shellac is added in the proportion of from 1 to 20 per cent. before the nitric acid bath. If extra firmness be required, the magma is worked up with a portion of quick or caustic lime.

The proportion of tar or pitch required is from 5 to 75 per cent. of the oil taken, the lower percentages being used for the more rubber-like forms.—C. A. M.

Compositions for Repairing Leaks in Pneumatic Tyres, Impts. in. C. S. Howe and J. W. Langley, Cleveland, Ohio, U.S.A. Eng. Pat. 27,761, Dec. 5, 1896. (See also this Journal, 1896, 818.)

Sodium silicate solution is mixed with thrice its volume of a solution of mucilage, starch, or dextrin, with or without glycerin, and is neutralised by an acid to form a thick jelly of silicic hydrate, which is then mixed in a churn with three additional parts of the gum or starch solution. Or, an aluminous magma may be formed by mixing a solution of alum or aluminium sulphate, or of an alkaline aluminate, with the solution of gum or starch, followed by treatment with an alkali or an acid, as the case may be, and subsequent procedure as with the before-described silicious magma. From 4 to 6 oz. of the composition are introduced into the tyre, any small puncture in which, is automatically repaired by its action.—E. S.

XIV.—TANNING, LEATHER, GLUE, SIZE.

Quebracho Tanning, Difficulties in. W. Eitner. Der Gerber, 23, [542], 81—82.

Quebracho has now taken a permanent place in tanning, and some correspondence with a sole-leather tanner using it along with oak bark, has brought to light difficulties in its use, and enabled the Versuchsstation to point out the proper method of using it. In the case in point, the tanner had extracted the quebracho with hot liquors made from oak layers. He found the leather had not the nice white appearance of a pure oak tannage, and had a very thick grain. The extraction of quebracho with hot liquors made from old wood is quite wrong, as a great excess of colouring matter is thus dissolved out; this causes not only a bad colour, but a thick grain, owing to the layer underneath the grain absorbing the colouring matter, and causing the grain to stand out.

The proper way is to mix the quebracho with fresh oak bark and to use the combination as a layer material. Quebracho tannin extracts quite as easily as oak tannin, and, used in this way, gives a light colour. For the preparation of sour liquors, the oak and quebracho from the layers is mixed with pine bark and extracted cold.

At 88° C. all useful matter is extracted from quebracho; the warm liquor should not, however, be run direct to the pits, but first over a sour bark.—J. T. W.

Egg-Oil. M. Kitt. Chem. Zeit. 1897, 21, [31], 303.

See under XII., *page* 448.

Leather Dyeing with Artificial Dyestuffs. Leipziger Färber- u. Zeugdrucker Zeit. 46, [4], 162.

See under VI., *page* 439.

PATENTS.

Tanning Extracts and Dyestuffs, Impts. in and relating to the Manufacture of. H. H. Lake, London. From Lepetit, Dollfus, and Gansser, of Milan and Susa (Piedmont), Italy. Eng. Pat. 8582, April 23, 1896.

See under IV., *page* 436.

Horn, Impts. in the Manufacture of Artificial. P. H. Hansen, and J. K. Westengaard, Copenhagen, Denmark. Eng. Pat. 1550, Jan. 20, 1897.

About 100 parts of separated milk are taken, and one part soda and $\frac{1}{300}$ part pure hydrochloric acid added. After standing from one to three days, about three-fourths of the water will have separated, and the casein is removed and allowed to ferment for a few days longer. This is then pressed in linen bags and the solid portion dried at about 60° C. It is then ground, and 70 parts are mixed with about 28 parts of ground bone, kaolin, gypsum, or the like, about 2 parts of stearin or paraffin wax, and as much water as is necessary to form a stiff dough, the temperature being raised to 90° C.

The dough is placed in a hot mould and worked into sheets, a pressure of about 10 atmospheres being employed. The material is brushed over with burnt magnesite, placed in water for a few days, and dried.—A. L. S.

XV.—MANURES, Etc.

Basic Slag, Chemical Composition and Agricultural Value of. G. Paturel. Bull. Soc. Chim. 1897, 17, 319—321.

Working with basic slags of French origin containing 14—19 per cent. of phosphoric acid, the author confirms the view that they contain calcium tetraphosphate and silicate, with uncombined alumina, magnesia and oxides of iron and manganese. He finds that the solution of the free lime in ammonium chloride liquor (which is without effect on the phosphate and silicate) gives results in accordance with those calculated on the above supposition from the analysis of the whole sample. The solvent action of acid ammonium citrate solution on the phosphoric acid varies with the method of operation and with the proportion of free lime present. Since those samples which contain most free lime are the least soluble in the reagent, it would seem to indicate that the acid citrate was not suitable for determining the value of the slag as a fertiliser. The normal alkaline ammonium citrate made by Joulie's formula, contrary to the accepted belief, may be made to dissolve the whole of the phosphoric acid in the slag, if the latter be sufficiently finely ground and the agitation of the mixture be prolonged. Since no natural phosphate yields even a trace of its phosphoric acid to this solvent, it may be assumed that no tricalcium phosphate is present in the slag. At the same time, this reagent affords a ready means of determining the quantity of natural phosphate in an

adulterated sample. The slag exerts a profound influence upon nitrification in acid soils, and it is therefore desirable to work a considerable quantity of the slag into such soils, the proportion of possible nitrification being increased with each addition of slag. The action being due to the free lime present, it is evident that with equal phosphate contents, the slag containing most lime is the best; and the determination of the latter substance is of the highest importance from an agricultural standpoint.—W. G. M.

Phosphates in Bone Meal, Citrate Solubility of. A. Keller. Chem. Zeit. 1897, **21**, 323.

Several portions of bone meal, of best quality and fineness (each 10 grms.), were placed in beakers and covered with water (5—10 mm.), which was renewed from time to time as it evaporated. The beakers were exposed for four months, some (*a*) to direct sunlight, the rest (*b*) being in the shade.

The following analytical results show the percentage composition of the original bone meal, and of the meal after decomposition:—

—	Fresh Meal.	Decomposed Meal.	
		a.	*b.*
P_2O_5-total	23·62	..	..
" citrate-soluble	15·81	7·52	10·03
N-total	5·23	4·99	5·03
N as NH_3	0·03	1·74	1·22
Fat	2·06	..	..

Assuming dicalcium phosphate to be present in the meal, the results indicate that the diminished solubility is due to formation of dicalcium ammonium phosphate. This may account for the want of agreement between the results of manurial experiments with bone meal and the analytical results (citrate solubility of the phosphoric acid). These, and other experiments made by the author confirm the opinion that the degree to which bones are freed from fat is often of greater influence than the quality of the bones. The percentage of fat in bone meal should therefore be determined, as well as the citrate solubility.—N. H. J. M.

PATENT.

Artificial Manure or Fertiliser, A New or Improved, and Process for Manufacturing the same. O. G. Blunden, London. Eng. Pat. 10,109, May 12, 1896.

Carbonised peat, peat ash, and the liquid product of the destructive distillation of peat, are mixed with gypsum or its equivalent in suitable proportions to form a manure. —E. S.

XVI.—SUGAR, STARCH, GUM, Etc.

Sugar in Water, Law of Contraction on the Solution of. A. Wohl. Ber. 1897, **30**, 455—460.

From an investigation of the contraction which takes place when sugar is dissolved in water, the author has arrived at the following empirical laws:—

For a solution containing x parts of sugar and consequently $1-x$ parts of water, the contraction $d\,v$ referred to unit volume is given by the equation:—

$$d\,v = 0{\cdot}00303\,\frac{x\,(1-x)}{1-\sigma\,x}$$

where 0·00303 is a constant, representing the influence of sugar and water on each other, and $\sigma = 0{\cdot}35965 = 1 - \frac{1}{s}$, where s represents the density of sugar supposed to be in the liquid state (1·56165).

The specific volume of the sugar solution is—

$$v = (1-\sigma\,x) - \frac{0{\cdot}00303\,x\,(1-x)}{1-\sigma\,x}$$

and the reciprocal of this, *viz.*, the specific gravity of the solution, is—

$$s = \frac{1-\sigma\,x}{(1-\sigma x)^2 - 0{\cdot}00303\,x\,(1-x)}$$

The author claims that the specific gravity can thus be calculated correctly to the fourth or fifth decimal place. —J. S.

Sugar Refining, Methods of Defecation in Louisiana. F. C. Thiele. Chem. Zeit. **21**, [16], 136—137.

The author discusses the relative methods of cold and hot defecation of cane juice. On most of the plantations in Louisiana, the lime is added in the form of a cream to the cold juice. In some few instances the milk of lime is poured into the defecator, and the steam turned on immediately, the juice being allowed to run on to the hot pipes; but this method is not generally practised. Experience has shown that in order to obtain a white sugar, the lime should be only added in such quantities as just to avoid the evolution of ammonia; that is, the juice must be kept slightly on the acid side. As a general rule, about 0·1 to 0·15 per cent. of lime has been found to be sufficient. If the lime be added to the previously heated juice, it is difficult to hit the exact quantity, as the colour reactions of the hot juice are not the same as those observed in the cold. An excess of lime may thus be added unawares, in which case the resulting product would be of a dark colour.—L. S.

Cane-Sugar Juice, Carbonating of. Prinsen Geerligs. Chem. Zeit. 1897, **21**, 241—242, 262—263.

The author concludes from his experiments that double carbonating of cane-sugar juice is to be avoided if a product ready for direct consumption is required, because in the warming and neutralisation of the mud, some portions pass into the juice. This process also requires more lime and carbonic acid, and also time, than simple carbonating, for if the calcium glucate is to be retained in the defecation mud, cold filtration must be adopted. Thus, the juice, which filters with difficulty in the cold, must be mixed with a considerable amount of calcium carbonate in order to start the filtration of the mass. If simple carbonating be employed, the brown colour dissolves as saturation proceeds, and the juice can then be filtered hot.—J. L. B.

Diffusion Juice, Conditions of Inversion of the Sugar of. L. Jesser. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1897, **14**, 822—828. Die deutsch. Zuckerind. Jan. 2, 1897.

The question whether there are indeterminate losses from destruction of sugar during the diffusion process and the subsequent manipulation of the juice, has not been answered categorically, although this is one of the most burning questions of the sugar manufacture.

It is well known that raw beet juice is strongly acid, but of varying amount to different indicators. This fact, and the difference of alkalinities resulting from the use of litmus and of phenolphthalein in the clarification of the juice with lime water, make it doubtful that acid salts are the sole cause of the acidity; and it is thus impossible to conclude as to the invertive power by the acidity of the juice alone.

There are three reasons for losses of sugar during diffusion, which are the consequence of some modification of the juice: loss by fermentation, loss by direct overheating, and loss by inversion of sugar caused by the non-sugar.

In the actual conditions of work, fermentation may be dismissed as non-existent, the temperature being too high to allow of the existence or the vitality of micro-organisms. An infection of diffusion juice is only possible when the works is badly served with water, or when residual waters are returned without previous purification. These being at a temperature of 50° or 60° C., and meeting cold slices, may cause destruction of sugar.

Loss by overheating may be caused by want of cleanliness, or by using direct instead of exhaust steam. Under normal conditions the indeterminate loss in diffusion may amount to from 0·3 to 0·5 per cent. of sugar per 100 kilos. of beetroots. But it cannot be admitted that this loss is produced by

inversion. This results from the experiments of Claassen, who found the invert sugar in diffusion juice always less than that contained in the beetroot. These researches, however, are encumbered with the uncertainty of manipulations with Fehling solution.

It is of the first importance to know and prove if, in general, it be possible to attribute an invertive power to diffusion juice, and of what importance it may be from one year to another. Raw juice is known to contain a series of substances which, on boiling with bases, are converted into acids. If, on boiling the original juice, invert sugar be formed, the acid formed during subsequent treatment by alkali must be increased proportionally. This conclusion can only be drawn after having proved that the non-sugar has suffered no change on boiling. To decide this point, sugar was added to diffusion juice and the increase of acid noted in the two cases. If the increase of acid is proportional to the quantity of sugar added, it is possible to infer that the juice has an invertive action, and to measure it. The results of a number of experiments made by the author show that the indispensable conditions for inversion really exist in diffusion juice both during and after diffusion, and that it varies from one year to another. It may be said with certainty, however, that a loss greater than 0·15 per cent. is impossible under normal conditions, this figure being reached only once by boiling for an hour.—L. J. de W.

Beetroot and Diffusion Juice, Acidity of. L. Pannenko. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1897 **14**, 829—830.

Pannenko carried out during a whole campaign a series of comparative tests of beetroot juice (cossettes) and corresponding diffusion juice for acidity. The acidity was expressed in c.c. of normal caustic soda per 100 c.c. of juice, and calculated to 100° Brix.

The tables given show the diffusion juice higher by 0·02 to 0·60 than that from the cossettes. The acidity of the beetroot juice during the campaign varied greatly, ranging from 5·0 to 11·2 c.c. of normal caustic soda. Healthy normal beetroots invariably yielded juice the acidity of which did not much exceed 5·0 c.c. of normal soda, and these juices worked very well in the factory. Diffusion juices which showed an acidity of 11 c.c. of normal soda, passed the filter presses very badly: in proportion as the acidity lessened, the work made better progress.

The author found but slight differences between the acidity of diffusion juice as it left the battery and that of the same juice from the measuring tank. The latter was always slightly higher. According to the author, the principal cause of this increase is to be sought in the fact that the measuring tanks are not always emptied; there remains always a portion of an older juice (the acidity of which has increased by the reason that it is older), and this mixes with the juice newly arrived. When the tanks are completely emptied and cleaned, the differences in acidity become less marked. A table given, shows that, before cleaning, juices varying from 6 to 9 of acidity, increased by 0·53 to 0·91; after cleaning, the increase was only from 0·10 to 0·43.—L. J. de W.

Sugar Solutions, Crystallisation of. A. Herzfeld. Zeits. f. angew. Chem. 1897, 221—224.

The author reviews the theory of Wulff and the processes for crystallisation in motion, leading towards the ideal of the raw sugar manufacture—the production of a single product and molasses.

Manoury's process for returning the whole of the syrup to the first product must eventually so increase the quantity of mother-liquor that it can no longer be worked rationally. There is more prospect of success when two qualities of sugar are made, the first syrup being incompletely exhausted, the after-product boiled to grain and then stirred for a few days to produce a second product and molasses. The author has satisfied himself of the feasibility of this process, which has been worked during the last campaign in Russia and Austria.—L. J. de W.

Molasses [Sulphites], Process for the Purification of. K. F. Kastengren, Stockholm. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1897, **14**, 811. (From Magyar czukoripar.)

The object of the process is to facilitate the purification of molasses, and even to convert it into an edible product, whatever its degree of impurity may be. This result is obtained by boiling the molasses under pressure, after adding an alkaline sulphite, calcium sulphite, or even sulphurous acid in excess.

By boiling under pressure, reactions are induced in the molasses, resulting in purification and decoloration.

The excess of sulphurous anhydride may be added before boiling. If, after boiling, an excess of lime be still found, it is easily eliminated by saturating with carbonic acid.
—L. J. de W.

Molasses, Extracting Sugar from, by Baryta. H. R. Langen. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1897, **14**, 811—812. (From Centr. Zuckerind. d. Welt.)

Langen, of Euskirchen, has patented a process for the extraction of sugar from molasses by means of baryta. He employs, for the precipitation of the sugar, so-called barium hydroxysulphide, obtained as a mixture of barium hydroxide and hydrosulphide by the solution of barium sulphide. Dubrunfaut and Leplay showed that the reaction between barium sulphide and sugar in presence of water gave barium sucrate: for this reason, the patentee claims only the method of regenerating the barium sulphide.

The process is relatively simple if the carbonic acid at disposal be rich. The mother-liquor, filtered from the barium sucrate, contains the excess of barium and the whole of the sulphur. On treating it with carbonic acid, barium carbonate is obtained and sulphuretted hydrogen is liberated. This latter, on combustion, is converted into sulphuric acid.

The acids obtained serve to convert barium carbonate into sulphite or sulphate, and the latter, on reduction with carbon, gives rise to barium sulphide again.

Sulphuretted hydrogen, however, only enters into combination when it is relatively pure. To obtain it in this state, it is necessary to have rich carbonic acid, for gases mixed with ordinary carbonic acid likewise mix with the sulphuretted hydrogen and dilute it. Rich carbonic acid as an industrial product cannot be reckoned upon. The author has therefore sought to modify his process so as to be able to use carbonic acid of average strength.

The modification is as follows:—To the mother-liquor filtered from the sucrate, magnesium sulphite is added. Insoluble barium sulphite is formed and magnesium hydroxide, while sulphuretted hydrogen is disengaged, and is converted by combustion into sulphurous acid. The precipitate formed, filters badly, on account of the gelatinous nature of the magnesium hydrate. To render it filterable, it is converted into magnesium carbonate by a current of carbonic acid gas. The mud separated from the mother-liquor is treated with sulphurous acid, converting it into magnesium sulphite, which, being soluble, is easily separated from the barium sulphite. It can thus be returned to the cycle of operations.—L. J. de W.

Raffinose, Occurrence of, in American Sugar Beets. W. E. Stone and W. H. Baird. J. Amer. Chem Soc. 1897, **19**, 116—124.

Attention has been called to the fact that the amount of raffinose found in sugar beets is dependent on climate, soil, seed, conditions of growth, &c., and since the conditions under which beets are grown in America differ in many respects from those of Northern Europe, it might be expected that this minor constituent would be absent. The authors find, however, that raffinose occurs in the juice of American sugar beet in appreciable quantities. The circumstance that raffinose, like cane sugar, forms an insoluble compound both in hot and cold solutions with alkaline earths, accounts for the accumulation of the first-mentioned sugar in molasses and secondary products. Raffinose, to a large extent, remains uncrystallised in the presence of

considerable quantities of cane sugar, and this fact, in conjunction with its behaviour with alkaline earths, explains why nearly the whole of this sugar is found stored up at the close of the campaign in the molasses and second-grade syrups.

Unsuccessful attempts were made to detect raffinose in syrups by means of the mucic acid test, this acid being held in solution in the form of salts. Kodyl and Scheibler's methods were unsuccessfully employed for the isolation of raffinose from molasses. The authors have devised the following process, which is a combination of the preceding ones:—2,000 grms. of syrup were diluted with water and treated with a large excess of basic lead acetate. The solution was decanted from the precipitate, and, after further dilution, was treated with ammonia until strongly alkaline. After standing in a warm place a heavy precipitate settled, which was supposed to consist of 1 mol. of raffinose or cane sugar with 3 mols. of lead oxide. This was washed by decantation and treated with sodium carbonate and carbon dioxide until all the lead was removed. The filtrate obtained was concentrated to a thin syrup, and strontium oxide added in the proportion of 3 mols. to one of sugar. The mixture was maintained at the temperature of boiling water for three hours, during which time a granular precipitate was thrown down, consisting of strontium saccharate and raffinosate; the strontium compounds thus obtained were decomposed by carbon dioxide, the solution filtered and evaporated to a thick syrup. This, on standing, became solid, and after careful fractionation with methyl alcohol and water, a product was obtained having an $[\alpha]_D$ 103·7°.

It is to be noted that the influence of raffinose is manifested, not in the appearance of isolated raffinose crystals, but in a modified and abnormal appearance of all the crystals, which are similar to each other, but entirely distinct from those of cane sugar. This tendency of raffinose to modify so distinctly the crystals of cane sugar would seem, in the opinion of the authors, to afford a means of readily identifying the presence of small amounts of raffinose in cane sugar.—J. L. B.

Beetroot Sugar Analysis. Resolutions of the Austro-Hungarian Chemists engaged in the Sugar Industry at the Meeting at Buda-Pesth, June 22, 1896. Zeits. angew. Chem. 1897, 123.

See under XXIII, *page* 472.

Sugar Solutions, Nitrate of Lead: The Use of, for Decolorising Various. H. Pellet. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1897, **14**, 794. (This Journal, 1897, 67.)

See under XXIII., *page* 472.

New German Sugar Tax, Effects of the. Export, **19**, [14]. 185.

See under Trade Rep., *page* 477.

PATENT.

Adhesive Compounds, Impts. in. A. J. Boult, London. From C. M. Higgins, New York, U.S.A. Eng. Pat. 2746, Feb. 2, 1897.

This is a slight modification of the inventor's previous patents, 22,682 and 22,685 of 1891 (this Journal, 1892, 417), in that the borax is added to the cold solution of dextrin instead of to the hot solution.—A. L. S.

XVII.—BREWING, WINES, SPIRITS, Etc.

Dextrose (d-Glucose). A. L. Stern. J. Fed. Inst. Brewing, 1897, **3**, 179—194.

The following are given as typical analyses of commercial glucose:—

—	1.	2.	3.	4.	5.
Water	10·5	9·9	15·7	17·8	16·0
Dextrose	80·0	70·0	67·4	64·9	65·3
Maltose	..	5·1	11·0	12·4	2·1
Dextrin	..	..	..	4·3	1·2
Unfermentable bodies	8·2	14·8	4·3	not determined.	14·3
Ash	1·3	0·2	1·6	0·6	1·1
	100·0	100·0	100·0	100·0	100·0

No. 1 is a very good sample, and much above the average. No. 2 is met with in large quantities at the present time, and is an excellently manufactured product; the small ash probably indicates that oxalic acid was the converting agent. No. 3 is not properly converted, and this is indicated by the amount of maltose, the low percentage of unfermentable matter, and the high percentage of water; the ash is also excessive. No. 4 shows a bad analysis, the neglect to determine the unfermentable matter making the figures valueless. No. 5 is a fairly good product, but the water and the ash are too high. In the valuation of a sample of commercial glucose, the following points have to be considered:—The colour of the solution should not be excessive; the solution should be fairly bright; the ash should not much exceed 1 per cent.; the extract should not be much under 77 lb. per 2 cwt. per barrel, and no samples should give less than 73 lb. per 2 cwt. per barrel; the fermentable matter should not be less than 70 per cent., or less than 80 per cent. if calculated on the dry matter, and there is no reason why this should not partly consist of maltose.

—A. K. M.

Dried Yeasts, Observations on the Vitality of. H. Will. Zeits. f. d. ges. Brauw. **20**, [6], 91—92. (See also this Journal, 1897, 155.)

The various conserves were re-examined, at the expiration of $10\frac{1}{4}$ years, for the presence of living yeast cells, those in tins being in the best condition, without any apparent smell or evolution of gas being noticed on opening. In view of the presumably small proportion of living cells still existing, larger samples (5 to 6 grms.) than before were taken for examination and sown in sterile hopped brewery wort. The bacteria present retarded the reproduction of the weakened yeast until themselves repressed by acidifying the medium, but when the cultures are left alone the bacteria entirely suppress the yeasts, and infest the dead cells of the latter. In view of the weakening of the dried yeast cells, it was deemed inadvisable to acidify the first culture, but on re-inoculation in fresh wort a little tartaric acid was found advantageous.

The results of the cultures at 25° C. demonstrated that in the asbestos conserve No. 7, wild yeasts alone were present; asbestos No. 8 containing chiefly wild, with a few culture yeasts; charcoal No. 9, mostly culture yeasts; charcoal No. 10, a predomination of wild yeasts, and wood pulp No. 13 C^1 (stored in ice) the same. *S. apiculatus* could not be detected in any. The wood-pulp conserve No. 13 C^2 (kept in a stoppered bottle in the window) and paper pulp No. 2 (top yeast) merely contained dead cells.

Bacteria (cocci and diplococci) were present in nearly all the cultures, and acetic ferments in 13 C^1 and 10. Nos. 8 and 10 showed brisk fermentation after 48 hours, but no living yeast cells could be identified with certainty. In Nos. 13, 9, and 7 signs of fermentation appeared only on the third, fourth, and fifth day, and new cells were discernible. The irregular development of the yeasts in parallel cultures showed that the living cells are very unevenly distributed in the conserves.

Compared with the results of the ninth year's examination, those now obtained were concordant, except with charcoal 9 and wood pulp 13 C^2, in both of which the proportion of living culture yeasts was relatively greater than in the others.

A fresh proof of the unfavourable storage conditions (higher temperature, influence of light) of the wood pulp

13 C^2 as compared with C^1 was afforded by the presence of a comparatively large number of living yeast cells in the latter and their total absence in the other.—C. S.

Yeast, Chinese (Amylomyces Rouxii). Utilisation [Spirit Manufacture] of, in European Fermentation Industries. A. Boidin and E. Rolants. La Bière, **5**, [3], 33—36.

This yeast has already been described by Calmette (Ann. Inst. Pasteur, 1892, 604). The present authors find that the organism produces less alcohol, by about 12 per cent., than is obtained from rice by the ordinary method practised in France, the shortage being due to consumption of hydrocarbons during the aerobic existence of the ferment.

Working with yeast manufacturers' wort, they find that the action of the organism is even more restricted than in the former case, ceasing when the alcohol amounts to 4 per cent. of the liquid. A study of the effect of acidification on the saccharification of rice by *Amylomyces R.* led to the conclusion that the formation of acid during the process is in inverse ratio to the amount of sulphuric acid added; that the diastase is paralysed by an excess of acid and also by a neutral condition of the paste, its activity being greatest in a faintly acid medium; that in presence of an excess of calcium carbonate the sugar and alcohol formed are rapidly eliminated; and that the sugar formed is glucose and not maltose.

When applied to the residual liquor from the distillation of the fermented wort in yeast factories—which liquor contains about 10 grms. of dextrin, 7·2 grms. of albuminoids, nearly 4 grms. of sugar, and 13 grms. of non-nitrogenous bodies per litre—and left to ferment, the *Amylomyces* produces acid in inverse proportion to the original acidity of the medium, and when the latter amounted to over 3·40 grms. (expressed as sulphuric acid) per litre, a portion is eliminated. More acid is formed by surface culture than by bottom culture, the crop of *Amylomyces* increases with the aëration, and the amount of hydrocarbons eliminated, when the medium is neutral and aërated by blowing. By prolonging the time allowed for the operation to 7 days, between 0·9 and 1 per cent. of alcohol was obtained—a result also attainable in the shorter time by fermenting at 34°—35° instead of 26°—30° C.

Further investigations pursued on a practical scale at Collette's yeast factory at Seclin, resulted in the production of some 270 litres of absolute alcohol from 300 hectolitres of the residual still-liquor and grains, the value of the product being 93 francs, at a cost, for fuel and rectification, of 22 francs, so that the nett profit from 300 hectolitres of otherwise waste liquor is 71 francs, or, in an establishment producing 750 hectolitres of liquor per diem, a daily gain of 177·50 francs (*7l. 2s.*), which figures will give some idea of the value of *Amylomyces Rouxii* for the treatment of distillers' grains.—C. S.

Wine Yeasts. Kayser and Barba. Bulletin Ministère Agric. 1896; Zeits. f. d. ges. Brauw. **20**, [7], 108.

Pure cultures were made of yeasts isolated from the sediment of wines from the department of Gard (France) and compared with a number of other wine yeasts. Acidity and temperature of fermentation being the two chief points of interest, were investigated, the cultures being sown in malt germ water containing 188·76 grms. of sugar, 6·02 grms. of tartaric acid, and 223·70 grms. of total extract per litre, and allowed to ferment at 25° and 35° C. On comparing the results it appears that with the higher temperature and a medium rich in sugar and acid, the yeasts work themselves out before all the sugar is eliminated, the strongest yeast degrading only 140 out of 180 grms. of sugar per litre, and the indigenous yeasts leaving less behind than those from other districts; at the lower temperature the results were more nearly approximate. The amount of acid produced was irregular, but, as a rule, greater with the higher fermentation temperature than at 25° C., and the indigenous yeasts generally gave rise to less acidity than the others.

Check experiments were made to determine the influence of the percentage of sugar and acid. In the one series, with a medium containing 176·87 grms. of sugar per litre, the results obtained indicate that at 25° C. the influence of the acid is negligeable, but with higher temperatures, an increase of acid helps to restrict the elimination of the sugar, the southern yeasts being throughout more energetic than the more susceptible varieties. The latter are retarded by temperature alone, both as regards the production of alcohol, fixed acids, and yeast crop, but the formation of the volatile acids and fermentative energy are generally increased; in presence of greater acidity the results, however, vary according to the individual character of the yeast.

In the second check experiment, with a medium containing only 86·206 grms. of sugar and 2·22 grms. of tartaric acid per litre, similar results were obtained, both sets confirming the influence of the higher temperature on the elimination of sugar, especially in a rich and strongly acidified medium, and also that an increase of sugar, particularly in conjunction with a high temperature, retards elimination. On the other hand, the influence of acidity, apart from temperature, is much greater on the indigenous yeasts than on some of extraneous origin, which are more susceptible to temperature.—C. S.

Beer, A hitherto undescribed Turbidity in. H. Will. Zeits. f. d. ges. Brauw. **20**, [5], 77.

This turbidity is caused by the presence of a number of minute pale drops, resembling, to some extent, the so-called glutin-bodies, except that they are apparently liquid, and exhibit no dark outline. Even when present in considerable numbers, they do not seem to produce more than a "haze," and the quantity is never large enough to enable their chemical nature to be ascertained. They stain slightly with aniline colours and with iodine and potassium iodide, but do not seem to store up the colouring matter, like albuminoid bodies do even when the granules are small; so that it can hardly be assumed that they consist of albuminoid matter; neither, in the author's opinion, can they be ascribed to precipitated resins or hop-constituents.

Similar drops were found by the author in the nutrient gelatin prepared from the fermented worts of old mycelium cultures of low-fermentation yeasts.—C. S.

Beer, Yellow Foam and Ferruginous Flavour in. J. Brand. Zeits. f. d. ges. Brauw. **20** 141.

Instances frequently occur wherein beer, initially good, acquires, after standing several weeks in the transport casks, an inky flavour, and exhibits a persistent yellow foam, which, on examination, is found to consist of bubbles enveloped in a strong albuminoid integument. In a sample of this kind examined by the author, the inky flavour disappeared after several days, but was succeeded by another, reminiscent of bad pitch. Iron being found present, the pitchy flavour suggested that its source was the ochre used for colouring brewers' pitch; however, several samples of these substances were tested by immersion in beer for some days, but in no case could any trace of dissolved iron be detected, either by taste or reagents. (It is finally considered inadvisable to use ochre for colouring brewers' pitch.)

On the other hand, a piece of metallic iron immersed in 200 c.c. of beer, produced, in two hours' time, the same yellow froth and inky flavour as in the former case, 0·15 grm. of iron having been dissolved (chiefly as ferrous oxide). On removing the metal, and leaving the beer for a few days, the dissolved iron was found to have become oxidised to the ferric condition, the flavour diminishing as in the original sample. By this and other experiments, the cause of the phenomena was traced to the iron bung-hole rings fitted to the transport casks, the iron of which is dissolved by the acids in the beer. As wort is also capable of the same solvent action, it is therefore advisable to coat all iron utensils or appliances, exposed to beer or wort, with varnish or other protective covering.—C. S.

Beer, Influence of Dissolved Iron on the Colour of the Froth of, and on the Taste of. Wochenschr. f. Brau. 1897, **14**, 118.

It has been recently shown by Brand that beer has a very decided solvent action on metallic iron, which imparts to it a strong inky flavour, whilst the froth becomes of a yellow colour (see preceding abstract). The iron was found to be

present partly in the ferrous and partly in the ferric condition. Beer wort is much slower in its action on iron, and this is attributed to the absence of carbonic acid. The fact that so few complaints are heard of iron contamination, in spite of the extensive employment of iron bushes, may perhaps be due to a protecting coating of oxide. This suggestion, which is brought forward by Brand, does not accord, however, with the experience of Widemaier, who finds that the formation of rust assists the solution of iron by beer. The coating of oxide mentioned by Brand is, however, probably not ordinary rust, but rather a stable oxide artificially produced.—A. K. M.

Beer, On the Carbohydrates in. P. Petit. Comptes Rend. 1897, **124**, 510—511.

By keeping beer for three months at a low temperature, it contains about one-half of the extracts in the state of dextrin. From the behaviour of a certain quantity of dextrin isolated from 30 litres of beer, the author concludes that it differs from the dextrin of commerce. On hydrolysing the crude dextrin from beer, glucose is obtained, but at the same time other sugars are formed having a lower reducing power and a smaller or negative rotation. The presence of other sugars is also indicated after inversion, by the formation of two osazones, one separating in the heat, the other in the cold. Whilst commercial dextrin was inverted by dilute hydrochloric acid in an hour and a quarter, the dextrin from beer was not completely transformed even after three hours.—J. S.

Boiling with Hops, Shortening the Time of, and its Effect on the resulting Beer. W. Windisch. Wochenschr. f. Brau. 1997, 101—102.

Wort is boiled with hops for the following reasons:—

1. To destroy the diastase. This is effected before the boiling point is reached.

2. To sterilise the wort. This is effected after about 15 minutes' boiling.

3. To dissolve the soluble constituents of the hop. Hop tannin cannot be completely dissolved even by long boiling, and so the author recommends that the tannin be extracted separately, and added to the wort. Hops contain one hard resin and two soft resins; only the two latter are of value to the brewer, and they are very soon dissolved by the hot wort. If the hops have been previously soaked in hot water, this takes place in about 15 minutes. Since long boiling converts these soft resins into hard resins, it is be avoided. Hops contain a certain amount of albuminoids, some of which are valuable for producing a "head" on beer; these are completely dissolved after about 10 minutes' boiling. As hop oil is volatile, the longer the worts are boiled, the less of this will they contain.

4. In order to precipitate albuminoids from the wort. About two-thirds of the wort albuminoids are thus separated. This takes place quickly, and if the boiling be prolonged, a portion redissolves.

5. The concentration of the wort is, of course, proportional to the time of boiling. However, if care be exercised in mashing and sparging, it is possible to dispense with a large amount of evaporation. The author is thus led to conclude that the boiling is frequently prolonged much longer than advisable, and considering that wort is very easily coloured by heating, and, being an acid liquid, must be decomposed by prolonged boiling, he is led to recommend a considerable shortening of the time of boiling. He considers that from 1 to $1\frac{1}{2}$ hours are ample in all cases, but recommends that the wort be first boiled without hops, and that these, having been first soaked in warm water for two hours, be only boiled with the wort for 15—20 minutes.

—A. L. S.

Worts, Boiling of. C. H. Field. J. Fed. Inst. Brewing, 1897, **3**, 150—155.

The main objects aimed at in boiling worts are stated to be: (1) the extraction of certain constituents of the hops; (2) the precipitation by heat, and by the combined action of heat and hop tannin, of a portion of the nitrogenous matters; and (3) the fixation of the carbohydrate constituents of the wort, and the determination of the flavour or character of the beer. When an open copper was employed, the wort was found to acquire a distinctly harsh flavour, which is attributed to the oxidation of soft into hard resins; but this result is not obtained when a domed copper is used, and the latter is therefore preferable where delicately flavoured beers are required. With regard to the fixation of the dextrinous constituents, the higher the temperature at which the wort is boiled, the greater will be the stability of the product; and the conclusions arrived at are that, in boiling by steam, more actual units of heat are taken up, but the wort is in contact with lower temperatures, than when boiling by fire is adopted. In the author's opinion, however, equally good results are obtainable by either method of boiling, but where steam is used, a steady pressure of 50 lb. should be maintained throughout the boiling period. Where boiling under pressure is adopted, a much higher temperature can, of course, be obtained, but the circulation of the wort is probably much less rapid, and this is likely to lead to a greater increase in colour.—A. K. M.

Alcoholic Fermentation, A Revolution in the Knowledge of. A. Fernbach. La Bière, **5**, [3], 36.

Referring to the recently published researches of E. Buchner (this Journal, 1897, 156—157), the author remarks that we have now another instance of purely chemical action hitherto considered as inseparable from the physiological functions of living organisms. Zymase he considers as belonging to the diastase group, the *rôle* of which is extended by this discovery, and appears to be concerned in all the transformations erstwhile regarded as resulting from vital action. It seems as yet uncertain whether the discovery can have any practical value.—C. S.

Wine, On the Oxidation and Souring of. V. Martinand. Comptes Rend. 1897, **124**, 512—513.

The author shows that the oxidation of wine can take place under certain conditions of acidity without the intervention of oxidising diastase. If, before oxidation takes place, the wine be extracted with ether, a substance having the properties of tannin is removed, and such wine, which is neutral to Helianthine, suffers no further change on oxidation.

—J. S.

Wine, Composition of the, produced from Chinese and Japanese Species of the Grape Vine acclimatised in France. L. Lindet. Comptes Rend. 1897, **124**, 569—572.

Since 1882, certain varieties of the grape vine (habitat, the cool and moist hilly districts of China and Japan) have been successfully acclimatised at Damigny, near Alençon, and at present are being introduced, by way of experiment, in other wine-growing districts. These vines are of a hardy and vigorous type, and apparently resist well the attacks of maladies to which the vine is subject.

The most important feature, however, is the early period of ripening of some of the varieties. For example "Precoce Caplat," a Japanese species, yields ripe fruit as early as Sept. 15—20; and several other kinds (V. Romaneti, V. Pagnucci) ripen about the same time. The fruit of most of the varieties is throughout (pulp included) of a dark red colour, and yields a very deep, full-coloured wine, the intensity of the coloration being 4—5 times as great as that of an average claret.

In the subjoined table, the chemical composition of some samples is given.

It will be seen that these wines, so far as alcoholic strength is concerned, are on a par with a light claret; but, on the other hand, contain about twice as much extract, tartar, tannin, &c.

According to expert opinions, these wines are, in some respects, superior to the deep-coloured wines of similar character, produced in Spain and Central France, and are especially suitable for replacing the latter for blending purposes.

—		"Precoce Caplat." Analysed in 1895.	The Same Analysed in 1896.	*Romaneti cardifolia.*	*Romaneti trilobia.*	*Pagnucci pentalobia.*	*Spinovitis Davidi.*
Alcohol	Per Cent.	7·0	7·5	6·0	6·9	6·5	5·4
Extract at 100° C.	Grms. per Litre.	42·5	39·1	40·9	45·9	43·0	45·7
Acidity, as H_2SO_4	"	13·7	12·6	11·0	13·0	10·8	13·2
Tartar	"	4·3	3·4	5·5	6·4	5·3	5·2
Tannin	"	1·0	4·2	3·7	5·3	4·4	3·9
Ash	"	2·7	2·1	4·1	4·6	4·2	4·0

—H. T. P.

Yeast, Quantitative Determination of, in Fermentation Experiments. R. Kusserow. Wochenschr. für Brauerei. 1897, **14**, 117.

See under XXIII., *page* 473.

Alcohol and Water, Distillation of very Dilute Mixtures of. Application to the Determination of Alcoholic Solutions containing from 1 *in* 3,000 *to* 1 *in* 10,000. M. Nicloux and L. Bauduer. Bull. Soc. Chim. 1897, **17**, 424.

See under XXIII., *page* 473.

Wine, Detection and Quantitative Determination of Cane-Sugar in. P. Kulisch. Zeits. angew. Chem. 1897, 205.

See under XXIII., *page* 473.

PATENTS.

Grains, An Improved Method of Treating Brewers' and Distillers'. F. B. Pike, Streatham Hill, Surrey. Eng. Pat. 6147, March 19, 1896.

The grains are mixed with any meal, grain, or seed in sufficient proportion to absorb the moisture of the grains and render the mixture suitable for being readily worked into the condition of meal or prepared for baking and the like.—A. L. S.

Beer specially adapted for Invalids. J. C. Harvey, Knottingley, Yorkshire. Eng. Pat. 9783, May 8, 1896.

The wort is fermented as far as possible so that no sugar remains in it, and saccharin is added to produce the necessary sweetness; also alcohol if necessary.—A. L. S.

Brewing or Priming Ales or Beers, Manufacture of Material for Use in. B. W. Valentin, Llanelly, Carmarthen. Eng. Pat. 13,262, June 16, 1896.

A starch transformation product is prepared containing large quantities of maltose and dextrin, and the syrup obtained by concentration *in vacuo* is heated until it becomes to some extent caramelised. The product thus obtained is slowly fermentable under the influence of the cask yeasts and gives continuous life and fulness to the beer.—A. L. S.

Alcoholic Liquids, Impts. in, and Apparatus for Treating or Purifying, Rectifying, Ageing, Deodorising, and Decolorising. J. Y. Johnson, London. From The Electric Rectifying and Refining Company, New Jersey, U.S.A. Eng. Pat. 29,965, Dec. 29, 1896.

The apparatus consists of glazed metal cylinders holding about 100 galls. of spirit, an ozone generator with suitable oxygen containers, and an air-pump. The whole is connected together by suitable tubing, with valves, &c. The spirit vessels are mounted on revolving platforms or on trucks and furnished with attemperating coils.

The spirit to be treated is filled into the cylinders, the air exhausted, and the spirit brought to a cool temperature; ozone is then led into the vessel, and this acts on the spirit and produces the required purification.—A. L. S.

XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

(*A.*)—FOODS.

Cooked Fish, The Composition of. K. I. Williams. Proc. Chem. Soc. 1897, [178], 88.

Twenty-two species of fresh fish and five species of preserved fish and oysters were examined after cooking.

Determinations were made of the following constituents:—Water; carbon and hydrogen; nitrogen (total) by Ruffle's method; nitrogen by soda-lime combustion; ash; sulphur; phosphorus; fat; proteids; carbohydrates convertible into glucose; nitrates extracted by dilute alcohol; heats of combustion. The results are recorded in a series of tables.

Grapes, Solubility of the Red Colouring Matter of. The Sterilisation of Fruit Juices. A. Rosenstiehl. Comptes. Rend. 1897, **124**, 566—569.

The conclusions are:—

1. In opposition to the accepted belief, it is found that the red colouring matter of grapes and other fruit is soluble in the juice *before* fermentation.
2. By the action of air the colouring matter is rendered insoluble.
3. This is one of the causes of the disease known as "goût de cuit."
4. It is possible so to preserve (sterilise) fruit juices, that the colour, flavour, and aroma of the fresh fruit are perfectly retained.—H. T. P.

Meat Extract. Its Meaning as Food or Relish. C. v. Voit. Münchener Med. Wochenschr., **44**, 219—222.

The author states that, in his opinion, meat extracts even when they do contain some added albuminoids are practically useless as food. But what renders them valuable in admixture with other foods is the large quantity of stimulating constituents they contain.—L. de K.

Margarin, The Question of Colouring. R. Henriques. Chem. Rev. Fett-u.-Harz. Ind., **4**, [5], 68—70.

It is pointed out that some ready means of distinction of butter from margarin—such as artificial coloration—is required for the protection of the consumer. Soxhlet's proposal to add phenolphthalein to margarin to act as a "latent" colour until developed by alkali is inefficaceous owing to the ready solubility of the alkali compounds of this indicator in water. On the other hand dimethylamidoazobenzene (the alternative "latent" colouring medium proposed), whilst not open to this objection, is already largely employed, under the name of "Butter Yellow" for heightening the tint of pale pure butters, so that its use for margarin would not facilitate the purpose in view. Moreover, being developed, not only by mineral acids, but also by the lower members of the fatty acid series, it is possible that the rose-red colour would appear concurrently with the formation of butyric acid as the butter or margarin aged, notwithstanding that von Buchka has not found this to occur in old butters coloured with this substance.—C. S.

Margarin, Addition of Dimethylamidoazobenzene to. A. Partheil. Chem. Zeit. 1897, **21**, [27], 255—256.

In place of phenolphthalein (Soxhlet), the author advocates the use of dimethylamidoazo-benzene for the production of the "latent colour," in the proportion of 1 grm. to 100 kilos. The required quantity is dissolved in oil and added to the margarin at the same time as the colouring matter, and before the butter flavour is imparted. Margarin thus prepared shows a rose colour on the addition of hydrochloric, sulphuric, or other mineral acids, or with potassium hydrogen sulphate when a few drops of alcohol have been added. On the other hand it gives no reaction with organic acids, even with acetic or oxalic acids. Thus the spontaneous appearance of a red colour points to the presence of a mineral acid.

According to Prof. Munk, the substance is completely harmless, and it has a further advantage over phenolphthalein in that it is only soluble in traces in water, whether made alkaline or acid; and thus cannot be washed out from the margarin, whilst on the other hand it is very much more soluble than phenolphthalein in oils or melted fats.—C. A. M.

Coffee Beans, Determination of the Weight of the Layer of Caramel on, which have been Roasted with Sugar and upon the Examination of Coffee in General. W. Fresenius and L. Grünhut. Zeits. anal. Chem. **36**, 225.

See under XXIII., *page* 473.

Sugar in Chocolate and Foods, Determination of. H. Pellet, Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1897, **14**, 790.

See under XXIII., *page* 474.

PATENTS.

Butter and Cheese, Impts. relating to the Manufacture of. Rudolf Baekhaus, Hesse, and Philipp Schach, Hesse. Eng. Pat. 1253, Jan. 17, 1896.

"A process for producing butter or cheese, the characteristic feature consisting in the preservation of the butterine," preferably by the addition of 10 per cent. of salt, after freeing it from butter milk as far as possible. When required for use the butter is melted down and made into an emulsion with milk poor in cream. From this emulsion butter may be churned or cheese separated in the usual way.—W. P. S.

Grains, An Improved Method of Treating Brewers' and Distillers'. F. B. Pike, Streatham Hill, Surrey. Eng. Pat. 6147, March 19, 1896.

See under XVII., *page* 456.

Milk, Cream, and other Liquids, Impts. in or relating to the Preservation of. H. Higgins, Cambridge. Eng. Pat. 7011, March 31, 1896.

The liquid to be sterilised is placed in an air-tight vessel, on the top of which is an opening for the introduction of the liquid; this opening is closed by a suitable stopper. A second orifice is made in the top, to which is attached a tap or vent plug, protected by a filter of cotton wool and connected with a rod, which passes through a stuffing-box for opening and closing the tap. This tap is open during the sterilising process and is closed as soon as the process is completed. The receptacle is also provided with a draw-off cock, surrounded by a vessel containing an antiseptic fluid. —W. P. S.

Foods and other Perishable Goods, Impts. in or relating to the Preservation of. H. Higgins, Cambridge. Eng. Pat. 7042, March 31, 1897.

The improvement claimed consists of a sterilising chamber fitted with shelves for holding the vessels containing the article to be sterilised. There are doors at each end, and a vent, surrounded by cotton wool in a suitable manner.

The containing vessels are closed up, excepting a small hole, and are run into the sterilising chamber on trolleys. They are finally sealed up with a mixture of paraffin wax and one or more of the following: French chalk, zinc oxide, kaolin, plaster of Paris, or chalk.—W. P. S.

Coffee Substitutes from Grain, Impts. in the Process for Producing. Kathreiner's Malzkaffee Fabriken mit beschränkter Haftung, Bavaria (through R. Haddan, London). Eng. Pat. 7880, April 14, 1896.

An electrical current adapted to the kind and quantity of the grain used, is caused to act on the substances lying between the shell and the starch body of the grain during the preliminary soaking before roasting. By this means, the unpleasant taste caused by the decomposition of the nitrogenous matters during roasting is much lessened.

The soaking vessel is provided with a suitable mixing contrivance, and the wall of the vessel itself or portions of it, serve as a pole.—W. P. S.

Bread, Biscuits, and the like, and a Compound therefor; Impts. in the Manufacture of. W. W. Macadam, Brockley. Eng. Pat. 10,131, May 12, 1896.

This invention relates to a "diastasically prepared" extract of barley, wheat, maize, rice, oat, or rye, malt mixed with gluten (containing not more than 10 per cent. of starch), the said ingredients being thoroughly incorporated at a temperature of from 50° to 168° F. The above compound is used for mixing with flour for bread or biscuit making. From 4 oz. to 32 oz. diluted with water, may be added to each 280 lb. of flour. It is claimed that the yield of bread, &c. from a given quantity of flour is thus increased.—W. P. S.

Butter, Margarine, and the like, and in the Method of Employing such Apparatus; Impts. in and relating to Apparatus for Testing. R. Wells, Scarborough. Eng. Pat. 10,627, May 18, 1896.

See under XXIII., *page* 467.

Tea, Impts. in the Manufacture of, Products from. J. Davidson, Belfast. Eng. Pat. 11,060, May 21, 1896.

The concentrated infusion of tea is, after the addition of preservatives, further evaporated until the residue becomes pasty or even solid, and may then be kept in bottles or jars.—W. P. S.

Artificial Food for Swine, Cattle, and the like, An Improved Manufacture of. S. P. Sörensen, Vejle, and A. P. Heyman, Copenhagen. Eng. Pat. 27,114, Nov. 28, 1896.

The artificial food consists of a mixture of specially treated blood, mixed with molasses and corn. The blood is diluted with water, filtered, and heated to about 75° C., a dilute solution of alkali being added to keep the mixture fluid. Very dilute acetic acid is now poured in and the temperature increased to 100° C. Coagulation takes place when the comparatively solid mass may be removed from the liquid portion, pressed and dried. After grinding it is ready to be mixed with other constituents of the food.—W. P. S.

(*B.*)—SANITATION; WATER PURIFICATION.

Water, The Bacterial Purification of. Percy Frankland. Proc. Inst. Civil Eng., Nov. 1896, 3—31.

In April 1886, the author laid before the Institution the results of investigations on the bacterial purification of water which he had been carrying out by means of the then new bacteriological methods. In the present paper he gives a *resumé* of the progress of our knowledge during the past decade.

1. *Purification by Natural Processes.*—Numerous investigations have shown that the number of bacteria in a running stream becomes diminished during its natural flow. In seeking for an explanation of this, the remarkably destructive effect of *sunlight* on bacteria has been supposed to be a potent factor; but experiments by Procaccini have shown that the action of sunlight on microbes suspended in water

is confined to the upper layers, and that even Italian sunshine acting continuously for three hours in the height of summer failed to destroy a very large proportion of the microbes in water at a depth of only 50 centimetres.

Buchner, in experiments made on the Starnberger lake, near Munich, was unable to detect any bacterial effect at a depth of 5 feet, after $4\frac{1}{2}$ hours continuous sunshine. Hence, insolation cannot be regarded as a material factor in the bacterial purification of water, especially in such an uncertain climate as that of England. Incidentally it is pointed out that the practice of storing filtered water in underground reservoirs is sound, as the influence of light, whilst having but a limited bacterial effect, would encourage the growth of green plants and lead to contamination of the water by decaying vegetable matter.

The purifying effect of *sedimentation*, first established by the author's experiments, has now been shown to be of great importance in connection with waterworks engineering. Thus, Thames water which, close to the West Middlesex Co.'s intake at Hampton, was found to contain 1,437 bacteria per c.c., after passing through one storage reservoir contained only 318, and after passing through a second reservoir only 177 bacteria per c.c. Researches carried out on some Continental rivers have shown that where the rate of flow is such as to permit sedimentation, as in flowing through lakes, the number of bacteria in the water falls, but when the flow is rapid, no falling off occurs, and disturbance of the mud at the bottom will often increase the number of bacteria.

Pettenkofer contends that the bacteria of river water are not removed by sedimentation, but rather by *oxidation* and the action of *algæ*. The available evidence favours the view that algæ do promote a purifying effect, and that the simultaneous existence of algæ and large numbers of bacteria are incompatible; but there is room for much more research in this connection.

Dilution with the purer water of springs and tributary streams is one cause of the diminution of bacteria in river water. The influence of summer *temperatures* in increasing the bacterial life of streams seems to be more than counterbalanced by other seasonal effects. Thus, the author has shown that the rivers Thames and Lea are purer in summer than in winter; doubtless due chiefly to the fact that during summer these rivers are largely fed by spring water, whilst in winter they are largely composed of water which has washed the surface of cultivated land.

Great diversity of opinion exists as to the influence of the *movement* of running water; the experiments hitherto published show that the growth of the ordinary water bacteria is favoured by agitation, but typhoid bacilli in sterilised water were destroyed. Probably further investigation would show that agitation is favourable to some and not to other kinds of bacteria.

Our present knowledge leads to the conclusion that *filtration through porous strata* is the most efficient of all natural processes of water purification, in illustration of which the author points to the fact that in the natural chalk water of the Kent Company he has frequently found less than 10 microbes in 1 c.c., whilst surface waters, such as the Thames at Hampton, contain from 1,000 to 120,000 organisms in the same volume.

2. *Purification by Artificial Processes.*—In connection with waterworks practice, the experiments of Karlinski and others are instructive, showing that the number of bacteria is much larger near the surface of a water basin, and diminishes as the depth increases; hence water companies should draw their supply from a considerable depth, but not low enough to disturb the mud at the bottom which often swarms with bacteria. The great importance of ample storage accommodation has been already referred to, and in the periodical cleaning of storage tanks, the muddy water should on no account be allowed to pass on to the filters; experiments made at the Altona waterworks showed that the settled mud may contain upwards of 17,000,000 bacteria per c.c.

As regards sand filtration, the author's early experiments showing that the number of bacteria in the filtrate depends upon the rate of filtration, have been verified by numerous investigators, and there has since been a gradual tendency to reduce the rate. Thus at the London waterworks, the average rate per square foot per hour fell from 1·73 galls. in 1868 to 1·28 galls. in 1885. The new filters erected at Hamburg in 1892–93 were designed to deliver only 1·27 galls., but the more recent filters built at Tegel for the Berlin supply yield 2·05 galls. At Zürich, however, where the unfiltered lake water is of high quality, a filtration rate of 5·58 galls. per hour yields excellent results. Uniformity in the rate of filtration is of great importance, and great care must be taken not to disturb the layer of slime which forms upon the surface of the sand. The importance of this surface slime, first proved experimentally by Fränkel and Piefke, is now generally recognised, but it is necessary to have a sufficient depth of fine sand beneath it to obtain a filtrate of satisfactory bacterial purity. Thus Reinsch has shown that water which had passed through the slime layer still contained 29 per cent. of the bacteria present in the unfiltered water, but 40 cm. of fine sand beneath the slime layer reduced the remaining 29 per cent. to less than 1 per cent. As the power of arresting bacteria does not diminish with the age of a filter bed, scraping should only be practised when rendered necessary by a diminished yield, and the depth of fine sand must never be too far reduced. The German Imperial Board of Health has fixed 12 ins. as the minimum thickness of fine sand. The process of "airing" to increase the yield of a filter is strongly condemned.

The size of the sand grains has a great deal to do with the efficiency of filtration, and the Lawrence experiments have shown that with very fine sand having an "efficient size" of 0·09 mm. to 0·14 mm. and a depth of 4 to 5 ft. it is almost impossible to force through more than a very small percentage of the bacteria, even when no slime layer exists. The finest sand which will give the required yield should therefore be used. The relative merits of intermittent *versus* continuous filtration have formed the subject of extensive experiment at the Lawrence Station, but sufficient data has not been accumulated for a final conclusion to be drawn. The fact that frost in winter may render intermittent filtration inoperative, condemns it from a practical point of view. The Lawrence experiments have, however, taught the important fact that the more vigorous nitrification which goes on under "intermittent" conditions was no bar to the passage through the filter, in a living state, of all the bacteria experimented upon. Attention is finally drawn to the deleterious influence of frost. The exceptionally large number of bacteria found in some of the London waters on two occasions during the winters of 1886 and 1887 coincided with a very low temperature of the river water, and it is of great importance that the deleterious influence of frost, at a time of year when the more efficient filtration is needed, should be reckoned with and guarded against.

Mechanical analyses by Mr. Allen Hazen of the sand actually employed at many of the chief European waterworks, and the Regulations issued by the German Government in regard to the filtration of surface waters used for public water supplies, are given in two appendices.—L. A.

Drinking Water, Analysis of. G. Romijn. Nederl. Tijdschr. v. Pharm. Chem. en Tech. 1897, 87.

See under XXIII., *page* 467.

Lead Poisoning, A Cause of: Artificial Flower Making. J. B. Charcot and P. Yvon. J. Pharm. Chim. 1897, **5**, 375—379.

The authors investigated the cause of the lead poisoning in the case of an artificial flower maker, whose work consisted in wrapping strips of paper (the end moistened on the tongue) round the stems of the flowers. An examination was made of all the papers used in flower making. Some do not contain lead, but a series ranging in colour from greenish-yellow to greenish-blue, contain the metal in the form of chromate. This may be extracted by caustic potash, and precipitated from the solution by acetic acid. In quantitative determinations the paper was burnt, and the lead converted into sulphate. The greater number of the papers examined, contained from 0·147—0·888 grm. of lead as chromate in each sheet (76 × 50 cm.). The authors

find that saliva (which is alkaline) is able to dissolve appreciable quantities of lead chromate, and to this property they attribute the absorption of the metallic poison.
—A. C. W.

PATENTS.

Sewage Filters, Automatically Alternating the Delivery of Liquid to Two Chambers or Receptacles; Impts. in Apparatus for, particularly applicable to. D. Cameron and F. J. Commin, Exeter. Eng. Pat. 3003, Feb. 10, 1896.

The effluent from the filter which is in use rises into a well, and when the well is full the liquid overflows into a bucket fixed at one end of a rocking lever. This bucket, in filling, overweights the end of the lever, which descends, and in doing so, diverts the sewage on to another filter, at the same time opening a valve at the bottom of the well, and causing the effluent from the full filter to be rapidly discharged.—L. A.

Sewage or Impure Waters, Impts. in the Manufacture of Materials or Compounds for Use in the Treatment of. H. B. Sharp, Heaton Moor, near Stockport, Lancaster. Eng. Pat. 6132, March 19, 1896.

Finely-ground mill or tap cinder is treated with sulphuric acid diluted with twice its weight of water, and the solution of ferrous sulphate is then further diluted with water and decanted from the insoluble residue. Part of the clear solution having been mixed with finely ground ore containing aluminium and iron, *plus* more sulphuric acid, is heated until the ore is dissolved, and, while boiling, the rest of the ferrous sulphate solution, to which sodium nitrate has been added, is run in. The "nitric acid gas" liberated during oxidation is condensed in water, and the solution of iron and aluminium sulphates is boiled down and allowed to solidify into blocks.—L. A.

Sewage and Impure Liquids, Improved Apparatus for Filtering. W. Birch, Manchester. Eng. Pat. 9374, May 4, 1896.

Improvements are referred to in the apparatus described in Eng. Pat. 4372 of 1890 (this Journal, 1891, 61). The endless filtering band is made of cotton cloth having a raised pile surface and occasional cross wires or plates to keep it of proper width and flat; two fixed guide plates keep the band in position. The cleansing roller presses the band upon the circumference of a drum covered with thick felt, and jets of water playing upon both surfaces of the band assist in loosening and removing the deposit.—L. A.

Sewage and other Liquids, Impts. in the Purification of. E. W. T. Jones and J. Garfield, Wolverhampton. Eng. Pat. 11,243, May 23, 1896.

The filtering medium is composed wholly or in part of broken coal or bituminous shale. Preferably, the filter is composed entirely of coal, arranged as follows, from the bottom upwards:—6 ins. of about $\frac{1}{2}$-in. cube, 12 ins. of about $\frac{1}{4}$-in. cube, 30 ins. of about $\frac{1}{16}$-in. cube, 24 ins. of slack and fine coal.—L. A.

Sludge or Deposit from Foul Waters and the like, Impts. in and Apparatus or Means for Treating the. H. W. Kearns, Manchester. Eng. Pat. 15,208, July 9, 1896.

A rectangular brick tank is divided into a serpentine channel by parallel walls, which extend alternately from one outside wall nearly to the opposite one. The tank is completely roofed in by brick arches springing from the dividing walls. On the bottom, rows of drain pipes are laid, covered by furnace ashes. One end of the channel communicates with a furnace and the other end with a chimney. The sludge is run on to the ashes, and the water is allowed to drain away. The furnace is then lighted, and the sludge is dried and burnt by the hot air and gases passing over it.—L. A.

Town Sewage, Refuse, and the like; A New or Improved Method for the Treatment of, for the Destruction of Animalculæ and the Production of a Fertilising Compound. S. de Petroff, Brussels, Belgium. Eng. Pat. 23,676, Oct. 24, 1896.

The essential part of the process is the distillation of sewage with lime slags from dephosphorising processes, condensing the ammonia in a solution of acid calcium phosphate.—L. A.

(C.)—DISINFECTANTS.

PATENTS.

Organic Substances, Impts. in or relating to Preservation of [by Hydroxyquinoline and its Salts]. A. J. Boult, London. From F. Fritzsche and Co., Hamburg, Germany. Eng. Pat. 7480, April 8, 1896.

Hydroxyquinoline and its acid and neutral salts, as well as such soluble double salts as hydroxyquinoline potassium sulphate (quinosol), have an extraordinary preservative power. The body of a freshly killed rabbit, if sprinkled with, or preferably rubbed with, fossil meal containing 4 per cent. of quinosol, will remain sweet if only 1 grm. of the powder be used. Meat dipped in dilute quinosol solution becomes dry without becoming foul; eggs may be preserved in the solution, and liquids such as sugar solution, beer, &c., are preserved by the addition of 1 part in 10,000 to 1 in 100,000 of quinosol. Fruits may be preserved by embedding them in fossil meal containing quinosol. The patentee claims the above-described methods of preserving food, &c., by means of hydroxyquinoline and its salts. (See this Journal, 1896, 609 and 917.)—L. A.

Disinfecting, Cleansing, and Deodorising Mixture, A Combined. W. Hanlon, Innishannon, Cork, Ireland. Eng. Pat. 8712, April 25, 1896.

A mixture of potassium permanganate and sodium carbonate.—L. A.

Formaldehyde, Manufacture of Chemical Compounds of Starch and Dextrin with. A. Classen, Aix-la-Chapelle, Germany. Eng. Pat. 1144, Jan. 15, 1897.

Starch, dextrin, gum arabic, or any other similar substance is mixed with formaldehyde (40 per cent. solution) and heated under pressure, in order that the formaldehyde may react on the starch or other substance. About 24 hours at 124° C. is necessary, and this must be repeated with fresh formaldehyde. The excess of formaldehyde is removed from the finished compound by water, and the product dried at 100° C.

This product may be used for treating wounds, &c. when the use of formaldehyde itself is not possible.—A. L. S.

XIX.—PAPER, PASTEBOARD, Etc.

Paper, The Action of Sunlight upon Size contained in. W. Herzberg. Mitt. k. t. Versuchsanst. zu Berlin, **14**, [506], 309—316.

A series of observations as to the action of sunlight upon resin-sized paper were made at the German Papier-Versuchsanstalt in 1889 (this Journal, 1890, 98—99). Five different samples of resin-sized papers, which were proved to be fully sized by Leonhardt's method, and upon which characters written in ordinary ink were clear and sharp, were exposed between two windows, upon which the sun shone only in the mornings, on June 13, 1888, and were taken out and again examined and tested on October 26, 1888, after four months' exposure, with the following results:—

No. 1. A white document paper, made from linen and cotton, without mineral loading. The ink characters slightly blurred, and the paper was not fully sized.

No. 2. A white foolscap, made from linen, cotton, and wood cellulose, without mineral loading. The ink characters blurred, the thicker strokes sinking into the paper, which was only about half sized.

No. 3. A yellowish common writing paper, made from chemical and mechanical wood pulp, and containing 13·5 per cent. of ash. The ink characters remained clear, the paper being fully sized.

No. 4. A white foolscap, made from straw, wood cellulose, cotton, and linen, containing 13·8 per cent. of ash. All the ink characters blotted and the thicker ones sunk into the paper. The paper was only about half sized.

No. 5. A document paper made from unbleached linen, without mineral loading. The results were similar to those in No. 2.

No. 5 was unaltered in appearance; Nos. 1, 2, and 4 were turned slightly brown, and No. 3 was distinctly brown.

These observations led to experiments in which resin- and animal-sized papers were exposed to light to note its effect upon the size, and the following results were obtained:—

No.	Name of Size.	Composition of Pulp and Amount of Ash.	Observations made on the 1st Dec. 1889.	1st Jan. 1890.	1st Feb. 1890.	1st Mar. 1890.	1st April 1890.	1st Dec. 1891.	Appearance of Paper on Dec. 1st, 1891.
I.	Resin	Wood cellulose and straw cellulose. Ash 18·5 per cent.	Characters clear and sharp, hardly to be seen at back.		Characters clear, but show through a little more.	Characters clear, but thick strokes show through.	Characters blot slightly, and show through strongly.	Characters blot and sink in.	Slightly brown.
II.	,,	Cotton and linen, with addition of straw and wood. Ash, 16·8 per cent.	No observation on this date.	Characters clear, but thick ones visible on back.	Characters blot, show through, and thick strokes sink.	Characters, especially thick ones, blot, and mostly sink in.		All characters totally blot, and all show through.	Very brown.
III.	Resin and animal.	Cotton, with a little linen. Ash, 1·3 per cent.	Characters are clear and sharp, and not visible at the back.					All characters blot, but are not visible on the back.	Brown.
IV.	,,	Cotton and linen, with some straw and wood. Ash, 18·5 per cent.	Characters clear and sharp, not visible at the back.				Characters blot slightly, and some show through.	All characters blot, the thick ones sink and show through.	Slightly yellow.
V.	Animal	Linen, with a little cotton. Ash, 3·5 per cent.	Characters clear and sharp, slightly visible at back.				Characters clear and sharp, but a few thick ones show inclination to blot.	All characters blot and show through, the thicker strokes sinking right through.	,,
VI.	,,	Cotton and linen. Ash, 1·5 per cent.	Characters clear and sharp, and visible at the back.					All characters blot and show through, the medium and thick strokes sink through.	,,

No. II. in the table was specially sized for exposure, by soaking an entirely unsized plate-paper in an ethereal solution of resin; thus it contained only resin size. This sample lost its size very rapidly. It will be observed from the table that the animal-sized papers also deteriorated, and this being contrary to the observations of other experimenters it was determined to expose some papers to the influence of the atmosphere, but to cover parts of the samples from the action of light. At the end of the experiment the covered parts of the resin-sized papers were proved to have kept their size, but the covered parts of the animal-sized papers had deteriorated. This proves that in the case of resin-sized papers, it is the light which causes the deterioration, but with animal-sized papers it is evidently some other destructive agent.

Samples of both resin- and animal-sized papers were exposed to a temperature of 98° C. for some time, but did not lose their size even when the papers had been heated so long that they crumbled to pieces on handling; so that heat has no effect on the size.

During last year other experiments were made by different experimenters (Papier Zeit. 1895, No. 94–99), with the following results:—

Wolesky and Haase ascertained from their observations:—

1st. That the composition of the pulp does not play any part in the deterioration of the size by sunlight.

2nd. That animal-sized papers are not much affected by the light.

3rd. That resin-sized, highly glazed papers show a considerable deterioration, the ink characters blotting and going through after three to four weeks' exposure.

4th. That unglazed resin-sized papers resist the action of light slightly more than glazed, but the resistance only affects the result by a few days.

5th. That only the side exposed to the sun loses the size.

Liesegang obtained the same results as the Versuchsanstalt obtained in 1888. Paper which did not contain ground wood-pulp deteriorated, but paper containing ground wood did not deteriorate on exposure to sunlight.

On comparing these results some contradictions may be noticed:—

1. Wolesky and Haase and the later experiments of the Versuchsanstalt show that the composition of the pulp has no influence on the deterioration of the size, while Liesegang and the former experiments of the Versuchsanstalt show that ground wood does affect the deterioration.

2. The animal-sized papers were not altered in Wolesky and Haase's experiments, but in the experiments conducted by the Versuchsanstalt they completely lost their size.

It is hardly possible to state the exact reason why contradiction No. 1 should occur. By further experiments it was, however, shown that resin- and also animal-sized papers lost their size on exposure to sunlight, whatever their composition, if exposed for a sufficient time.

Different reasons have been advanced to explain the loss of size. Wolesky and Haase suppose that the resin enters into combination with the fibres of the paper, and that the sunlight decomposes the compound thus formed. They say that if the resin mechanically filled the spaces between the fibres, the light would have no effect; but this supposition may be wrong, as it has been observed that when resin is exposed to sunlight it cracks and numerous flaws appear, and this may be what takes place in the paper.

Liesegang is of opinion that the deterioration is caused by chemical action. He observed that a paper which had been exposed to sunlight on one side only, when dipped into an alcoholic solution of pyrogallol, turned an intense orange-brown colour on the exposed side, the other side remaining almost colourless. Papers containing ground wood did not show any difference between the sides on being treated in the same manner, and therefore Liesegang came to the opinion that ground wood acts in the same manner as the sodium sulphite acts with the pyrogallol in photography: the sodium sulphite, being more easily oxidised than pyrogallol, is oxidised first, and prevents the solution going speedily brown. Liesegang says that in the same manner the light acts first upon the ground wood in the paper before taking effect upon the size.

Dr. Burkhardt's opinion is as follows :—The sizing with resin is a purely physical occurrence, the size being fixed between the fibres in a finely-distributed state, and by the influence of sunlight the resin is decomposed, and its sizing power lessened. On animal-sized papers no deterioration by sunlight can be proved.

A fact that may have some bearing is, that when resin is exposed to light it becomes insoluble. On the papers Nos. I. to IV. in the table, before exposure, a distinct ring of resin was formed by dropping a little ether on them, and allowing it to evaporate; but no such ring could be obtained after exposure.

With reference to the deterioration of animal-sized papers in the parts covered from the light, it is probable that the deterioration was produced as follows: In consequence of the change of temperature and moisture in the air, fine cracks are produced in the thin coat of animal size, through which cracks the ink penetrates. This supposition is corroborated by the manner in which the ink characters spread. The same result is observed when an animal-sized paper is rubbed between the hands and afterwards written upon.—S. P. E.

PATENT.

Paper Machines, New or Improved Apparatus for Cleaning and Sorting the Water derived from. O. Schmidt, Andritz, near Graz, Austria. Eng. Pat. 6855, Mar. 28, 1896.

An arrangement of tanks and connecting pipes for mechanically treating, on the continuous flow system, the water from a paper machine, so as to intercept and return the pulp to the paper machine, eliminate the greasy scum, and fit the clear water for use in the rag engines.—L. A.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

Hydrogen Peroxide, Preservation of. Sunder. Bull. Soc. Ind. Mulhouse, 1897, 95–96.

The addition of a small quantity (up to 2 per cent.) of alcohol or ether is effective in retarding the decomposition of hydrogen peroxide solutions. A solution of hydrogen peroxide at 5·6 volumes, reckoned to a litre of 100, and containing the amount of alcohol above stated, tested 96·2 per cent. after standing for two days, and 48·9 per cent. of its original strength after 84 days, whilst, without this addition the numbers obtained were 78·6 and 1·3 respectively.—T. A. L.

Note on the Preceding. G. Freyss. Bull. Soc. Ind. Mulhouse, 1897, 97.

The author reports that he has examined the method proposed by M. Sunder, and can testify to its efficiency. The titrations were made with potassium permanganate in a cold solution, acidified with sulphuric acid, under which conditions absorption of the permanganate is not due to oxidation of the alcohol.—T. A. L.

Alkaloids isolated from a Species of Jaborandi, Two New. A. Petit and M. Polonovski. J. Pharm. Chim. 1897, **5**, 369—370.

By submitting 1 kilo. of the leaves of *Pilocarpus spicatus*, a species of jaborandi, to the treatment generally employed for that plant, the authors obtained 3 grms. of a mixture of bases, the nitrates of which, crystallised from alcohol, melted at about 140° C. The mixture of nitrates, on shaking with caustic soda and chloroform, gives up one base to the solvent, whilst the other remains in the form of a salt. The former, for which the name pseudo-jaborine is adopted, is a colourless syrup with a very alkaline reaction, soluble in water, alcohol, and chloroform; the base and its salts are inactive. The nitrate crystallises from alcohol in large thin plates, melting at 158° C.; the hydrochloride forms small prisms with the melting point 222° C. The second base, pseudo-pilocarpine, is similar to pilocarpine, but has no action on polarised light. Its nitrate crystallises in small needles which melt at 142° C., its hydrochloride in small prisms, very soluble in water and alcohol, and melting at 198°—199° C.—A. C. W.

Pilocarpine and Pilocarpidine. A. Petit and M. Polonovski. Journ. de Pharm 1897 (6), **5**, 370, 130.

The authors find that pilocarpine and pilocarpidine may be readily separated in a state of purity from the other alkaloids of jaborandi by reason of the property they possess of forming a definite compound with solutions of fixed alkalis, from which they are not washed out by chloroform, while the other alkaloids are removed; after this treatment, on neutralising the alkaline solutions with an acid and liberating the two alkaloids with ammonia, they are readily removed by means of chloroform.

Although the isolation of these two bases is thus relatively easy, their separation from each other is not so simple. The solubility of the two nitrates is so nearly identical, that fractional crystallisation of these salts is useless. It is found, however, that by crystallising the mixed hydrochlorides, an excellent separation is obtained; so that by three successive crystallisations from alcohol, the greater part of the pilocarpine hydrochloride is obtained in a state of purity. The pilocarpidine in the mother liquors is recovered in two crystallisations from alcohol. Pilocarpidine may also be readily obtained from pilocarpine by treating that base with sodium ethylate.

One grm. of metallic sodium is dissolved in 30 grms. of absolute alcohol, 3 grms. of pilocarpine dissolved in the same amount of alcohol are added, and the mixture heated on the water-bath for several hours. When reaction is completed the alcohol is distilled off and the alkaline residue extracted with chloroform, which removes the decomposition products. It is re-dissolved in hydrochloric acid, the base liberated with ammonia, and removed by means of chloroform or ether.

At present, pilocarpine has been isolated solely in the form of an amorphous syrup, but the authors hope ultimately to obtain it in a crystalline condition. It is readily soluble in water, alcohol, or chloroform (Hardy and Calmels state that it is but slightly soluble in the last solvent), less soluble in benzene, and almost insoluble in petroleum ether. It obstinately retains traces of the solvent by which it has been extracted, and requires prolonged exposure over sulphuric acid to render it perfectly dry. A 2 per cent. solution at 18° C. has the optical rotation $[\alpha]_D = +106°$, and the salts are also strongly dextro-rotatory. The nitrate crystallises from water in large transparent prisms, from alcohol in small prismatic needles, melting at between 177°—178° C. Its optical rotation in a 2 per cent. solution at 18° C. is $[\alpha]_D = +82·2°$. The amount of combined nitric acid determined by titration, corresponds to the formula $C_{11}H_{16}N_2O_2HNO_3$. The solubility in water at 18° C. is 1 in 6·95; in 95 per cent. alcohol at the same temperature, 1 in 146.

The pilocarpine hydrochloride occurs in transparent prisms melting at 200° C. It contains no water of crystallisation. Its optical rotation in 2 per cent. solution at 18° C. is $[\alpha]_D = +91°$. The amount of combined hydrochloric acid agrees with the formula $C_{11}H_{16}N_2O_2HCl$. Its solubility in water at 18° C. is 1 in 0·4; in 95 per cent. alcohol, 1 in 10·4. The hydrobromide, sulphate, salicylate, and picrate were all obtained in a crystalline form. Although Hardy and Calmels have stated that four gold salts may be obtained according to varying conditions, the authors have only been able to prepare two, the acid monochloro-aurate, $C_{11}H_{16}N_2O_2HClAuCl_3$, melting, when anhydrous, at 130° C.; and its modified form, melting at 167° C., and not at 86° C. as stated by Hardy and Calmels: this has the formula $C_{11}H_{16}N_2O_2AuCl_3$. The first-named salt is obtained by the interaction of pilocarpine and gold chloride in the presence of hydrochloric acid. It forms lemon-yellow crystals containing 1 mol. of water of crystallisation. The modified form is obtained by treating this salt plentifully with boiling water. The solution becomes markedly acid and begins to deposit the modified chloro-aurate in the form of small bright yellow lamellæ.

Pilocarpidine closely resembles pilocarpine in its chemical reactions, but differs in physiological effects, in the melting

points of its salts, and in its optical properties; the combinations of its salts with the fixed alkalis, on account of the formation of pilocarpidic acid are slightly levo-rotatory.

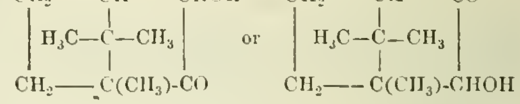
As usually obtained, pilocarpidine is a white syrup, but from the pure nitrate the authors have obtained it in large oblique prisms, very hygroscopic, and soluble in water, in alcohol, and in chloroform. The optical activity of a 2 per cent. aqueous solution at 18° C. is $[\alpha]_D = +50°$. The salts are all dextro-rotatory, but the combination with fixed alkalis, as stated above, becomes levo-rotatory. The nitrate is obtained by crystallising from water in large prisms or transparent tablets. From boiling alcohol it is deposited in brilliant white scales melting at 158° C. Its optical rotation in a 2 per cent. solution at 18° C. is $[\alpha]_D = +38 \cdot 5°$. The formula is found to be $C_{11}H_{16}N_2O_2HNO_3$ and its solubility in water is 1 in 8·04; in 95 per cent. alcohol 1 in 135, both at 18° C. The hydrochloride crystallises from a mixture of alcohol and ether in brilliant lamellæ, containing approximately $\frac{1}{2}$ a mol. of water of crystallisation. It melts at 124° C., and is completely dehydrated at 100° C. when it melts at 161° C. The determination of the hydrochloric acid in the dry salt agrees better with the formula $C_{11}H_{16}N_2O_2HCl$ than with $C_{10}H_{14}N_2O_2HCl$. The hydrobromide, salicylate, and picrate were all obtained in a crystalline state. The chloro-aurates corresponded precisely with the similar salts of pilocarpine. The authors find, moreover, that the precipitate obtained on treating pilocarpidine hydrochloride with gold chloride is not soluble in excess of that salt. In this their results are diametrically opposed to the statements of Merck, Hardy and Calmels, and Harnack, who state that it is soluble. The salt melts at 160° C., and contains no water of crystallisation. The formula is $C_{11}H_{16}N_2O_2HClAuCl_3$. To boiling water this salt gives up the molecule of hydrochloric acid, and the resulting modified chloro-aurate, $C_{11}H_{16}N_2O_2AuCl_3$, crystallises in bright yellow lamellæ melting at 190°.—J. O. B.

Oil of Celery, High-Boiling Constituents of. G. Ciamician and P. Silber. Ber **30**, 492—501.

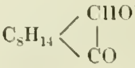
In a previous paper (Ber. **29**, 1799) on the oil of *apium graveolens* L. (oil of celery), the authors showed that it contained limonene. That it did not consist wholly of this was at once evident from the smell, which pointed to the presence of a non-terpene-like substance. Commercial oil of celery, however, does chiefly consist of terpenes as it appears that a very small proportion of the odorific principle will scent a large quantity of the oil. The examination of the higher boiling portion of oil of celery showed that it contained a terpene, probably $C_{15}H_{24}$, palmitic acid and phenolic substances, a lactone $C_{12}H_{18}O_2$, and an acid $C_{12}H_{18}O_3$. The lactone is present as such in the plant whilst the acid is only obtained after saponification. Palmitic acid and the phenols are only present in very small quantities, and the terpenes are found principally in the first portions of the distillate. The "latter runnings" obtained on distilling oil of celery were extracted with cold dilute potash ($2\frac{1}{2}$ per cent.) to remove palmitic acid and phenols, and the residual oil which forms the bulk, was treated with twice its weight of caustic potash solution of 25 per cent. strength and heated in an oil bath under an inverted condenser. The oil unacted on, was removed with ether and gave results on analysis corresponding with the formula $C_{15}H_{24}$. It boils at 262°—269° C., and forms about 80 per cent. of the total "latter runnings." The alkaline liquid contains the potash salts of two acids, which the authors have termed sedanolic acid $C_{15}H_{20}O_3$ and sedanonic acid $C_{12}H_{18}O_3$. The former is an oxyacid, and is converted with very great ease into the corresponding lactone, sedanolide $C_{12}H_{18}O_2$. After acidifying the alkaline solution, extracting with ether, and distilling off, the oil remaining, is shaken with three times its weight of sodium carbonate, when the sedanonic acid dissolves and the sedanolic acid yields the lactone, which is extracted with ether and distilled under 17 mm., when 80 per cent. passes over at 183°—185° C. In order to obtain sedanolic acid, the lactone is carefully treated with caustic potash and poured into cold dilute sulphuric acid. After filtering off, taking up with ether and precipitating with petroleum ether, the final purification of the sedanolic acid is effected by precipitating it repeatedly from benzene or ether with petroleum ether, avoiding a rise in temperature. The acid crystallises in white needles melting at 88°—89° C. As already observed, it is very easily converted into sedanolide especially on distilling the acid from a small retort. The lactone boils under 17 mm. at 185° C., and forms a thick colourless oil, smelling strongly of celery. Sedanonic acid is contained in the alkaline solution referred to above, obtained by digesting the saponification product of oil of celery with sodium carbonate. The acid separates from benzene in white crystals and melts at 113° C. The substance is an unsaturated ketonic acid. It forms an unstable hydrazone with phenylhydrazine, melting at 130°—131° C. The oxime crystallises from benzene in white shining crystals and melts at 128° C.—T. A. L.

Hydroxycamphor. O. Manasse. Ber. **30**, 659—670.

By the reduction of camphor-*o*-quinone, the author has obtained an oxycamphor which has all the characteristics of a ketone alcohol, and differs from all previous products similarly designated. The substance has one of the two following formulæ:—



and differs in its physiological properties from campherol—



to which, of the oxycamphors hitherto known, it comes nearest in its chemical and physical properties. The camphor quinone was obtained by boiling the solution of nitrosocamphor in bisulphite with dilute sulphuric acid, an 80 per cent. yield being obtained. The reduction is carried out by dissolving the quinone in ether, adding dilute sulphuric or hydrochloric acid, and running in zinc dust suspended in water until the solution is decolorised. After filtering, drying, and distilling off the ether, the product is purified by steam distillation. Other reducing agents (aluminium amalgam, zinc dust and acetic acid) give the same result, and under proper conditions the reaction is a quantitative one. The new substance melts, when pure, at 205° C., it dissolves to a 2 per cent. solution in cold water, and can be precipitated almost quantitatively from its aqueous solution by salt. Its aqueous solution turns the plane of polarisation to the right (specific rotation = 9°·5).

When treated with chromic acid, the substance yields camphorquinone, whilst under the action of hydrogen peroxide it gives camphoric acid. Hydroxycamphor is of pharmacological interest, owing to the fact of its being a soluble camphor derivative, and it possesses the following properties:—It reacts neutral, and a 2 per cent. aqueous solution is without action on albumin, although globulin is thrown down as a flocculent precipitate. On cold-blooded animals (frog), hydroxycamphor has a similar action to camphor, producing brain paralysis, together with a curare-like paralysis of the motor nerve fibres. With warm-blooded animals, the only visible effect is a decreased respiration, and the lowering of the excitability of the respiratory organs has given good results in certain cases. The blood pressure as well as the heart's action are not affected. Hydroxycamphor has proved an active remedy in different forms of malady involving difficulty of respiration.

—T. A. L.

Antipyrine, Combinations with Phenol; Constitution of. G. Patein. Bull. Soc. Chim. 1897, **17**, 314—316.

The author has previously shown that antipyrine combines with phenols, forming compounds in which both constituents retain their properties, so that they are united not through a carbon atom but through one of the trivalent nitrogen atoms, which passes over into the pentavalent condition. He has already indicated, on theoretical grounds,

that it is nitrogen atom No. 2 that acts as the intermediary. He now compares antipyrine with monomethylphenylpyrazolone, in which the nitrogen atom No. 1 is identical with that of antipyrine, whilst the second atom is differently placed. He finds that the pyrazolone derivative in no case combines with phenols, and hence concludes that it is by nitrogen atom No. 2 that the antipyrine may be united to them. The combination of antipyrine with the phenols is irreconcilable with E. von Meyer's formula for that substance (J. prakt. Chem. **54**, 177; see also this Journal, 1896, 670).—W. G. M.

Anethol, Derivatives of; Chlorine Addition and Substitution Products. G. Darzens. Comptes Rend. 1897, **124**, 563—565.

Anethol Dichloride.—Prepared by slowly mixing cooled solutions of anethol and chlorine respectively in carbon tetrachloride, and subsequently removing the solvent by distillation under reduced pressure. The dichloride is a mobile, amber-coloured liquid, which cannot be distilled, even *in vacuo*, without suffering decomposition into HCl and chloro-anethol.

Mono-Chloro-Anethol ($C_6H_4.OCH_3.C_3H_4Cl$).—The preceding compound is distilled, the distillate washed with weak alkali, and fractionated. The chloro-derivative possesses an odour closely resembling that of anethol. It boils, without decomposition, at 258° C., and solidifies at 0° C. The solid substance melts at about 6° C. Specific gravity at 0° C. = 1·350.

Chloro-Anethol-Dichloride.—Forms small white crystals, melting at 35° C., and soluble in all organic solvents. It is obtained when carbon tetrachloride solutions of monochloro-anethol and of chlorine are mixed, and allowed to evaporate spontaneously.

Chloro-Anethol Dibromide.—Prepared on the same lines as the corresponding dichloride, is obtained in small, white crystals, which melt at 45° C.—H. T. P.

Saccharine and its Preparation. Jan Burda. Časopis pro průmysl Chemický, 1897, **7**, 9.

Toluene is best sulphonated, with continuous agitation, by 4 parts of sulphuric acid of 66° B., to which several per cents. of fuming acid, containing 20 per cent. SO_3, have been added. After pouring the product on 1½ parts of ice, the separated ortho acid is converted by means of chalk into the calcium salt, and this, by treatment with soda, into the sodium salt.—J. T. C.

PATENTS.

Pharmaceutical Substance [Thyroid Extract], Impts. in the Manufacture of. Farbwerke vorm. F. Bayer and Co., Elberfield, Germany. Eng. Pat. 9576, May 5, 1896.

The active principle of thyroid glands, iodothyrine, contains iodine, the activity of its preparations being in proportion to the amount of iodine contained in them. This patent therefore claims the improvement on the processes described in Eng. Pats. 12,295, 1895 and 20,827, 1895 (this Journal, 1896, 470 and 740), which consists in boiling the glands with dilute acid or alkalis until the residue contains the greatest or the least quantity respectively of iodine. The process for the isolation of iodothyrine which consists in digestion of the glands with artificial gastric juice and extraction of the residue by alcohol, is claimed, together with processes for the separation of compounds of iodothyrine with globulin and serum albumin by digestion of the glands with dilute solutions of common salt, filtering, and precipitation by boiling the filtrate with a little dilute acetic acid, with or without the previous separation of the globulin compound by dilution or action of carbon dioxide. The patent also claims the process for the isolation of iodothyrine from its albumin compounds before described by the processes previously claimed, and lastly, the globulin and serum albumin double compounds as new products.
—A. C. W.

Cinnamic Aldehyde, Impts. in the Manufacture or Production of. C. F. Boehringer and Söhne, Waldhof, Germany. J. Y. Johnson, London. Eng. Pat. 10,103, May 11, 1896.

The production of cinnamic aldehyde by the condensation of benzaldehyde and acetaldehyde at a low temperature by means of a concentrated alkali solution, is claimed. The alcoholic solution of the aldehydes cooled to −10° is agitated with a 25—30 per cent. caustic soda solution previously cooled to the same temperature. Formation of resin is thus avoided.—A. C. W.

Piperidine and its Homologues, Manufacture of Derivatives from. [Compounds with Phenols and Acids.] J. Turner and Co., Ltd., Queensferry; P. Schidrowitz and O. Rosenheim, London. Eng. Pat. 11,061, May 21, 1896.

In this patent are claimed the compounds produced by the action of piperidine or its homologues on guaiacol, hydroquinone, tannic acid, pyrogallol, nitro-phenols, 1:2:4 dinitro-α-naphthol, *m*-nitro-benzoic acid, 1:3:5 dinitrobenzoic acid, with the process which consists in bringing about the condensation with or without a solvent and with or without the application of heat.—A. C. W.

o-Sulphamine Benzoic Acid Alkyl Esters, and Anhydro-o-Sulphamine Benzoic Acid [Saccharine], Impts. in the Manufacture of. Chemische Fabrik von Heyden, Radebeul, Germany. Eng. Pat. 1164, Jan. 15, 1897.

This patent claims the manufacture of the esters of *o*-sulphamine benzoic acid by heating the acid in alcoholic solution with a comparatively small quantity of mineral acid, the manufacture of the pure acid by oxidation of *o*-toluene sulphonamide or a mixture of the *o*- and *p*-compounds in alkaline solution by potassium manganate, permanganate, or other oxidising agent, and in the case of the mixture separation of the acids by fractional precipitation, and also claims the process for producing anhydo-o-sulphamine benzoic acid, which comprises the two above processes and subsequent formation of the anhydro acid according to known methods. In the first operation the alcohol must be as free as possible from water and concentrated sulphuric acid or hydrochloric acid gas is used.—A. C. W.

XXI.—PHOTOGRAPHY.

Metol. L. Paul. Zeits. f. angew. Chem. 1897, 171—174

This product, which is used as a photographic developer, is the sulphate of methyl-*p*-amidophenol, and is said to be obtained by methylating *p*-amidophenol. The author, however, concludes from his experiments that it is not produced by the direct methylation of *p*-amidophenol, and he obtains it by an indirect method, described later. For the preparation of *p*-amidophenol, he heats 250 grms. of *p*-nitrophenol, 45 grms. of hydrochloric acid (20° B.), and 500 c.c. of water in an iron vessel, provided with an agitator, to 98° C., and adds gradually, in portions of 15 to 20 grms., about 400 grms. of iron borings, so long as a vigorous reaction takes place. The whole is then boiled for half an hour and extracted with about 2 litres of water and 25—30 grms. of sodium carbonate. The filtered solution deposits *p*-amidophenol on cooling and the mother-liquor is used for the next extraction, which is continued as long as *p*-amidophenol crystallises out on cooling. The yield is 140 grms., corresponding to 71 per cent. of the theoretical. By boiling it with methyl alcohol and sulphuric or hydrochloric acid the base remains unaltered. The same is the case if *p*-amidophenol hydrochloride be heated in an autoclave with methyl alcohol to 150°—160° C. By the action of methyl chloride under pressure on the base, higher methylated products are formed. The methyl chloride was obtained by passing hydrochloric acid gas through methyl alcohol and zinc chloride, and condensing the gas in methyl alcohol cooled with ice. Metol can, however, be obtained by boiling for about an hour in aqueous solution, two molecules of *p*-amidophenol and one molecule of chloracetic acid. On cooling, *p*-hydroxyglycine crystallises

out, which on heating turns brown at 200° C., begins to melt at 220° C., and is completely melted at 245°—247° C., when it decomposes into carbonic acid and metol according to the equation—

$$1.4.C_6H_4(OH)NHCH_2CO_2H = 1.4.C_6H_4.OH.NH_2 + CO_2.$$

The metol obtained melted at 86° C. and gave the characteristic nitroso compound.—T. A. L.

Indigo Salt, Action of Light on Kalle's. E. Kopp. Bull. Soc. Ind. Mulhouse, 1897, 87.

See under IV., *page* 432.

PATENTS.

Photographic Emulsions, Impts. in. [*Addition of White of Egg.*] H. Wandrowsky, Cologne-Ehrenfeld, Prussia. Eng. Pat. 5992, March 17, 1896.

The addition of white of egg to emulsions prepared with gelatin, starch, gutta-percha, &c., which is made insoluble after the emulsion has been spread on glass or paper, is claimed.—A. C. W.

Uranium Intensifiers and Reducer, Improved, as applied to Photographic Negatives and Positives. A. Hill and The Cresco-Fylma Co., Ltd., London. Eng. Pat. 10,072, May 12, 1896.

The patent claims the use of uranium acetate in combination with a suitable acid and potassium ferricyanide, for intensifying and reducing negatives and positives; the same mixture, with the addition of strontium nitrate or potassium sulphocyanide, for toning bromides and transparencies; and lastly, tabloids containing amylacetate celluloid, uranium acetate, potassium ferricyanide, and citric acid.—A. C. W.

Colouring Photographs, Improved Means for, suitable also for Dyeing and Printing. J. A. Michel-Dansac and L. Chassagne, Paris. Eng. Pat. 18,131, Aug. 15, 1896.

The following five mixtures are claimed, and their application to photographs on any support:—

Albumin for Shadows.—Dissolve 200 grms. of blood albumin in 500 grms. of water at 37°—40° C.; add to 500 grms. of water, 1 grm. each of the chlorides of platinum, sodium, palladium, ammonium, iron, chromium, cobalt, gold, tin, barium, nickel, strontium, cadmium, mercury, and silver. In a 125-c.c. flask put 10 grms. of cocaine hydrochloride; fill up with water. Let each mixture stand 24 hours. Add to the first solution, 5 grms. of sodium sulphate, 1 grm. of oxalic acid, and 0·25 grm. of corrosive sublimate; take 100 grms. of this mixture, whip up in it a fresh egg and a pinch of hæmoglobin, then mix with the remainder of the albumin, and add the cocaine solution. Finally, mix with the solution of the chlorides, and let stand 24 hours.

Albumin for Reliefs.—Take half of the shadows albumin, add 1 grm. of picric acid, 1 grm. of chromic acid, 0·1 grm. of formic acid, and incorporate with a mixture of 125 grms. of water, 5 grms. of sodium chloride, 1 grm. of platinum chloride and 50 grms. of fresh casein.

Blue Pigment.—To 100 c.c. of the shadows albumin, add a solution of 1 grm. of sodium chloride in 1 kilo. of water; to 100 grms. of this, add 5 grms. of indigo carmine and 5 grms. of oxalic acid; then mix with the remainder.

Green Pigment.—Add 50 c.c. each of shadows and relief albumin to a solution of 1 grm. of sodium chloride in 1 kilo. of water. In 50 grms. of the mixture dissolve 1 grm. each of the chlorides of nickel, chromium, and copper, 1 grm. of sulphate of copper, and 1 grm. of copper nitrate. In a second 50 grms., dissolve 1 grm. of picric acid and 1 grm. of indigo carmine. Mix the two 50-grm. solutions, and then add to the remainder, agitate, and let stand.

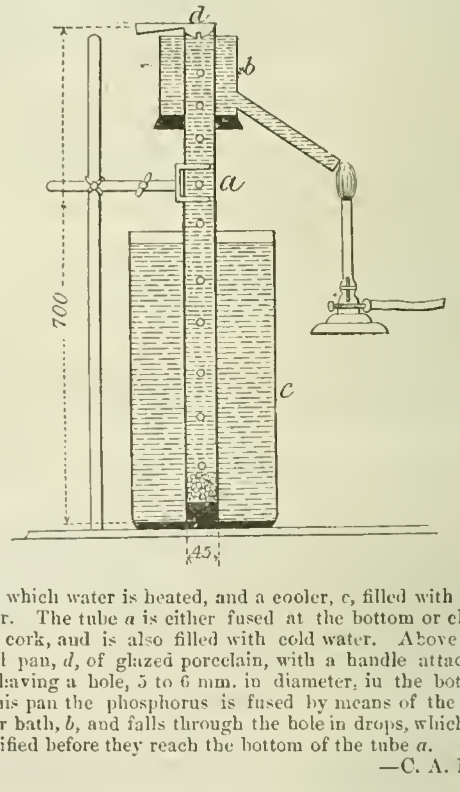
Red Pigment.—Add 100 c.c. of albumin for reliefs to a solution of 1 grm. of sodium chloride in 1 kilo. of water. To 50 c.c. of this solution add 1 grm. of each of the following: cinnabar, iron chloride, iron sulphate, uranium acetate. Add this mixture to a solution of 5 grms. of ammonium sulphocyanide in a second 50 c.c., then add 30 grms. of fresh casein, mix with the remainder, and let stand.

The mixtures should be kept several months before use. The process consists in washing the photograph with the liquids in the following order: shadows albumin, relief albumin, blue pigment, shadows albumin, green pigment diluted with 10 volumes of shadows albumin, reliefs albumin, red pigment diluted with reliefs albumin, shadows albumin; the albumin solutions being diluted for use with 10 volumes of 0·1 per cent. sodium chloride solution. The photograph is not washed between the different applications. —A. C. W.

XXII.—EXPLOSIVES, MATCHES, Etc.

Phosphorus, Apparatus for Granulating. A. Harpf. Zeit. physikal. u. Chem. Unterr. 1896, 9, 286.

This apparatus is constructed on the principle of a shot tower. It consists essentially of a tube *a*, of well-annealed glass 700 mm. long and 45 mm. in diameter, a copper vessel,



b, in which water is heated, and a cooler, *c*, filled with cold water. The tube *a* is either fused at the bottom or closed by a cork, and is also filled with cold water. Above is a small pan, *d*, of glazed porcelain, with a handle attached, and having a hole, 5 to 6 mm. in diameter, in the bottom. In this pan the phosphorus is fused by means of the hot-water bath, *b*, and falls through the hole in drops, which are solidified before they reach the bottom of the tube *a*. —C. A. M.

XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

Hydrometer Scales. G. Rauter. Zeits. angew. Chem. 1897, 215—217.

It is shown that the degrees of the Baume hydrometer are not of equal value. The formula for the so-called rational Baume hydrometer is $d = \frac{144 \cdot 3}{144 \cdot 3 - n}$ where d denotes the specific gravity and n the degree Baume. These values plotted out in rectangular co-ordinates, where $x = d$ and $y = n$, give a hyperbola, the asymptotes of which are the y axis, and the line $y = 144 \cdot 3$, parallel to the x axis.

If $y = n =$ 0,	$x = d =$ 1·000	
1	1·007;	difference 0·007
65	1·819	
66	1·842;	difference 0·023.

Thus from 0° to 66° the value of a degree varies from 0·007 to 0·023 sp. gr. The variation diminishes slowly to − 100°, but increases enormously on passing to 144°.

In addition to this inherent disadvantage, confusion has been introduced by the different bases used by various authors. The Dutch system gives $d = \frac{144}{144 - n}$ where 1·842 sp. gr. corresponds to 65°·8 B. The American, $d = \frac{145}{145 - n}$ where 1·842 sp. gr. is equal to 66°·3 B. Gerlach's is $d = \frac{146{\cdot}78}{146{\cdot}78 - n}$ where 1·842 sp. gr. is 67°·1 B., and others. This hydrometer is therefore not to be recommended for general use.

The Twaddell hydrometer has degrees of equal magnitude, each equal 0·005 sp. gr.

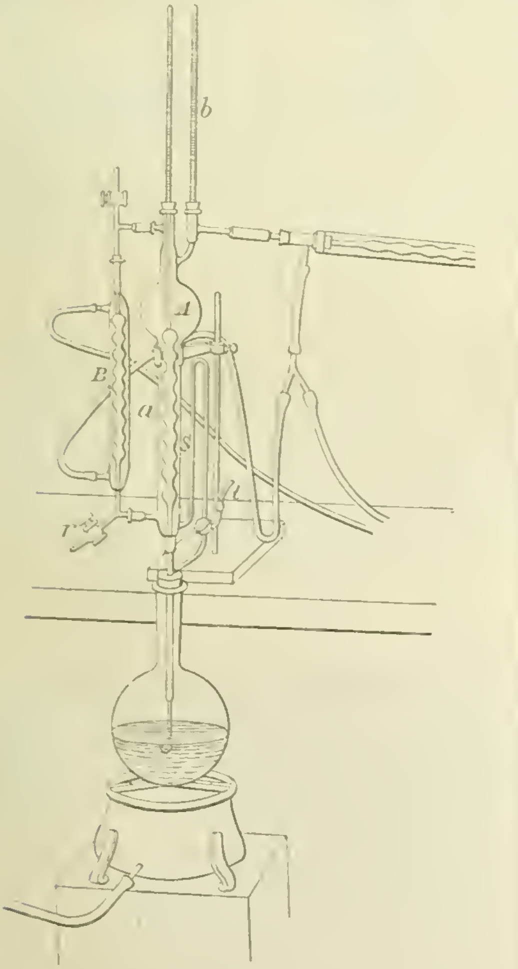
The densimeter of Fleischer is still simpler. Each degree represents 0·010 sp. gr.

The degrees Baume from 0° to — 55°·7 run very closely with degrees Twaddell; degrees Baume from 0 to about 60° do not deviate much from densimeter degrees.

—L. J. de W.

New Fractional Distilling Apparatus, for Laboratory and Industrial Uses. A. Tixier, Chenal-Ferron-Douilhet and Co. Bull. Soc. Chim. 1897, **17**, 392—395.

The apparatus consists of alternate analysers and condensers. The figure shows one analyser A and condenser B. The former is a vertical reservoir *a* traversed by a bulb



tube A connected to the flask; the upper end of the reservoir is furnished with a side tube carrying the thermometer *b* and connected to the condenser. The upper end of the bulb tube A communicates with the side condenser B, which leads into the lower part of the analyser. The siphon S connects the flask with the analyser. The three-way cock with side tube *t* on the siphon enables the action of the siphon to be started when it does not take place spontaneously, and permits of the regulation of its flow. The cock *r* serves to empty the apparatus and for the taking of samples during the progress of the operation.

The flow of water through B, the flow of the siphon, and the rate of boiling may all be separately regulated. When condensation is complete in B, the liquid in the reservoir distils by the heat of the vapours in the bulb tube A; if condensation is not complete in B, the vapours will bubble through the liquid in *a* and analyse it.

The thermometer in the central tube A rises steadily, but that in the side tube passes rapidly from one boiling point to the other.

Several examples of the results achieved by this apparatus are given; one is quoted. A mixture of 1 litre of alcohol of 89·2 and 1 litre of wood spirit for denaturing, containing 250 c.c. of acetone, gave the following fractions:—

C.	C.c.
56—59	245 acetone.
59—66	45
66—69	165 methyl alcohol.
69—79	115

whilst in the flask 830 c.c. of alcohol boiling at 80 were left. Diluted with 50 per cent. of water, the separation would have been more complete. The technical apparatus contains several analysers and condensers; it is fully described in the patent specification.—A. C. W.

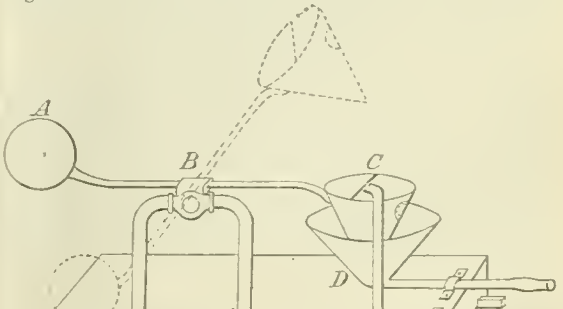
Aluminium, The use of, for Condensers. T. H. Norton. J. Amer. Chem. Soc. 1897, **19**, 153—156.

In his experiments, the author used a condenser so constructed that the condensing vapours did not come into contact with any substance other than aluminium. When organic liquids, such as ethyl alcohol, benzene, nitrobenzene, chloroform, ethyl ether, and acetone were distilled, the residues in the distillate showed that the aluminium was practically not attacked. The results obtained from the distillation of water, demonstrate that as far as the purity of the product is concerned, aluminium possesses about the same advantages over glass as tin, but in lightness and conductivity, it exhibits marked superiority to the latter metal. The high thermal conductivity, and the absence of brittleness in aluminium, are factors in its favour as compared with glass, for distillation purposes.—J. L. B.

Gas-Tap, Automatic. H. Michaelis. Ber. **30**, 282—284.

To obviate the inconvenience and risk liable to occur when working with constant-level water-baths or with Liebig condensers, through failure of the water supply, the author has devised the following gas-tap, which closes automatically when the water current, through any cause, ceases.

The apparatus is connected with both the gas and water supply, and its construction is shown in the accompanying figure.



The gas flows through the tubes *h*, *i* so long as the lever attached to the tap B remains horizontal. This lever carries at one end a weight A and at the other a funnel C. The tube E is connected with the water-supply, the tap being at *g*, and through it water flows into the funnel C, running away partly through a small opening at the apex

of the funnel and partly through an overflow opening somewhat below the funnel rim, into the lager funnel D. So long as the funnel C is filled with water—*i.e.*, so long as the water-feed continues—it balances the equal weight A, and the tap remains open. Should the water-supply cease, the water in C runs gradually away through the small apical opening, and the weight A then bears the lever down until it takes up the position shown by the dotted lines, in which the tap is closed.

The water-supply to the funnel C may be the overflow from the water-bath or condenser. The water must, naturally, be fed into C more quickly than the lower opening in the funnel will allow it to flow into D.

There is also a small hook, not shown in the figure, to hold the lever horizontal, should it be desired to use the burner alone and without the water-supply.—J. T. C.

Chlorine, A Carbon Tetrachloride Solution of. G. Darzens. Comptes Rend. 1897, **124**, 563.

The author remarks on the great advantages resulting from the use of a carbon tetrachloride solution of chlorine for laboratory purposes, and its superiority over other chlorinating agents. A 10 per cent. solution of chlorine may be kept almost indefinitely in a well-stoppered bottle.
—H. T. P.

Reagents free from Arsenic, Preparation of. J. Habermann. Zeits. angew. Chem. 1897, 201—202.

Hydrochloric acid and ammonia sold as pure almost always contain traces of arsenic. When arsenic is contained in ferrous sulphide used for generating sulphuretted hydrogen, it not only passes over with this gas, but accumulates in ammonium sulphide made from the latter. Some years ago the author proposed (Verhandl. d. naturf. Vereins in Brünn, **17**, 11) the use of calcium sulphide, magnesium chloride, and water for the preparation of sulphuretted hydrogen free from arsenic. Sulphuretted hydrogen can be easily freed from arsenic by the method of O. Jakobson (Ber. **20**, 1999). The sulphuretted hydrogen, deprived of the greater part of its moisture, is passed over a small quantity of iodine and then washed by distilled water.

To remove arsenic from so-called "pure" hydrochloric acid of commerce, a small quantity of potassium chlorate is added (less than 0·5 grm. per litre) and the liquid distilled from a glass retort, the neck of which is connected by a Liebig's condenser to a receiver containing so much distilled water that when two-thirds to three-quarters of the contents of the retort have passed over, the liquid in the receiver contains 20 to 25 per cent. of hydrochloric acid. The distillate, of course, contains chlorine, but this is no disadvantage in acid used to destroy organic matter in testing for arsenic. If it be desired to obtain acid containing no free chlorine, the first portion of the distillate is collected separately and the receiver changed as soon as the liquid distils over absolutely colourless. In any case, the whole of the arsenic remains in the residue in the retort.

The traces of arsenic contained in commercial ammonia may be removed by adding a few c.c. of a not too dilute solution of potassium permanganate and distilling from a glass retort into a receiver containing a quantity of distilled water equal to two-thirds of the volume of the ammonia used. It is advisable to interpose between the retort and receiver a small vessel containing water for washing, arranged so as to prevent regurgitation. Ammonium sulphide may then be made from this purified ammonia and purified sulphuretted hydrogen so as to be absolutely free from arsenic.
—L. J. de W.

Gas-Analysis Apparatus. O. Bleier. Ber. **30**, 697—701. (See also this Journal, 1896, 294, 615, 672.)

The jacketed burette A (Fig. 1) contains 100 c.c. between the upper and lower taps h^2 and h^1. This space is divided in $\frac{1}{5}$ c.c., with the exception of the upper 30 c.c., where the tube widens. If volumes smaller than 30 c.c. have to be measured, a graduated levelling-tube, Fig. 3, is used. The upper tap h^2, shown in detail in Fig. 2 (*a* and *b* are alternative forms), puts the measuring-tube in communication with any one of four capillary tubes, to three of which gas pipettes I., II., and III. are attached; the fourth capillary serves for the admission of the sample and for the attachment of additional pipettes.

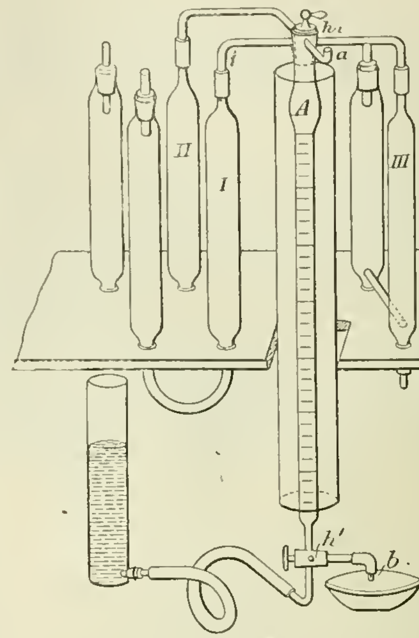


Fig 1

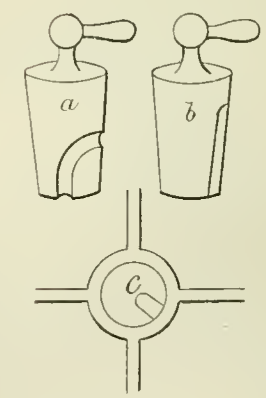


Fig. 2.

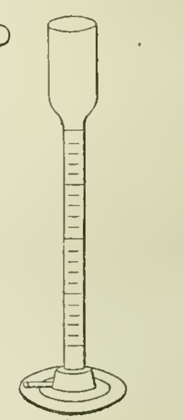


Fig 3

After ascertaining that each capillary is filled up to the tap h^2 by the absorbing liquid from the pipette, the burette is put into communication with the capillary *a*, and the reservoir is then raised until burette and capillary are filled with water. The gas is now allowed to enter at *a*, the water in the burette flowing out at *b*. Analysis follows in the ordinary way, by putting the burette in communication with each pipette in turn.

The tap h^2 may have as many as 6—8 communicating tubes; and, if necessary, two or more pipettes may be placed in series, as in the Orsat apparatus.

The ordinary apparatus used for subjecting gaseous mixtures to the action of the electric spark or of electrically-heated wires may be advantageously replaced by the explosion tubes *a*, *b*, and *c*, Fig. 4. These are capillary tubes carrying a bulb, through which wires are fused, or into which wires are carried through a rubber stopper, as in *c*. The method of using these tubes is as follows:—The tube employed is connected to a pipette and filled with water; the open end is then connected to the vessel (or burette) containing the gas, and the electric circuit closed, the current passing through the water. Now the gas is allowed

to enter. The explosion is not transmitted by the wet capillaries, and in this way any quantity of an explosive mixture may be safely burnt by one passage from pipette

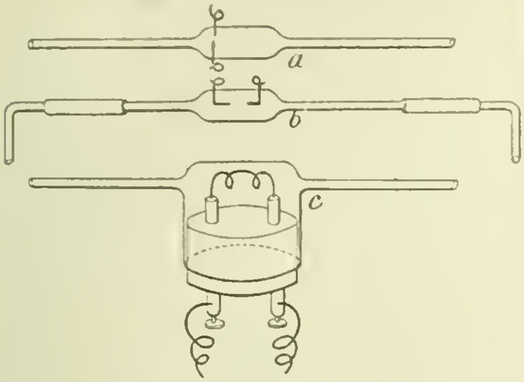


Fig 4

to burette. If, however, the explosive mixture contain other gases, the passage through the capillary must be several times repeated. The tube c is specially adapted to the combustion of methane and other hydrocarbons; in combination with the author's apparatus for gas titration (this Journal, 1896, 672), more accurate results are obtained than with the burette.

The tube b is suitable for direct nitrogen determinations. The mixture, with oxygen in the proper proportions, must be led backwards and forwards between the burette and a pipette filled with dilute alkali The rapidity of absorption is less than was observed by Rayleigh and Ramsay with a spark of equal length. More rapid absorption is obtained by the use of several capillaries in series.—A. C. W.

PATENTS.

Thermometer, An Improved. A. Luraschi, 15, Cross Street, Hatton Garden, London. Eng. Pat. 8547, April 23, 1896.

The thermometer has within its bulb (an outer bulb) a spiral tube, which forms an upward continuation of the fluid index column of the thermometer, the lower end of the spiral being connected with the bore of the thermometer stem, of which it is the continuation. The spiral continues upwards to the highest part of the interior of the bulb, and there it terminates, remaining open to the interior space there. The bulb and the upper part of the spiral are filled with a suitable fluid, and a small quantity of mercury is introduced into the stem and the lower part of the spiral.
—R. A.

Butter, Margarine, and the like, and in the Method of Employing such Apparatus; Impts. in and relating to Apparatus for Testing. R. Wells, Scarborough. Eng. Pat. 10,627, May 18, 1896.

The method depends upon the difference of time in the solidification of butter and margarine, after they have been melted by a gentle heat. The melted fat is poured upon the surface of water contained in a glass cylinder, the temperature of the water being 45°—55° F. If the article tested be pure butter, it is stated that solidification will ensue in from 15 to 20 minutes. If adulterated with margarine, a longer time will be required, and, in accordance with a table given, the proportion of admixed margarine may be read off.—W. P. S.

INORGANIC CHEMISTRY.—QUALITATIVE.

Drinking Water, Analysis of. G. Romijn. Nederl. Tijdschr. v. Pharm. Chem. en Toxicol. 1897, 87—98.

In order to lessen the time required for an analysis, and also to prevent disagreement between analysts, the author proposes a scheme which does not require a quantitative process, but which still gives sufficient data as to the composition of the sample. For example, if the water in a certain locality never contain more than 0·03 grm. of chlorine per litre, it is best tested by taking 200 c.c. of the sample, adding a little potassium chromate, and then 5 c.c. of a solution of silver nitrate containing 5·75 grms. of that salt per litre. If now the precipitate remain white, there is at once the proof of an abnormal excess of chlorine. In a similar manner excessive quantities of lime, sulphates, &c. may be proved by adding to a measured quantity of the sample, a solution of ammonium oxalate in dilute acetic acid, or a solution of barium chloride of known strength. After filtering there should be no further turbidity produced by these reagents. As regards ammonia, the author clarifies the water by means of sodium carbonate and then adds Nessler solution, and nitrites are tested for with meta-diamidobenzene in presence of sulphuric acid; their very presence should condemn the sample. Organic matter is determined by means of permanganate solution; any sudden excess required, must be looked upon as suspicious. Metals should be entirely absent.—L. de K.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Liquid Ammonia, Analysis of Commercial. A. Lange and J. Hertz. Zeits. angew. Chem. 1897, 224—228.

Although liquefied ammonia has been used in the refrigerating industry for some years, and has been prepared on the large scale in Germany, nothing has hitherto been published on the subject with the exception of the work of Strombeck on American ammonia (this Journal, 1892, 736), and a recent method of analysis by Bunte and Eitner (J. für Gasbel., March 13, 1897). The authors have examined the impurities of a number of German products without attempting a quantitative separation.

If a large quantity of commercial ammonia, say 40 lb., be allowed to evaporate spontaneously, a residue is left of a yellow or dark-brown colour which, besides mineral impurities (oxide of iron), often contains a small quantity of machine-oil floating on the surface, and removable in a separating funnel. Ammonium carbonate is almost always present, often crystallised in fine needles. The residue also naturally contains more or less NH_3 in solution. By treatment with dry potassium carbonate the liquid was freed from the water, and the greater part of the ammonia driven off, the remainder being removed by heating under a vertical condenser. The remaining liquid was purified from the brown colouring matter by a single distillation. It distilled between 70° and 150° C. This was separated into fractions boiling at 70° to 90° and 116° to 130° C. The intermediate portion was a mixture of the two. The higher fraction consisted principally of pyridine and its higher homologues; that boiling above 140° smelt strongly of nicotine. Occasionally from the residue of the distillation which contains machine-oil, scales separate out which were recognised as naphthalene by the melting point, 80° C.

The fraction boiling from 70° to 90° C. had a distinct smell of pyridine, which could not be removed by repeated distillation. When much pyridine is present, it may be estimated by titration with $\frac{1}{2}N$-H_2SO_4, and methyl orange as indicator. The quantity of concentrated sulphuric acid calculated from the titration is added to the strongly cooled liquid, and the lighter layer separated from the heavier layer of sulphate, the separation being precise. When only a small quantity of pyridine is present, or to remove the last traces, the liquid is agitated with freshly-fused zinc chloride, filtered and rectified. It boils at 74° to 77° C., and therefore appears to be simple. It is decomposed by boiling alkali into ammonia and acetic acid, but contains only 23·9 to 24·1 per cent. of nitrogen, while acetonitrile, which the above reaction indicates, contains 34·1 per cent. of nitrogen. It must, therefore, notwithstanding its constant boiling-point, be a mixture. In coal-tar, acetonitrile is always associated with ethyl alcohol, from which it cannot be separated by fractional distillation. From the work of Vincent and Delachanel (Bull. Soc. Chem. 1880, 33, 405), the mixture must consist of 70 per cent. of acetonitrile and 30 per cent. of alcohol. Both constituents were further

chemically identified. In the light boiling fraction, benzene was found. Other alcohols, acetone, and ammonium salts found by Strombeck could not be detected.

For the valuation of liquefied ammonia, it is usually sufficient to determine the residue after volatilisation, without attending to its composition. For this purpose the bottle is placed horizontally, and an open steel tube screwed on the valve. By opening the valve, 20 to 50 grms. are allowed to flow into an Erlenmeyer flask. The previously-tared flask is quickly weighed with the ammonia and the stopper. The stopper has two perforations for a potash tube and a glass tube bent at right angles, the latter being closed and the former open, so that the volatilised ammonia must pass through the potash tube. At the ordinary temperature, two to three hours suffice for the dissipation of the ammonia; occasional shaking assists the operation. The residue, which contains more or less ammonia, according to its composition, is heated not above 40° C., and then dry air is passed in by the glass tube. Both tubes are then closed, and the apparatus is again weighed, giving data for the residue.

By testing a number of samples from the same bottle under various conditions, it was shown that the contents were homogeneous, with no tendency to settle out. Samples, each of 33 grms., drawn from one and the same bottle, in $\frac{1}{4}$, in 1, and in five minutes, gave 0·9, 0·9, and 1·0 per cent. residue. The whole contents of the bottle, 19·5 kilos., were allowed to evaporate in 36 hours, and a residue was obtained of 185 c.c., with a sp. gr. 0·87, corresponding to 0·84 per cent. Under the inverted condenser this lost only 3 per cent. of NH_3, which does not alter the result. Experiments were also made to show that the volatilised ammonia does not carry off any of the impurities. Linde's Ice Machine Co. were perhaps the first to attempt a volumetric determination. In this method, however, no account is taken of the difference in specific gravity of the ammonia and the residue. The authors independently arranged a similar apparatus, taking account of specific gravity. They determined that liquid ammonia, at the usual temperature of − 38° C., has a specific gravity of about 0·68. That of the residue, varies with its composition, but 0·9 is a sufficiently exact average.

The essential part of the apparatus is a glass tube of 30 to 40 mm. internal diameter, at the lower end of which a narrow glass tube is fused, of 5 mm. bore, and holding 1·1 c.c. The whole tube holds about 100 c.c., with a mark at 49 c.c. representing 33·3 grms. of ammonia. The lower narrow portion is divided into 15 parts, so that each division represents 0·2 per cent. When filled to the mark with ammonia, a grooved cork is inserted, and the contents are allowed to evaporate spontaneously. The operation is complete in 3 hours, or if hastened by placing the lower tube in water, in $\frac{1}{2}$ hour. The process is at fault if the ammonia contain much water, as then the residue may contain 25 per cent. of ammonia; otherwise the error from dissolved ammonia is so slight as to be negligeable.

The authors state that small quantities of gas are dissolved in liquid ammonia, which escape with that gas on evaporation, and are not found dissolved in the residue. They are engaged in the investigation of these gases.

—L. J. de W.

Sodium Bicarbonate, Analysis of. G. Lunge. Zeits. f. angew. Chem. 1897, 169—171.

Prof. Lunge gives the details of a process devised by K. J. Sundstrom for the rapid analysis of sodium bicarbonate, which has been in use for some years past at the ammonia-soda works at Trenton (Michigan, U.S.A.). It has the advantage over Lunge's method in not requiring semi-normal ammonia, and although not so accurate as the gas volumetric method, is sufficiently so for all practical and commercial purposes. It is based upon the fact that a sodium bicarbonate solution, when treated with caustic soda lye, decomposes according to the following equation—

$$NaHCO_3 + NaOH = Na_2CO_3 + H_2O.$$

When the whole of the bicarbonate has been converted into normal carbonate, the addition of a single drop of caustic soda lye has the effect of causing the mixture to give a brown coloration with silver nitrate solution; and this point is determined by spotting on a porcelain plate. The total alkali is determined by titration with N-acid, and the two estimations give all the data necessary for the calculation. The caustic soda lye is prepared by making up a solution of 20° B. from commercially pure caustic soda, precipitating with barium chloride, saturating with barium hydrate, and diluting to N-strength. The titration value is determined with N-hydrochloric acid in the cold, using phenolphthalein as an indicator. In a given sample, two lots of 4·2 grms. were weighed off, the total alkali in one being determined with N-hydrochloric acid. The other portion was placed in a 250 c.c. porcelain dish (Lunge prefers to use a glass beaker), and covered without stirring with 100 c.c. of water at a temperature of 15°—20° C. Approximately the amount of the N-caustic soda solution was then run in. In this case, knowing that the bicarbonate was at least 96 per cent., 48 c.c. were used, and the mixture was stirred until the whole was dissolved, which required about one minute. The addition of the N-NaOH was then proceeded with, adding first 0·5 c.c., subsequently 0·2 c.c., and finally drop by drop. After each addition, a drop of the solution is spotted with silver nitrate solution (20—25 per cent.) on a porcelain plate, until a brown coloration is formed at once. The percentage of bicarbonate is obtained by multiplying the N-NaOH used by 2; that of the normal carbonate by subtracting the N-alkali from the N-acid, and multiplying the difference by 53/42nds.

Thus, 4·2 grms. of bicarbonate require—

	C.c.
N-acid	49·9
N-alkali	49·7
	0·2

Therefore—

$NaHCO_3$ = 2·0 × 49·7	=	99·4
Na_2CO_3 = 0·2 × 53/42nds	=	0·25
NaCl	=	0·0
Difference	=	0·35
		100·0

An attempt to use ordinary N-NaOH in place of that recommended by Sundstrom was, according to Lunge, not attended with good results, and, moreover, since the solution contains caustic baryta, a turbidity in it at once demonstrates the presence of carbonate. It is therefore advisable in all cases to allow the liquor to settle, to estimate its value with N-acid, and any indicator, before each series of determinations, and to use it from a burette which can be filled from the lower end, the upper end communicating with the stock bottle, which is provided with a soda-lime filter for the entering air.—T. A. L.

Thoria, Behaviour of, with Oxalic Acid and Ammonium Oxalate, and the Estimation of. C. Glaser. Zeits. Anal. Chem. **36**, 213—219.

In a previous paper upon the analysis of monazite sand (Chem. Zeit. 1896, **63**, 612; this Journal, 1896, 675—677), the author stated that he had found that when thoria had been kept in solution as oxalate by ammonium acetate and afterwards was re-precipitated by ammonia and redissolved in mineral acid, it was then no longer possible to effect its complete precipitation as thorium oxalate by saturating the nearly neutralised and boiling solution with ammonium oxalate, and allowing the liquid to cool. He has since investigated this behaviour, and he finds that the reason of the incomplete precipitation is because, in nearly neutral solutions, thorium oxalate is somewhat soluble in ammonium oxalate—a fact which was not known at the time of his former paper, but which has since been established by other workers. The facts that he has elucidated are as follows:—From a boiling nearly neutral mineral acid solution, thoria is not precipitated at all by ammonium oxalate, even when no excess of the latter is present; thorium oxalate is only precipitated on allowing the solution to cool, when it settles down in a crystalline form. If, however, a *large* excess of ammonium oxalate be present it may be entirely prevented from precipitating even in the cold. Ammonium acetate

merely increases the soluble action of the liquid, but, contrary to what the author had surmised, it has no other influence.

Thoria is quantitatively precipitated as oxalate by oxalic acid, or ammonium oxalate even in excess, and in the presence of ammonium acetate, by rendering the solution strongly acid with hydrochloric acid.

The composition of the oxalate formed varies according to the mode of precipitation. If the thoria be dissolved in hydrochloric acid and the solution boiled before oxalic acid is added, a precipitate of thorium oxide and oxalate forms on adding the oxalic acid; sometimes also the oxide is partially precipitated before the oxalic acid is added, and remains insoluble on addition of more hydrochloric acid. On the other hand, if the oxalic acid be added before the solution is boiled, the normal oxalate precipitates on cooling, having the composition $Th(C_2O_4)_2 + 2H_2O$ (dried at 100° C.).

The oxalate, which is precipitated by ammonium oxalate (not in excess) from nearly neutral solutions by boiling and then allowing to cool, is also not of normal composition. Neither is the oxalate precipitated by ammonium oxalate in the presence of excess of hydrochloric acid. In the latter case it was found that the oxalate formed corresponded to the formula $ThH_2(C_2H_4)_3 + 2H_2O$ (dried at 100° C.).

The fact of the solubility of thorium oxalate in ammonium oxalate, raises the question whether in the scheme of analysis of monazite sand given in the author's earlier paper (*loc. cit.*), some of the thoria would be held in solution by the excess of ammonium oxalate used and so be included among the zirconia earths forming soluble oxalates. It is evident that in separating thoria from zirconia, more ammonium oxalate should be avoided than what is necessary to keep the zirconium oxalate in solution, and this can easily be done when only these two earths are present in the solution. When cerium is also present, it is not so easy to avoid an excess of ammonium oxalate, owing to the formation of the insoluble cerium oxalate.

The action of a large excess of ammonium oxalate on thoria in the presence of ceria was therefore next investigated, and it was found that, even in the presence of a very large excess of ammonium oxalate (about 5 grms.), about half the thoria was precipitated as oxalate along with the cerium oxalate, the precipitation being effected by adding the ammonium oxalate to a boiling, nearly neutralised solution of the earths in sulphuric acid, and then allowing the liquid to cool. The remainder of the thorium was kept in solution by the excess of ammonium oxalate used, and was precipitated from the filtrate from the oxalate precipitates, by the addition of hydrochloric acid. About one-half the thorium was precipitated with the cerium, both when relatively much or little cerium was present.

It was further found that in separating the oxalates of thorium and cerium by ammonium acetate, it was important to avoid a large excess of the acetate, as it was found that cerium oxalate is slightly soluble in this reagent. If an excess of ammonium oxalate be used, it only requires the addition of a few c.c. of ammonium acetate solution to dissolve out the whole of the thorium from the cerium oxalate.

In order to see whether thorium was kept in solution by the excess of ammonium oxalate and included therefore among the zirconia earths when making an analysis of monazite sand by the author's published scheme, a sample of monazite sand was analysed by this method, but after separating the cerium earths and the thoria as insoluble oxalates, the filtrate, containing the zirconia earths, &c., was treated with excess of ammonia, the precipitated bases filtered off, and redissolved in an excess of hydrochloric acid, oxalic acid added, and the liquid allowed to stand for some hours in a warm place. No precipitate of thoria was formed. The zirconia finally separated from the sample, was likewise examined in the same way, and found to be free from thorium.

The fact that thoria, although known to be soluble in excess of ammonium oxalate, had been completely separated, together with the cerium earths, causes the author to suggest the possible presence of some earth among the cerium earths which by a catalytic action brings about the complete precipitation of the thoria. Indeed, cerium itself has this effect in some degree. But as the conditions are not yet understood, in order to insure the determination of all the thoria, he recommends the treatment of the filtrate from the precipitates of thorium and cerium oxalate, and the subsequent examination of the zirconia, as above described.

If cerium has not to be determined, then the solution of the earths in sulphuric acid, after nearly neutralising, may be at once treated with excess of hydrochloric acid and oxalic acid, which will precipitate all the thoria and most of the ceria as oxalates, the zirconia and other earths passing into solution. The thoria and ceria can then be separated by ammonium acetate as usual. He found, however, that the thoria precipitated in this way was impure in colour, and he does not recommend this modification.

The following corrections and amplifications should be made in the table of the characteristic reactions of the rare earths given in his former communication:—

1. The statement that thoria and cerium protoxide are precipitated by ammonium carbonate in the same way as by ammonia is incorrect. In Graham-Otto it is said "thoria and cerium protoxide are easily soluble in an excess of ammonium carbonate, as are also the majority of the earths mentioned, and are precipitated from the solution on boiling."

2. The behaviour of thoria with ammonium oxalate should be stated: precipitates in the cold, soluble on heating, crystalline on cooling, provided a large excess of the reagent be avoided. Excess of hydrochloric acid precipitates the oxalate completely on cooling.

3. The statement that thorium oxalate, which is held in solution by ammonium acetate and ammonium oxalate, is in part precipitated by hydrochloric acid, must be enlarged by the addition that a sufficiently large excess of hydrochloric acid causes a quantitative precipitation.

4. It must be noted that zirconium oxalate is soluble in hydrochloric acid.

5. Cerium oxalate is slightly soluble in ammonium acetate.—H. S. P.

Silicon, A Rapid Method for the Determination of, in Silico-Spiegel and Ferro-Silicon. C. B. Murray and G. P. Maury. J. Amer. Chem. Soc. 1897, **19**, 138—139.

The authors describe the following method for the determination of silicon in a rapid and accurate manner:—0·5 grm. of the very finely divided sample is placed in a porcelain or platinum dish; 50 c.c. of water, 10 c.c. of hydrochloric acid (sp. gr. 1·20), and 12 c.c. of sulphuric acid (1 part of sulphuric acid (1·84 sp. gr.) to 3 parts of water) are poured on it, and the contents of the dish heated until copious fumes of sulphuric acid are given off. When cool, 10 c.c. of hydrochloric acid are added, and the whole is heated to soften the sulphate of iron; finally it is treated with 75 c.c. of water, and raised to boiling. The heating is discontinued, and note taken as to whether there is any effervescence when boiling ceases. Should this occur, the liquid must be evaporated until copious fumes of sulphuric acid are again given off, and then it is treated in the manner above described. The solution is filtered, washed with hydrochloric acid and hot water, ignited in a platinum crucible, and weighed. A few drops of sulphuric acid and enough hydrofluoric acid are added to dissolve the silica. The liquid is then evaporated to dryness, heated to decompose the sulphates, cooled, and weighed. The difference in the two weights represents silica. The whole operation can be accomplished in 30 minutes.—J. L. B.

Pig Iron, Determination of Sulphur in. A. A. Blair. J. Amer. Chem. Soc. 1897, **19**, 114—115.

Attention has frequently been drawn to the fact that the residue left from pig iron after treatment with dilute hydrochloric acid, contains sulphur. The author is able to convert all volatile sulphur compounds into hydrogen sulphide by passing the evolved gases through a tube filled with pumice, heated to redness. This method is not, however, of general application. It is pointed out that sulphur is liberated from pig irons under the following conditions:—1. As hydrogen sulphide, by solution of iron in hydrochloric acid. 2. As other compounds, not absorbed by alkaline lead salts, or oxidised by bromine or potassium

permanganate. 3. In some form upon which boiling hydrochloric acid has no action, but which is oxidised by nitric acid or aqua regia. 4. In some form not attacked by nitric acid, hydrochloric acid, or aqua regia. When sulphur exists in the last-mentioned state, it may conveniently be estimated by Bamber's method (this Journal, 1894, 665).—J. L. B.

Nickel, Estimation of, in Nickel Steels. J. Spüller. Chem. Zeit. 1897, **21**, 243—244.

The following colorimetric method is stated by the author to give satisfactory and rapid results. 2 grms. of the sample to be examined, and 2 grms. of a normal nickel steel containing a known amount of nickel, are weighed out into two 250-c.c. flasks, and about 60 c.c. of nitric acid (sp. gr. 1·2) added to each. The contents of the flasks are boiled until solution is complete, and nitrous fumes are no longer given off. When cold, the iron is precipitated with zinc oxide, and the green colours of the resulting filtrates are compared, a white homogeneous paper being used for a background. Since nickel steels generally contain from 1 to 7 per cent. of nickel, a fairly reliable result can be quickly arrived at, if the standard has about the same percentage of nickel as the sample. The author employs three standard nickel steels containing 1, 3, and 5 per cent. of nickel.

More accurate results may be obtained by comparing the colours in a calibrated vessel and adding a known volume of water to the test until the colour is identical with the standard. Steels containing under 1 per cent. of nickel cannot be submitted to this method, on account of the faint colour of the solution.—J. L. B.

Antimony, Estimation of, as Tetroxide. H. Baubigny. Comptes Rend. 1897, **124**, 499—502. (See also page 475, col. 1.)

The estimation of antimony as tetroxide was first proposed by Bunsen in 1858, but subsequent investigations by Bunsen himself and by Brunck and Read have thrown some doubt on the stability of Sb_2O_4 at high temperatures. Starting with the pentoxide Sb_2O_5, the author has ignited it in crucibles, from which the gases from the flame were carefully excluded at temperatures gradually increasing up to 800°, at which temperature its weight became constant. The analysis of the substance thus produced was found to correspond exactly to the composition required by the tetroxide Sb_2O_4.—J. S.

Basic Slag, Chemical Composition and Agricultural Value of. G. Paturel. Bull. Soc. Chim. 1897, **17**, 319—321.

See under XV., *page* 450.

Phosphates in Bone Meal, Citrate Solubility of. A. Keller. Chem. Zeit. 1897, **21**, 323.

See under XV., *page* 451.

ORGANIC CHEMISTRY.—QUALITATIVE.

Schiff's Reaction applied to Acid Magenta. L. Lefèvre. Bull. Soc. Ind. Mulhouse, 1897, 84—86.

According to Cazeneuve (Bull. Soc. Chim. 1896, 723; *cf.* this Journal, 1896, 560), Acid Magenta does not give Schiff's reaction. This, as the author points out, is erroneous, and he refers to Schiff's original paper (Comptes Rend. 1865, 45), pointing out at the same time that the difference observed by Cazeneuve is due to a time effect. The particular aldehyde employed also influences the test. Thus a solution of Acid Magenta (1 : 1,000) acidified with a drop of sulphuric acid and decolorised with bisulphite regains its colour when treated with a few drops of a 40 per cent. formaldehyde solution, whilst a much larger quantity of acetaldehyde is required to effect the same result. In all cases, however, the colour returns much more rapidly with Magenta than with Acid Magenta. Urbain (Bull. Soc. Chim. 1896, 455; this Journal, 1896, 560) states that condensation products are formed by the action of aldehydes on rosanilines, and Cazeneuve suggests (*loc. cit.*) that if this be the case the condensation takes place in the ring in Magenta at the position occupied by the sulphonic acid group in Acid Magenta, since the latter does not react with aldehydes. In Schiff's original memoir, however, it is pointed out that the regeneration of the colour of the decolorised Magenta solution is due to the formation of a new compound. The following is the quotation referred to:—On shaking a dilute sulphurous solution of the sulphate, or of any other salt of Rosaniline, with a few drops of aldehyde, sulphurous acid is given off; the solution turns red, then violet, and a precipitate is gradually formed consisting of small violet scales with a bronze-like lustre.—T. A. L.

Linseed Oil, Differences between Boiled and Raw. G. Morpurgo. Giorn. di Farm. di Trieste, 1896, **1**, 361; Chem. Zeit. **21**, 37.

The presence of lead, zinc, or manganese in linseed oil shows that the oil has been boiled. The absence of these metals does not, however, prove the oil to be raw oil, as some so-called boiled oils (prepared by an aëration process) are quite free from metallic oxides. The author has found the following method a reliable one for distinguishing boiled from raw linseed oil. The oil is saponified, the soap dissolved in water, and the resulting clear solution treated with common salt until no more soap separates out. If, after filtration, the filtrate be made strongly acid with acetic acid, a turbidity shows that the oil used was boiled, whilst the filtrate from a raw oil remains clear.—W. P. S.

Alkaloids, Action of Tannin on Some. O. de Coninck. Comptes Rend. 1897, **124**, 506—508.

The author describes some experiments on the action of tannin on alkaloids, and has arrived at the following method of distinguishing pyridine from piperidine. On adding a few drops of tannin solution (1·434 grm. in 20 c.c.) to solutions of a few drops of the two bases in 150 c.c. of water, a white amorphous precipitate is formed in the case of pyridine, whilst with piperidine a clear yellow liquid is obtained, which becomes gradually darker, and finally reddish-brown.—J. S.

Margarin, The Question of Colouring. R. Henriques. Chem. Rev. Fett u. Harz Ind. **4**, [5], 68—70.

See under XVIII. A., *page* 456.

Margarin, Addition of Dimethylamido-azobenzene to. A. Partheil. Chem. Zeit. 1897, **21**, [27], 255.

See under XVIII. A., *page* 457.

ORGANIC CHEMISTRY.—QUANTITATIVE.

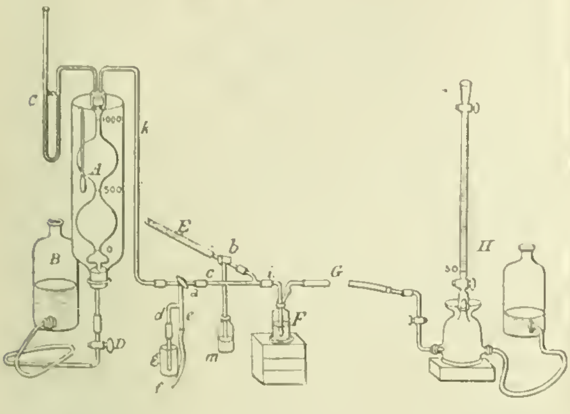
Free Nitrogen in Purified Coal-Gas, Determination of the. G. Arth. Bull. Soc. Chim. 1897, **17**, 427—432.

The determination of nitrogen in coal-gas by measurement of the residue after an eudiometric combustion is inaccurate, for the operation is only carried out with a small volume of gas, generally poor in free nitrogen, and the volume of the residue is influenced by the different impurities introduced in the previous operations.

In the purified gas, cyanides are present in such small quantity that it is generally impossible to show their presence in several litres; basic nitrogen compounds can be removed by sulphuric acid; thus Dumas' method may be employed.

In the figure, A is the measuring vessel, each of the bulbs containing 500 c.c.; the cork in the upper end carries the water manometer C and the tube *k*. To avoid the error due to measuring over water, when not in use, A is kept partly full of gas under a slight pressure. *a* is a three-way cock, the lower tube of which has two branches *d* and *e*. F is a small wash bottle containing strong sulphuric acid. G is a combustion tube, placed in a furnace, containing a layer of copper oxide 55 cm. long, and a coil of bright copper 15 cm. long. H is a nitrogen-measuring apparatus, such as is used in organic combustions. The end of G near this apparatus should be bent,

as shown, to prevent fracture of the tube by the water formed in the combustion. E is a tube, 25 cm. long, containing manganese carbonate dried at 100° in an



atmosphere of carbon dioxide; this tube is also placed in a furnace; it communicates with the three-way tap b, the lower limb of which dips into mercury. From a to i the tubes have a bore of about 2 mm.; c and i should be as near together as possible.

The copper oxide end of G is heated, the copper being withdrawn, if the tube has been used before oxygen is sent through; c is connected with the gas supply, the tube a i filled with gas, and the clip c closed. (F and G are not yet connected.) When G is sufficiently hot, E is heated, and, after carbon dioxide has bubbled through F for a short time, G is connected. In about 10 minutes the copper coil is introduced, the gas lighted under it, and H connected up.

The measuring vessel is now filled; the gas enters at f, is allowed to fill the upper bulb, the clip on f is closed, and B lowered until the air from the manometer tube enters A. B is raised and lowered several times to effect complete replacement of the air in the manometer; the gas is then expelled through d, and the whole process repeated. The whole of A is now filled with gas, and the water standing at the zero mark, D is closed, the pressure and the temperature read off. The air being driven out of G, the current of gas from E is diminished and allowed to pass out through m. The gas is then sent slowly through the combustion tube at a rate of about three bubbles per second (75 minutes will be required for 1 litre); the level of B is kept adjusted so that there is always a pressure of 4—5 cm. on the gas. If sufficient nitrogen collects in the burette, only 500 c.c. may be burnt, but if the volume be small, the whole litre should be used. When the liquid in A reaches the mark, D is closed, the gas allowed to flow until the previous pressure is shown on the manometer, then c is sharply closed, and the carbon dioxide allowed to sweep the gas out of F and G. The nitrogen is now measured in the ordinary way. If the barometer and thermometer have varied during the experiment, the volume of gas burnt must be corrected accordingly.

—A. C. W.

Paraffin, Estimation of, in Distillates of High Boiling Point from Brown Coal Tar and Crude Petroleum. D. Holde. Zeits. angew. Chem. 1897, 116—117.

Following the suggestion of Höland to apply his method (this Journal, 1894, 286), to petroleum products, the author found, as Aisinman's experiments showed, that the lubricating oil distillate from crude petroleum was with difficulty soluble in alcohol. 10 to 20 grms. of various Russian oils required 1 to 1·5 litres of alcohol for solution, besides the quantity for washing and reprecipitation. In addition to oil, this large amount of alcohol dissolved considerable quantities of paraffin, 1 litre of absolute alcohol at 0° C. dissolving 0·3 grm. The separated paraffin was with difficulty freed from particles of oil, and was therefore generally in a greasy condition.

The methods of Pawlewski and Filemonowicz and of Zaloziecki (this Journal, *loc. cit.*) were also found to be costly, troublesome, and inexact.

The best results were obtained by working out a proposal of Grotowsky, modified by Engler and Böhm (Dingl. 1886, **262**, 473; this Journal, 1887, 371—372), in which an ethereal solution of the oils is precipitated by alcohol in the cold, although, according to Holand, considerable quantities of paraffin remain in solution even at several degrees below 0° C., and the repeated crystallisations recommended by Engler and Böhm must waste much time. The author avoids these faults by using the smallest possible quantity of fluid for solution and precipitation at a very low temperature (− 18° to −20° C.), and by working as quickly as possible. The precipitated paraffin is washed at − 18° to − 20° on a double filter with the smallest possible quantity of a mixture of ether and alcohol. Analyses in duplicate of a number of typical lubricating oil distillates of various fractions gave results differing by only a few tenths per cent., and tests made on artificial mixtures of oil and paraffin gave only slight deviations from the known paraffin content. The operation requires only from 1 to 2 hours. Further details are to be found in the Mitt. aus den Kgl. Techn. Versuchsanstalten, 1886, Heft 4, 473.—L. J. de W.

Raw Fibres [Vegetable], A New Method for the Quantitative Estimation of. Lebbin. Arch. Hyg. 1897, **28**, 212.

The method depends upon the use of ammoniacal hydrogen peroxide, by means of which starch and most of the vegetable albuminoid substances, especially the characteristic glutins, are dissolved, whilst pure cellulose is barely attacked. The following mode of procedure, which may be modified according to circumstances, is given:—3 to 5 grms. of the meal or bran, powdered so as to entirely pass through a sieve of 0·2 mm. mesh, are well stirred into 100 c.c. of water, contained in a roomy beaker, so that no lumps remain, and the whole is boiled for half an hour. 50 c.c. of 20 per cent. hydrogen peroxide are then added, and the boiling continued for 20 minutes with gradual addition of 15 c.c. of a 5 per cent. ammonia solution. The boiling is continued for a further 20 minutes after the addition of the ammonia is completed, and the whole is then filtered hot through a weighed filter, washed with boiling water, dried, and weighed.

From the residue, the percentage of ash and, in substances very rich in nitrogen, the nitrogen percentage × 6·25, is to be subtracted. The method gives fairly concordant results, and possesses the advantage that the solutions obtained, filter readily.—J. T. C.

Linseed-Cake Fat, On the Iodine Number of. B. A. van Ketel and A. C. Antusch. Zeits. angew. Chem. 1897, 210.

In answer to the criticisms of Mastbaum (this Journal, 1897, 150), the authors remark that the various linseed oils extracted by them from the seed gave iodine numbers 181 to 187, or an average of 185; thus not differing greatly from 173 to 183, obtained by Mastbaum himself. They admit the possibility that, by warm or cold pressing and extraction, linseed oils may be obtained showing different iodine numbers, but, to their knowledge, this has never been actually tested; and the differences in Mastbaum's numbers for one and the same olive oil obtained at varying temperature and pressure, are insignificant. It is well known that the iodine numbers of linseed oils exposed to the influence of light and air decrease, but until it has been proved that, in well-kept cakes piled up one upon another, and so protected from light, an alteration of the oil, and therefore of the iodine absorption, occurs, Hübl's proposition must be accepted—that the age of the fat is without sensible influence on the iodine value. The iodine numbers, 166, 167, and 168, found by the authors for oils of extracted cakes, cannot, in their opinion, be fully explained by alterations which have possibly occurred in the oils in the cakes, and they therefore think that a determination of the iodine number of linseed-cake oils is always of importance as a test of purity.—L. J. de W.

Beetroot-Sugar Analysis. Resolutions of the Austro-Hungarian Chemists engaged in the Sugar Industry at the Meeting at Buda-Pesth, June 22, 1896. Zeits. angew. Chem. 1897, 123—126.

Polarisation.—For polarising substances containing sugar, only the half-shadow instrument is to be used. It is desirable to use the 400-mm. apparatus with contracted scale. During the observation, the polariscope must be in a fixed position, so far from the source of light that the polarising nicol does not become heated. As source of light, a lamp with an intense flame is recommended; a triplex gas jet with metal chimney, lens and reflector; gas with the Auer burner, electric lamps, duplex petroleum lamps, &c. The chemist must satisfy himself as to the accuracy of the instrument by correct quartz plates, and of the invariability of the light before and after the observation; also of the accuracy of the weights, the polarisation flasks, the observation tubes, and the cover glasses. Scratched cover glasses must not be used. Results are to be expressed to the half of one-tenth per cent.

Analysis of Raw Sugar.—On opening the sample, the upper layer is to be removed with a spatula or a spoon, and discarded. The remainder is to be completely emptied into a porcelain dish and mixed. Any lumps may be easily crushed with a spoon. The sample is then replaced and the weighings made forthwith. For the *polarisation* of first products, at least the normal weight should be weighed out, dissolved in distilled water without the aid of heat, and, after clarifying, made up to 100 c.c. at the ordinary temperature (not too far from 17°·5 C.). Of after-products it is advisable to take a multiple of the normal weight and make up to a corresponding volume. The flask must be standardised with water at 17°·5 C. For clarifying and decolorising, basic acetate of lead may be used (3 parts of lead acetate, 1 of litharge, 10 of water), concentrated solution of alum, and Scheibler's colloidal alumina. Char and decolorising powder are absolutely excluded. For sugars polarising over 99, alum or colloidal alumina alone will suffice; for first products, 1 c.c. of acetate of lead and 1 c.c. of alum solution, and for after products, 2 to 3 c.c. of acetate of lead, 1 to 2 c.c. of alum solution for each 100 c.c. After making up to the mark and wiping out the neck of the flask with filter paper, the clarified sugar solution is well shaken, and the entire contents poured on to a dry quick-filtering filter. The first portion running through is poured away, and the rest, which should be perfectly clear, is used for polarising.

For determining *water*, only water-baths are to be used, with or without vacuum pump. Air baths are absolutely excluded. 10 to 15 grms. are dried for two hours. Sugars containing more than 3 per cent. of water are dried for a double period of time. After cooling in a desiccator, loss is taken as water.

Ash is determined by incinerating at least 3 grms., according to Scheibler's method, with pure sulphuric acid in a platinum dish in a muffle at as low a temperature as possible, a dark red heat. The ash should not be fused, and if, on being moistened with water, it shows sand or other mechanical impurities, a fresh test should be made. 10 per cent. is deducted from the ash before entering the result on the certificate.

Litmus is to be used for determining the *alkalinity*. It is to be stated in the certificate whether the sugar is alkaline, acid, or neutral.

Invert sugar is tested for qualitatively by dissolving 10 grms. of sugar in 25 c.c. of water, adding 100 c.c. of Soldaini's solution, and boiling for five minutes, reckoned from the commencement of the boiling. In the quantitative estimation, the method of Herzfeld (Z. Zuck. 1886, 6) is followed. If less than 50 mgrms. of copper (corresponding to 0·05 per cent. of invert sugar) are obtained, 0·0 per cent. is to be entered on the certificate. An explanation of the reason of this procedure is to be endorsed on the certificate. If more than 0·05 per cent. invert sugar is found, the amount is to be expressed to two decimal places, the second figure being corrected by the third.

Molasses and Osmose Water. — At least 50 c.c. of molasses or osmose water should be freed from air by warming, and the mechanical impurities removed before the density is determined by the pycnometer. The heating must be so carried out that the density is not affected. The density is to be expressed to five decimal places, and both old and new Beaumé degrees are also to be added. For the direct polarisation the dilution is to be so chosen that 100 c.c. of the clarified solution represents the half normal weight. Clarification is to be made with 10 c.c. of acetate of lead to the half normal weight, and no correction for the lead precipitate. The reaction to litmus is to be stated, and no complaints will be entertained unless the test is made immediately on receipt of the sample. For determining the ash, 10 to 20 grms. are carbonised, extracted, and evaporated, &c. unless otherwise desired. Invert sugar is to be determined by Herzfeld's method. When more than 1 per cent. of invert sugar is present a correspondingly smaller amount is used for the analysis. For calculating the results the tables of Meissl and von Hiller are to be used (Zeits. angew. Chem. 1890, 191 and 446). If less than 0·05 per cent. be present it is to be entered as absent.

Analysis of Molasses by Inversion.—In the determination of sugar by the Clerget method, clarification with lead acetate is not usual, but in the case of abnormal molasses and osmose waters where char does not suffice, clarification is effected by basic nitrate of lead. In this case Herles's constants are to be used in the formula for calculation, but the direct polarisation is to be obtained by clarification with basic acetate of lead. Of normal molasses and osmose waters the half normal weight is inverted in a 100 c.c. flask, according to the prescription of Herzfeld; after filling to the mark it is shaken up, and, if necessary, clarified by extracted char. The result is calculated by the formula $\frac{100\,S}{132{\cdot}7}$ for the temperature of 20° C.; for other temperatures correction is made either according to the formula of Tuchschmidt $\frac{100\,S}{142{\cdot}7 - \frac{1}{2}t}$ or by the formula—

$$I20 = It + 0{\cdot}0038\,S\,(20 - t),$$

where S = P + I, *i.e.*, the sum of the readings before and after inversion without regard to sign, and t the temperature of observation of the inverted solution in centigrade degrees.

Raffinose is estimated by inverting as above and using the formula $Z = \frac{0{\cdot}5124\,P - I}{0{\cdot}8390}$ and $R = \frac{P - Z}{1{\cdot}852}$. In products containing more than 2 per cent. of invert sugar the determination of raffinose by the inversion method is to be abandoned. Abnormal products may be clarified by basic nitrate of lead under the conditions given above.

The analysis of char and manures is touched upon, and directions given with regard to samples. The resolutions were to come into force on Sept. 1, 1896.—L. J. de W.

Sugar Solutions, Nitrate of Lead; The Use of, for Decolorising various. H. Pellet. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1897, **14**, 794—796. (This Journal, 1897, 67.)

The substitution of nitrate of lead for basic acetate of lead, proposed by Herles, for decolorising the various sugar products to be polarised, is criticised by the author, who has not tried this reagent on beetroot molasses. He agrees with Weisberg that the advantage of having a liquor which, on inversion, is less coloured than by acetate of lead, is very slight. A few decigrms. of a special char will produce a very great decolorisation without absorption of sugar. Moreover, the use of nitrate of lead necessitates the use of caustic soda, and the formula to be applied for the inversion requires to be changed; this is a complication. Finally, the precipitate is so voluminous that a correction must be made.

When a lower polarisation is found on defecating with the nitrate than with acetate, it is necessary, as Weisberg remarks, to show that it is the former which is exact. The author holds that both are inexact if the filtered liquid is alkaline, as is generally the case; indeed, it may be more alkaline with the nitrate than with the acetate; the true polarisation is that given by the liquid freed from lead and polarised acid. This result is attained directly by using sulphurous acid, which is a powerful decoloriser, and likewise prevents coloration during the inversion. The liquor

freed from lead by sulphurous acid, and being acid, from sulphurous acid and acetic acid, is in a favourable condition for inversion, a partial or complete saturation of the hydrochloric acid by excess of acetate of lead need not be feared, whereas a destruction of sugar may occur from the action of the mixture of hydrochloric acid and nitrate, heated to 68° or 70° C. The author finds no advantage in substituting nitrate of lead and caustic soda solution for the procedure he has indicated for the analysis of molasses in general, both beet and cane, with the special modifications given for the latter.

Nitrate of lead and caustic soda were tried on cane molasses without any advantage as regards decolorisation; besides, the use of reagents of alkaline reaction should now be absolutely proscribed for the analysis of products of the cane containing reducing bodies (dextrose and levulose). The neutral acetate of lead should alone be used, and this also neutralised with acetic acid. Decolorisation is sometimes slight, and a very intense source of light should then be used. The author has given details relative to the use of acetylene for this purpose.—L. J. de W.

Yeast, Quantitative Determination of, in Fermentation Experiments. R. Kusserow. Wochenschr. für Brauerei, 1897, **14**, 117—118.

The examination of a number of commercial yeasts shows —(1) that the amount of dry yeast substance increases with the specific gravity of the yeast; and (2) that the specific gravity of the dry yeast substance is approximately a constant (1·509), which can be calculated from either of the above values. In order to determine the amount of yeast formed in small laboratory experiments, all that is necessary is to siphon off the fermented liquor, wash the yeast repeatedly with distilled water, and then transfer it to a specific gravity bottle, which is finally filled with distilled water, and weighed. From the weight obtained, the specific gravity and, consequently, the amount of yeast can be calculated. This method may also be applied to other micro-organisms which subside when washed with water.—A. K. M.

Wine, Detection and Quantitative Determination of Cane Sugar in. P. Kulisch. Zeits. angew. Chem. 1897, 205—210.

The amount of acid prescribed by the official method for the estimation of cane sugar in wines (0·05 grm. HCl. to 50 c.c. of wine) suffices only for wines that have been diluted to one-fifth. Undiluted wines require about 1 c.c. of 25 per cent. hydrochloric acid, although under these conditions somewhat too much sugar may be found. An excess of 0·025 grm. per 100 c.c. is to be considered of no importance. For wines not diluted to one-fifth, but at least to half strength, 0·5 c.c. of HCl, sp. gr. 1·124, will suffice.

Inversion by means of oxalic acid is more convenient, 1 grm. being ample for wines diluted more than half; for less dilute wines, 2 grms. to 50 c.c. Oxalic acid has the advantage of not affecting the optical properties of the other sugars occurring in wines along with cane sugar.
—L. J. de W.

Alcohol and Water, Distillation of very dilute Mixtures of. Application to the determination of Alcoholic Solutions containing from 1 in 3,000 to 1 in 10,000. M. Nicloux and L. Bauduer. Bull. Soc. Chim. 1897, **17**, 424—427.

Solutions of alcohol of strengths between 0·2 and 0·03 per cent. may be determined by means of a 2 per cent. solution of potassium bichromate, the yellowish green colour due to excess of bichromate being easy to distinguish from the bluish-green solution otherwise obtained.

In the distillation of ordinary dilute alcohol, all the alcohol is contained in the first third of the distillate. The authors find that very dilute solutions (0·2 and 0·03 per cent.) also behave in a similar manner. In solutions of this strength they find that (1) the first twentieth of the distillate contains about 50 per cent. of the alcohol, (2) the more dilute the solution the greater is the percentage of the total alcohol contained in any fraction distilled. (3) the alcohol in the 0·2 and 0·1 per cent. solutions is all contained in the first third, the alcohol in the 0·03, 0·02, and 0·01 per cent. solutions in the first quarter of the distillate.—A. C. W.

Ethyl Alcohol and Acetic Ether, Volumetric determination of Mixtures of. B. Kuriloff. Ber. **30**, 741—743.

Alcohol is volumetrically determined by permanganate, or by oxidation with chromic acid. The latter method is preferable, because the products of the first process are unknown. The author, in employing the chromic acid method according to the directions of Bourcart, always found 1—1·5 per cent. of alcohol too little; after varying the duration of the reaction and the strength of acid and bichromate, he finds that the following are the necessary conditions:—25 c.c. of alcohol solution, containing about 0·1 grm. of alcohol, are placed in a thick walled flask of 200 c.c. capacity, and 25 or 50 c.c. of chromic acid solution added, the cork is tied down, and the flask heated on the boiling water-bath for six hours. The bichromate solution contained about 1 grm. in 100 c.c.; the sulphuric acid was diluted with five or six volumes of water. After cooling, the contents of the flask are washed out, potassium iodide added and the liberated iodine determined by thiosulphate. The acetic acid formed is not further oxidised—

$$2K_2Cr_2O_7 + 3C_2H_6O + 8H_2SO_4 = 2K_2Cr_2(SO_4)_4 + 3C_2H_4O_2 + 11H_2O.$$

In the oxidation of acetic ether by chromic acid, saponification first takes place and then the alcohol produced is oxidised as above. According to the author's results the oxidation method is to be preferred to saponification with baryta.

In the analysis of mixtures of alcohol, acetic acid and acetic ether, free acid was determined in one portion, ether in a second by saponification, and total alcohol in a third by oxidation.—A. C. W.

Coffee Beans, Determination of the Weight of the Layer of Caramel on, after Roasting with Sugar, and upon the Examination of Coffee in General. W. Fresenius and L. Grünhut. Zeits. Anal. Chem. **36**, 225—233.

The methods of König (Zeits. f. angew. Chem. 1888, 631), Neubauer, Stutzer (Zeits. f. angew. Chem. 1888, 701), and of Hilger, were compared upon four different kinds of coffee beans, each of which kinds was roasted without sugar to three different grades, *viz.*, until it had lost 15 per cent., 18 per cent., and 21 per cent. respectively, and each of which was also roasted with additions of $7\frac{1}{2}$ per cent. and 9 per cent. of sugar respectively.

Hilger's method, which is the one preferred, was carried out as follows:—10 grms. of whole beans were covered with 100 c.c. of dilute alcohol (equal volumes of 90 per cent. alcohol and of water), and allowed to stand for half an hour at the ordinary temperature, after which the alcohol was poured off, and another portion of 100 c.c. of the same strength, added, and the coffee allowed to stand for a second period of half an hour, when the liquid was again decanted. This was repeated a third time with a third portion of alcohol, and then the three decanted portions of spirit were united, made up to a volume of half a litre, an aliquot part of this taken, evaporated to dryness, dried at 100° C. and weighed, after which it was incinerated, and the residual ash weighed.

By operating in this way upon coffee, which had been roasted without sugar, the amount of soluble extract derivable from the pure coffee beans was determined, and the difference between this amount and the total amount of extract obtained from the coffee beans coated with caramel, gave the weight of the caramel due to roasting with sugar. The authors found by this method that the average percentage of extract from pure roasted coffee beans was 1·15 per cent., equal to 0·85 per cent. of ash-free extract. After correcting by these amounts, the extracts derived from the caramelised coffee, the latter yielded from about 2·1 per cent. to 4·4 per cent. of extract, equal to, from 2 per cent. to 4 per cent. of ash-free extract. The authors point out that before accepting these or any figures as true average values, many more determinations should be made. For tables and further details, the original should be referred to (see also this Journal, 1889, 822).—H. S. P.

Sugar in Chocolate and Foods, Determination of. H. Pellet. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1897, **14**, 790—792.

X. Roques in a note on the determination of sugar in chocolate (*loc. cit.* Dec. 1896) advises the preparation of a solution of 15 grms. of chocolate in about 90 c.c. of distilled water, heating to 40° C. and stirring. 15 c.c. of 10 per cent. basic lead acetate are added and the solution filtered. 70 c.c. of this liquid are taken, and sulphate of soda and acetic acid added to eliminate the lead. But, as Stammer demonstrated many years ago, liquids containing simultaneously lead and saccharine matters, retain in solution some of the sulphate of lead, the remainder of which has been precipitated by acetic acid. As chocolate often contains notable proportions of glucose, it seems preferable to use "neutral lead acetate," neutralised with acetic acid, as for cane products, and to precipitate excess of lead by sulphurous acid. Even large quantities of sulphurous acid do not affect the determination of reducing sugars by means of Fehling's solution. The author used 2 grms. of crystallised sodium sulphite with 5 c.c. of Fehling's solution, and found that it required the same amount of a glucose solution, with and without sulphite; this being more than 20 times the amount of sulphite that would be present. The only precaution to be taken is to add a little soda with the copper liquor to saturate the free acid.

To determine the total sugar and glucose, Roques proposes heating 50 c.c. of the solution for three hours on a water-bath; the acidity due to the acetic acid sufficing to produce inversion. The procedure appears dangerous to the author, as the acidity may be insufficient to transform all the crystallisable sugar, and the method of heating is uncertain. It is better to plunge the flask into a boiling water-bath, and to note the temperature of the liquid inside the flask. A quicker method is possible. If dextrin is suspected, the sugar may be inverted by heating the solution to boiling for one or two minutes after adding ½ per cent. of hydrochloric acid. The sugar alone is inverted, the action of the acid on the dextrin commencing only after a much longer time and with a greater acidity. This quantity of acid is quite sufficient to convert, on boiling, all the sugar present. It is advisable to use the neutralised "neutral acetate" of lead for all food stuffs containing glucose, for the basic acetate may not only precipitate reducing sugars, as levulose, but an excess may affect the polarisation.—L. J. de W.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Calcium Carbide from Calcium Tartrate, Production of. S. Zinno. Boll. Chim. farmac. 1896, **35**, 555; Chem. Zeit. Rep. 1897, **21**, [9], 67.

The author obtained, by heating crude calcium tartrate, which forms the sediment in wine casks, in a cast-iron retort to 500° C., a dry, hard, grey, spongy mass, which on contact with water developed acetylene.—A. S.

Cochineal [Oxidation of]. C. Liebermann and H. Voswinckel. Ber. **30**, 688—691.

By oxidising the colouring matter from cochineal or cochineal carmine in an alkaline solution with potassium persulphate and extracting with ether, a residue is obtained easily soluble in water. This consists of a mixture of acids, two of which (neither containing nitrogen) were separated by means of their lead salts and designated cochinillic acid and coccic acid. The former $C_{10}H_8O_7$ (?) is precipitated from methyl alcohol by chloroform in thin white needles melting at 225° C. The aqueous solution gives a red coloration with ferric chloride and with bromine water, a white precipitate melting at 220° C., having the composition of a tribromo-cresotic acid, $C_6Br_3(OH)CO_2H$. Coccic acid, $C_9H_8O_5$, melts at 293° C. and crystallises from dilute alcohol in needles. It gives with ferric chloride a similar coloration to cochinillic acid, but somewhat more violet, and may possibly be a cresol dicarboxylic acid.

—T. A. L.

The "Shinia" Leaves of Cyprus. Imp. Inst. J., May, 155.

A despatch has been recently received from the Chief Secretary of Cyprus enclosing translations of pamphlets by M. P. Gennadius, Director of Agriculture in that island, on the industry connected with the cultivation of the shinia and laurel shrubs. *Pistacia lentiscus* is a shrub that grows abundantly in most parts of Cyprus, and is called in the island "shinia"; a variety of this shinia is the mastic shrub of Schio, the leaves only of which have a commercial value, serving as a tanning and painting material. From the wood of the shrub, charcoal of good quality is made, and from its seed oil can be extracted, which is fit, not only for burning purposes, but, in case of necessity, for food as well. For some time shinia leaves were exported from Cyprus to England in small quantities by the Cyprus Company, but the principal market for shinia leaves is Palermo, in Sicily, to which port there is an annual exportation from Tunis of 10,000 tons; they are there used chiefly for the adulteration of sumach (*Rhus coriaria*), which is grown in large quantities in Sicily, and is thence exported to England and France. A good quality of shinia leaves is also consumed at Lyons as a dyestuff for silks.

Aluminium Nitride [Ammonia from Nitrogen of Air], Preparation and Properties of. L. Franck. Chem. Zeit. 1897, **21**, 263.

The author has previously shown in conjunction with A. Rossel (this Journal, 1896, 273) that when aluminium powder is heated with calcium carbide, the product possesses the property of combining with atmospheric nitrogen. Aluminium nitride (Al_2N_2) is now prepared by mixing powdered aluminium with a known proportion of calcium carbide and heating the mixture before a blow-pipe flame in a porcelain crucible. When the reaction is finished, a spongy greyish-white or yellow product remains in the crucible, which does not absorb nitrogen until it has been exposed to the air for some hours. The nitride smells faintly of ammonia. In boiling water, ammonia is slowly liberated, but in caustic alkalis, generated in large quantities. One of the products prepared in the above manner contained 15—20 per cent. of nitrogen. A preparation containing more nitrogen could not be obtained by this method nor by leading pure nitrogen over the mixture. A product which absorbed a considerable amount of nitrogen was obtained by heating aluminium powder with dry slaked lime.

The author is carrying out experiments on the value of aluminium nitride as a nitrogeneous manure. He thinks from his results that it can be used directly as such, and that the loss of ammonia is in any case very small.—J. L. B.

Gold, Action of Phosphorus on. A. Granger. Comptes Rend. 1897, **124**, 498—499.

By heating finely-divided gold at a temperature of about 400° in the vapour of phosphorus and cooling rapidly, the author has prepared a compound Au_3P_4, which decomposes when heated in contact with air, and even in a current of carbon dioxide, at the temperature at which it is formed.

—J. S.

Highly Purified Substances, Observations on the Properties of some. W. A. Shenstone. Proc. Chem. Soc. 1897, [178], 2—3.

1. The author has compared the behaviour of oxygen under the influence of the silent discharge of electricity when saturated with water vapour, and when carefully dried. The results show that, contrary to the statement of previous investigators, oxygen is most freely converted into ozone when wet, and that well-dried oxygen yields only a very minute percentage of ozone. The results obtained also show that the ozone in ozonised oxygen is far more stable in the presence of water vapour than in its absence; that is to say, the change by which ozone is converted into oxygen is very greatly retarded by the presence of moisture.

2. Chlorine prepared by the electrolysis of silver chloride, and also carefully purified bromine and iodine, have been dried by very thorough treatment with prepared phosphoric

oxide, and then presented to the action of mercury prepared for the purpose by several distinct methods and thoroughly dried. In every case the metal and the halogen interacted instantly and rapidly.

3. Highly purified chlorine, when submitted to the silent discharge of electricity, does not undergo condensation.

4. The abnormal expansion of chlorine which has been described by several observers appears to depend upon the presence of impurities in the chlorine.

Zinc Dust as an Oxidising Agent in Presence of Acetic Acid. [Preparation of Camphoric Anhydride.] O. Aschan. Ber. **30**, 657—658.

By the action of zinc dust and acetic acid on benzoïn, Sudborough noticed the formation of benzil, and the author quotes another instance in which zinc dust acts as an oxidiser. He observed that when camphorquinone—

$$C_8H_{14}\left\langle \begin{matrix} CO \\ | \\ CO \end{matrix} \right.$$

is dissolved in six times its weight of glacial acetic acid, and treated with zinc dust, heat is evolved, and the golden yellow solution rapidly decolorises. On evaporating the filtered solution to half its bulk and adding water, a copious crystalline precipitate is obtained, which, after two crystallisations from alcohol, melted at 220° C., and possessed all the characteristics of camphoric anhydride.—T. A. L.

Antimony Tetroxide, Decomposition of, at High Temperatures. H. Baubigny. Comptes Rend. 1897, **124**, 560—562. (See page 170, col. 1.)

Antimony pentoxide, when heated, is perfectly stable at temperatures not exceeding 357° C.; but from 440° C. upwards to a faint red heat, it is slowly reduced to antimony tetroxide (Sb_2O_4) with evolution of oxygen. The transformation takes place rapidly at 750°—800° C., at which temperature the tetroxide formed is stable. At higher temperatures, equal to or exceeding that at which silver fuses, the tetroxide, is, however, in its turn reduced to trioxide, and at the melting point of gold, this conversion is rapidly effected. Antimony tetroxide cannot, therefore, be regarded as a "fixed" body; at least in so far as concerns its behaviour when strongly heated.—H. T. P.

Starch, Fixation of Iodine by Rice and Wheaten. G. Rouvier. Comptes Rend. 1897, **124**, 565—566.

The author has previously shown (Comptes Rend. **114**, 1366; **117**, 461; and **118**, 743) that when starch (rice or wheaten) is suspended in water and treated with iodine, definite compounds are produced, either $(C_6H_{10}O_5)_{16}I_2$, or $(C_6H_{10}O_5)_{16}I_5$ being formed, according as the starch or the iodine is employed in large excess; but that between these extremes, the quantity of iodine absorbed increases regularly with the quantity of iodine employed. The numerical results before obtained, the author considers, admit of an interesting theoretical interpretation. On the assumption that, between the limits already mentioned, compounds with three and four atoms of iodine are successively formed, and that these bodies possess the property of dissolving a certain proportion of iodine, it is found that the iodine thus dissolved (by the starch iodide), bears a constant ratio to the iodine remaining in solution in the supernatant liquid, in accordance with the well-known law as to the distribution of a soluble substance between two immiscible solvents. It appears, therefore, that iodine, when not present in sufficient excess to produce the compound $(C_6H_{10}O_5)_{16}I_5$, is fixed by starch, partly in definite combination, partly in a dissolved state.
—H. T. P.

Iodine Spring, Analysis of Water from a recently discovered. A. Lipp. Ber. **30**, 309—312.

The water analysed, is that of a spring (Marienquelle) discovered in 1891 in the neighbourhood of Fossen, in the Bavarian Algäu.

The water, which is medicinally valuable, is clear and colourless. Its temperature, summer and winter, is from 7—8° C. and its density 1·00216. The water possesses a slight characteristic odour, due to dissolved hydrocarbons, a slightly salty taste, bitter to alkaline. Its reaction is slightly alkaline.

In sealed vessels the water remained unchanged after two years; exposed to the air, however, it turns yellow, owing to the separation of iodine. A continuous evolution of gas bubbles was noticed in the spring, the gas being methane.

In addition to the complete analysis, estimation was also made from time to time of the total solids; the variation in the amount of these was only slight, as the following figures show:—

			Grms.	
In October	1891	the residue per litre was	2·786	dried at 180°.
„ July	1893	„ „ „	2·880	
„ April	1894	„ „ „	2·652	

The metals found in the water were: in larger quantities, sodium, calcium, and magnesium; in smaller amount, iron, lithium, aluminium, and potassium; in minute traces, barium, strontium, &c. These metals were chiefly combined with chlorine, iodine (0·015 grm. per litre), bromine (0·012 grm. per litre), and carbonic acid; to a slight extent with silicic, phosphoric, boric, and sulphuric acids. In addition it contained free carbonic acid, marsh gas, and some solid organic substance.—J. T. C.

New Books.

Einführung in das Studium der Alkaloïde, mit besonderer Berücksichtigung der vegetabilischen Alkaloïde und der Ptomaine. Von Dr. Icilio Guareschi. Mit Genehmigung des Verfassers. In Deutscher Bearbeitung herausgegeben von Dr. Hermann Kunz-Krause. Zweite Hälfte. (Schluss des Werkes.) 1897. Hermann Heyfelder, Schönebergerstrasse 26, Berlin, S.W. Price M. 18. H. Grevel and Co., 33, King Street, Covent Garden, London.

The appearance of the German translation of the first half of this work was noticed in this Journal, 1896, 926. The appearance of the second half is now announced. It is a quarto volume, unbound, and simply a continuation of Part I., the text extending from page 305 to 632. At the end there is an alphabetical index filling nearly 18 pages, and a table of contents.

Some Unrecognised Laws of Nature. An Inquiry into the Causes of Physical Phenomena, with special reference to Gravitation. By Ignatius Singer and Lewis H. Berens. With Illustrations. John Murray, Albemarle Street, London. 1897. Price 18s.

8vo volume, containing preface, table of contents, subject-matter filling 477 pages, and an alphabetical index. The text is illustrated by 67 woodcuts, and is subdivided into four books, each of which is separately divided into chapters. Book I. Chap. I. Introduction. II. Methods of Inquiry and Sources of Error. III. Methods of Verification. IV. The Basic Principle of the Current Theory of Gravitation. Book II. Chap. I. Persistence. II. Relative Resistance. III. Equalisation. IV. General Tendency of Nature. V. and VI. On "Forces." VII. "Matter" and "Energy." VIII. Conservation of Energy. Book III. Phenomenology, or the "Interconvertibility of Forces." Chap. I. Introductory. II. Acceleration and Retardation. III. and V. Heat. IV. Molecular Physics. VI. and VII. Electricity. VIII. Conduction. IX. Induction. X. and XI. Action at a Distance. XII. Magnetism. XIII. Polarity of Magnets. XIV. Para- and Diamagnetism. XV. Summary and Conclusions. XVI. Hardness and Solidity. Book IV. Gravitation. Chap. I. Introductory. II. Teachings of Newton. III. Review of the Newtonian Theory. IV. Definitions and Principles of Motion. V. Gravity. VI. The Earth. VII. Sun and Earth. VIII. Causes of Seasons and Variable Days and Nights. IX. and X. Diameter of the Earth's Orb. XI. Suggestions concerning Sun Spots. XII. Testimony of the Stars. XIII. Precession and Nutation.

Trade Report.

EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

The German Leather Industry.

Board of Trade J., May 1897, 542.

In a despatch to the Foreign Office, dated 17th April last, Viscount Gough, Her Majesty's Chargé d'Affaires at Berlin, forwards a précis, by the Commercial Attaché of the Embassy, of a book lately published, called the "Leder-Herold, 1897," which is, practically, a report on the whole leather industry of Germany. This is one of the most important of German industries, and may be said to occupy the third to fourth place, if all its branches are taken into account, and it is one that is daily expanding in every direction. According to the census of 14th July 1895, the number of persons engaged in it were 596,717, of whom 433,586 were occupied in the boot and shoe trade, 74,839 in making saddlery, harness, &c., 47,480 in tanning, and 16,278 in the glove trade. The commerce in hides and skins is immense, and is one of the leading branches of trade, as is also that of tanning materials. In 1895 the value of the imports of the former was 7,297,700*l.*, and that of the exportation amounted to 2,579,050*l.* Besides this, 1,549,500*l.* worth of tanning materials were brought into the country. The exports of worked-up hides, leather, and oil-cloth were valued at 4,925,450*l.*, and those of leather, harness, and pocket articles, 4,914,350*l.*

In consequence of the immense increase of the leather manufacture in the Hamburg neighbourhood, that town has become the emporium of the Continent for hides and tanning materials, and has quite eclipsed Havre and Antwerp in that respect.

There seems to be no doubt that this industry has a still greater future before it. It is as yet in a partly transition state from one of primitive and hand methods to one of mechanical and chemically-aided production on a large scale. The consumption of leather wares is greatly on the increase. At the present time it amounts to about 20,000,000*l.*, to which must be added the exportation of leather and manufactures thereof, which in 1895 were valued at 10,000,000*l.* The German army consumes alone an immense quantity of leather goods. 500,000 pairs of boots are yearly supplied to them; and further, there are large orders for resoleing. The manufacture of machine-made boots and shoes has advanced greatly, and is now a large independent branch of the whole.

As regards leather and its manufactures, Germany has now become fairly independent of foreign supplies, and will daily become more and more so. The rise of this industry suffered greatly from foreign competition; and the tanning trade could not formerly satisfactorily compete with North American manufacturers of leather, who imported hides tanned with hemlock—a very cheap process. It was this circumstance that led to a great agitation on the part of leather manufacturers and tanners in Germany for heavy protective duties. This demand was accorded them by the new tariff of 19th July 1879, by which the import duties were immensely raised. Prior to 1878 the quantity of raw hides imported had increased from 400,000 centners to 800,000 in that year. Since then the leather industry has grown and flourished, and the greatest care has been taken to make use of the newest methods of production, which have all been carefully experimented with. Particular attention has been paid to every detail in manufacture for the perfecting and cheapening of processes. The use of "Quebracho" wood as a tanning medium has caused a revolution in production, and, particularly in the neighbourhood of Hamburg, many special works for its use have been erected.

The technical side of leather production has had a great influence, from the attention bestowed upon it. Among the newest creations of the tanners' guilds are the establishment of a special trade school and technical institute. In Freiberg, in 1889, a tanning school was founded by the Saxon leather industry. A chemical technical laboratory was also started in 1881. Both these institutions have brought the production of leather to a wonderful pitch of scientific perfection. In many other special branches of this trade there are schools of the same nature—as, for instance, for sewing gloves, making saddlery, &c.

The year 1895 was in two respects unsatisfactory for the leather industry: firstly, because of the unexpected rise in price of leather and hides; and, secondly, from the uncertainty as to the result of the proposal to place a duty on the importation of "Quebracho" wood and other foreign tanning materials, which was supported in the Reichstag by a powerful party interested in improving the position of the tanning materials of Germany. The "Bundesrath," however, rejected the resolution of the "Reichstag." It has since been proved that the duty-free admission of "Quebracho" is necessary to the maintenance of that country's competition in the world's markets, and that any duty making leather manufacture dearer would do much harm. It was also undesirable, because that wood is becoming scarcer, and consequently German tans will in time acquire, naturally, a better price.

An attempt has been made last year to improve the statistics relating to leather industries, to obtain a reduction of railway freights for salted hides (it is believed they will come into force on 1st April 1897), and to still further apply scientific knowledge to all processes. Dr. Gürke, of the Botanical Museum in Berlin, has taken steps to obtain a collection of the world's tanning materials, which will be exhibited with details as to the production, use, and price.

The tanning school in Freiberg, Saxony, which was opened on May 1st, 1889, is the only one of its kind in Germany, and was then the only one in the world. Since that date, however, similar schools have been established in Vienna, Hungary, and North America, and Würtemberg and Rotterdam have both planned such schools.

The object of this Saxon tanning school is to teach the principal technical processes, all improved methods and technique, and the trading methods of dealing with raw material, &c. The special points of instruction are as follows:—Tanning and preparation of hides, with practical examples; scientific lectures on tanning; general chemistry; practical chemistry in tanning trade; chemical experiments; tanning methods, "tawing" ("weissgerberei") by fat and minerals; colouring of leather; physics; microscopic demonstrations; knowledge of machines; drawing; book-keeping; trade accounts; trade apprenticeship; German language, correspondence and business letters; first help to be given in accidents.

The course lasts one year. The fees for Germans are 10*l.*, and for foreigners, 17*l.* 10*s.*; they are payable in two instalments. A further 1*l.* 5*s.* is charged for use of the laboratory and for tanning apprenticeship. Rooms with or without board can be had; and at a cost of from 25*l.* to 45*l.* a year lodging and food can be had for the year. Prospectus is sent on application. For theoretical instruction there are collections available to students which cover all branches of the trade. There is a library of 400 volumes, in which are to be found the chief publications on this industry in all languages.

From 1889 to 1895, 173 Germans and 133 foreigners attended the school. (In the list of students for last year not one Englishman figures.)

The "Leder-Herold" for 1897 also treats of the course of study in the chemical technical laboratory of the Society of German Tanners, which is in Berlin, and was the first of its kind in Germany. A list is also given of the technical schools for boot and shoe makers, saddlers, harness makers, and paperhangers, and for sewing of gloves.

In addition, there are statistics most valuable to the leather trade, dealing with all branches for many years back, and an enumeration of the duties of the principal countries on leather and leather manufactures—including Germany, Belgium, France, Greece, Italy, Norway, Austria-Hungary, Roumania, Russia, Sweden, Switzerland, and the United States.

GENERAL TRADE NOTES.

Gunpowder and Dynamite in Greece.

Bd. of Trade J., May 1897, 545.

At the present time in Greece, according to official Greek statistics quoted in the *Bollettino di Notizie Commerciali*, there are 29 manufactories of powder—*i.e.*, three (the most important) in the neighbourhood of Athens, 15 at Dimitzana, one at Argos, and 10 at Phthiotide,—of which five only are at work. The largest is that of the limited company "Ἑλληνικὸν πυριτιδοποιεῖον," which turns out also the most dynamite, and is, besides, the only one to manufacture sulphate of iron and sulphuric acid for the demand of native manufacturers.

Two other establishments, at the foot of Mount Hymettus, make dynamite only: one belongs to a private individual, and the other, the larger, to the "Société Anonyme Franco-Hellénique de Matières Explosives et de Produits Chimiques," the headquarters of which are at Paris. A few years ago a factory for cartridges was established in the neighbourhood of Athens, which produces annually more than a million cartridges for military and sporting purposes, besides a large quantity of wads.

The total value of the manufactories is estimated at 2,000,000 drachma (80,000*l.*). The annual output of powder of all sorts for any purpose is 675,000 ocques—about 1,917,000 lb., and of dynamite 90,000 ocques, or about 255,600 lb., and the manufactories can increase their output if required. The greater amount of the production is for home consumption, but some is exported. The importation of these products decreases year by year, as can be seen from official statistics.

The Mount Pleasant Phosphates in Tennessee.

Eng. and Mining J., April 24, 1897, 404.

Although the Mount Pleasant phosphate deposits in Tennessee have been opened less than a year there are nine companies at work in it already. The *American Fertiliser* says that experience has already shown that a grade of rock thoroughly acceptable to the domestic market is produced without any treatment other than simply drying the rock as it comes from the ground and screening this product over a half-inch screen after crushing. The drying may be done by burning on wood, which must be resorted to in winter, but during the summer months the rock loses its moisture readily in the sun, and sun-drying has proven to be quite sufficient. In fact, some manufacturers claim that the sun-dried product works better on acidulation than the hot-air-dried rock, the explanation for this being that too much carbonic acid is driven off in the kiln-drying process.

The best posted authorities claim that if any of this rock is to be exported, however, it must be thoroughly washed to reduce the percentage of iron and alumina. There seems to be no doubt in the minds of any persons who have investigated the rock, that a product can be had that will run between 77 per cent. and 80 per cent. bone phosphate, but the great question so far has been how to reduce the iron and alumina below 3 per cent. in cargo lots. Preparations for export business are, however, being made. (See also this Journal, 1896, 837.)

Basic Slag in Germany.

Eng. and Mining J., May 8, 1897, 418.

During the year 1896 there were 83,765 tons of Thomas or basic slag imported into Germany, while the exports, chiefly to Belgium, were 134,257 tons. The fertiliser manufacturers now use large quantities of this slag instead of buying imported phosphate rock, and the total consumption of basic slag in Germany in 1896 is estimated at 800,000 tons.

Verona Earth.

Ch. of Comm. J., April 1897, 89.

A great quantity of terra di Verona is now exported to Great Britain, Malta, and other States, for the manufacture of colours. It goes by the name of Green Earth, Verona, or Veronese Earth. In the event of British colour manufacturers wishing to import it direct, British Consul de Zuccato gives the names of the best firms from whom the earth may be purchased. Such names were courteously given him by the Chamber of Commerce there, *viz.*, Signor Giovanni Colombari, Signor Pietro Bonomi, and Signor Domenico Brauzani, all living at Verona.

The Sumach Trade of Italy.

Ch. of Comm. J., April 1897, 88.

The French Consul at Palermo directs attention to the diminishing export trade of his province in sumach, which, he says, is undoubtedly due to the conduct of the exporters in stooping to adulteration for the purpose of increasing their profits. Mastic tree leaves, which are found on the Tunisian coast in great abundance, and which also contain a certain quantity of tannin, can, it appears, be mixed with sumach leaves without their presence being detected. This mixture has been habitually made in the proportions of 10 to 20 per cent. for sumach styled as first quality, sold in leaves at the rate of 21 to 26 frs. per 100 kilos., and in proportions of 40, 50, and even 75 per cent. for sumach of second quality, selling at 18 to 23 frs. per 100 kilos. delivered f.o.b. Palermo. Buyers have thus been rendered mistrustful, and the demand has diminished in a marked manner. To remedy this state of things and to remove the discredit which has fallen upon the trade, the Italian Government thought it *apropos* to levy a high duty on mastic leaves of Tunisian origin, and to instruct the provincial Agricultural Institute to seek the best means of discovering the adulteration to which sumach is subjected. As a result of this action, Signor Matteo Spica describes a scientific method of easily discovering the presence of foreign bodies in sumach, either in leaf or in powder. Pure sumach gives on analysis a colouring substance of which the intensity is invariable. When the colour sample submitted to examination does not correspond to the true type the adulteration of the sumach can be affirmed.

Effects of the New German Sugar Tax.

Export, **19**, [14], 185—187.

Disappointed with the results of legislative interference, a number of the German raw sugar makers proposed to form a syndicate to maintain the price of the article by restricting the output and sale, but, instead of the expected 50 per cent. of makers, only 38 per cent., representing 24 per cent. of the total annual production, joined the association, so no practical result is likely to ensue. A second syndicate, composed of refiners, was also projected for the purpose of inducing the first syndicate to supply none but the associated refiners, and a premium of 2 marks per 100 kilos. was to be given to purchasers dealing exclusively with the syndicate, but this proposal came to nothing. A similar combination of sugar makers in Austro-Hungary roused considerable opposition on the part of the Bohemian beet growers: so that the effects of the German sugar law has produced dissatisfaction all round, the Eastern German States being opposed to bounties at all.

In view of these manifestations, it is considered sugar premiums should be abolished, since, as at present bestowed, they are insufficient to afford any assistance, and it is assumed that any steps taken in this direction by Germany would soon be followed by France and Austro-Hungary.

—C. S.

The Mineral Resources of Wyoming.

Board of Trade J., May 1897, 555.

Deposits of soda covering vast tracts of country, formed by springs, containing a quantity of soda in solution, exist in the State of Wyoming. Under the influence of an excessively dry atmosphere, the solution evaporates and the soda is deposited in the form of crystals, giving the lakes the appearance of immense fields of ice. There are several of these lakes, some of them having an area of 197 acres, and a depth of from three to four yards. The Belgian Consul at Denver, in a recent report to his Government, states that the preparation of this soda for the market

comes to 16*s.* per ton, the cost of carriage from Wyoming to Chicago is 24*s.* per ton, and the soda ash is worth 4*l.* per ton at New York. Several manufactories are already at work near these lakes, and others are spoken of. These natural depôts probably contain many million tons. It may be noted that 108,169 tons of soda are annually employed in the making of sheet glass in the United States.

At Casper and Lander, in a barren region in the centre of the State, are immense deposits of petroleum, the excellent quality of which is well known: its purity is such that the railway companies use it in a raw state. Several companies have already undertaken the sinking of a certain number of oil wells, and the results obtained appear to show that the petroleum industry is likely to develop to immense proportions.

Coal occupies an important place among the mineral resources of the State, and is found of excellent quality throughout. There is also a bituminous coal which is worked into coke of good value and finds a regular place on the market.

The coal mines are worked on a large scale; during 1895 the output was 2,202,000 tons, of a value of 637,700*l.*

Immense quantities of nickel and asbestos have also been recently discovered, and asphalt, kaolin, and mica exist in several deposits.

German Spirit Drawback.

Chem. and Druggist, May 22, 1897, 819.

A German Imperial order has just been published naming certain tinctures and other spirituous preparations, in the preparation of which duty-free alcohol may henceforth not be used at all, or used only if the goods are sold for medicinal purposes exclusively. The preparations for which the employment of duty-free alcohol is forbidden are:—Essence and spirit of anise, essence of peppermint, hair-spirit (*Spiritus crinalis*), and tinctures of alkanet (acid), pimento, ambergris, ambergris and musk, cordial tincture (*Tinctura asperulæ*), tinctures of cochineal, turmeric, tonca-bean, iva, orris, pepper, quillaia, caramel, and all compounds of ether. Those in which duty-free alcohol may be used, provided the articles are employed for pharmaceutical purposes only, are:—Aromatic vinegar, extract of calamus, alcoholic extract of rhatany, *Mistura oleo-balsamica* (Hoffmann's life-drops), solution of salicylic acid, Spir. ammon. aromat. B.P., spirits of angelica, lavender, rosemary, soap (*Sp. sapon.*), and serpyl linus and tinctures of tolu, benzoin (simple and comp.), catechu, guaiacum wood, kino lavender (simple and comp.), musk, myrrh, pyrethrum, quassia, ratanhia (sweetened), sassafras, and sumbul. When tinctures enumerated in the first schedule are exported they are subject to drawback.

Australasian Sulphur and Arsenic.

Chem and Druggist, May 22, 1897, 820.

Arsenic is now produced in two qualities (white, containing 98 per cent., and grey, containing 92 per cent. of arsenious oxide) at the smelting works at Spottiswoode, near Melbourne. A small quantity of it is reported to have been shipped to London recently. The recent advance in the price of Sicilian sulphur has also led to the renewed working of the large New Zealand deposits. Hitherto the Australian sulphuric acid makers have imported their supplies from Italy and Japan, but on March 20, 320 tons of sulphur arrived at Sydney from the North Island of New Zealand, and another cargo of 600 tons is on the way.

Close of the Petroleum Inquiry.

Chem. Trade J., May 22, 1897, 352.

Although the Petroleum Committee have finished taking evidence, it is still uncertain whether they will be able to state the result of their inquiry this session. They have adjourned until after Whitsuntide, in order that Sir Vivian Majendie may have time to prepare a *précis* of the evidence as the basis for a draft report; but, in view of the varied and important issues that have been raised, a considerable period will be needed for formulating and considering the conclusions and recommendations, and in the circumstances it is quite within the bounds of possibility that the committee may have to be reappointed next year to bring their labours to a conclusion. The principal difference of opinion, when the committee come to consider their report, will probably be as to the suggested raising of the flashing point of petroleum. Other important questions to be considered refer to the manufacture of lamps and the regulations concerning storage; but these are not expected to arouse serious controversy.

The Mineral Production of Germany in 1896.

Eng. and Mining J., May 8, 1897, 451.

The mineral production of Germany, or at least the principal items, for the year 1896, is given by *Glückauf* in a recent issue, the table there presented being taken from advance notes of the official returns. The statement shows generally an appreciation in prices as well as an increase in quantities. Thus, among the metals, copper increased from 903 to 995 marks per ton, and zinc from 201 to 220 marks. The average value of pig iron in 1895 was 43·32 marks (10·83 dols.) per ton, and in 1896 it was 46·98 marks (11·75 dols.) per ton. The lightest increase in values was in coal, the average price last year being reported at 6·92 marks, against 6·81 marks in 1895; while brown coal (lignite) was valued at 2·27 marks, or less than in 1895, when the average was 2·34 marks.

The general summary of the production of minerals for 1896, as compared with the previous year, is as follows, in metric tons:—

—	1895.	1896.	Changes.		Per Cent.
Coal	79,169,276	85,639,861	Inc.	6,470,585	8·2
Brown coal (lignite)	24,788,363	26,797,880	Inc.	2,009,517	8·1
Petroleum	17,051	20,395	Inc.	3,344	19·6
Rock salt	686,940	755,833	Inc.	68,893	10·0
Kainit	680,174	856,290	Inc.	176,116	25·8
Other potash salts	841,748	924,140	Inc.	82,392	9·8
Iron ores	12,349,600	14,162,315	Inc.	1,812,715	14·7
Zinc ores	706,423	729,872	Inc.	23,449	33·1
Lead ores	161,614	154,660	Dec.	6,954	38·7
Copper ores	633,354	717,306	Inc.	83,952	13·3
Gold and silver ores	10,845	18,487	Inc.	8,358	77·4
Cobalt, nickel, and bismuth ores.	5,180	4,087	Dec.	1,093	21·0
Iron pyrites	127,036	124,950	Dec.	2,086	1·6

The year was evidently one of great activity, all the important products showing increases of considerable importance. The most valuable of the potash salts reported in 1896 were 174,515 tons of chlorate of potash and 71,958 tons of Glauber salts. The principal iron ore producing districts last year were Elsass-Lothringen, 4,811,633 tons; Luxemburg, 4,758,741 tons; Bonn, 2,680,889 tons; Breslau (Silesia), 529,602 tons.

The output of the metallurgical works is given below, gold and silver in kilos.; all other products in metric tons:—

—		1885.	1886.	Changes.		Per Cent.
Gold	Kilos.	3,547	2,487	Dec.	1,060	42·5
Silver	"	391,979	428,429	Inc.	36,450	9·2
Copper	Tons	25,777	29,319	Inc.	3,542	13·3
Lead	"	114,491	117,722	Inc.	3,231	2·8
Zinc	"	150,286	153,100	Inc.	2,814	1·9
Pig iron	"	5,417,548	6,295,272	Inc.	877,724	16·2
Nickel and bismuth salts.	"	1,143	1,391	Inc.	248	21·8
Antimony and manganese.	"	989	1,326	Inc.	337	34·0
Arsenic products	"	3,014	2,637	Dec.	377	12·6
Copper sulphate	"	4,638	6,046	Inc.	1,408	30·6
Sulphuric acid	"	537,928	590,888	Inc.	52,960	9·8

Here, again, all the important products show considerable gains in 1896 over the preceding year. Included in nickel and bismuth salts are all products of nickel and bismuth ores, such as oxides, colours and the by-products, uranium salts, &c. Of the lead, 113,792 tons were metallic lead,

3,930 tons litharge and oxides. Of the sulphuric acid, 575,942 tons were ordinary acid, 14,946 tons concentrated acid.

Phosphorus for Australia.

Chem. and Druggist, May 8, 1897, 731.

Shippers of phosphorus to Victoria should note that, in the opinion of the Chief Inspector of Explosives of that colony, the zinc drums at present in use are too thin to prevent leakage of the water, and that in consequence there is great risk from fire. Recently a fire through the leakage of a drum was narrowly averted at Melbourne. The inspector thinks the drums should be thicker and painted outside with several coats of white paint. It is thought that the Victorian Customs may soon make drastic regulations regarding the landing and storing of phosphorus.

BOARD OF TRADE RETURNS

Summary of Imports.

Articles.	Month ending 30th April	
	1896.	1897.
	£	£
Metals	1,683,525	1,701,612
Chemicals and dyestuffs	528,531	608,029
Oils	628,705	522,568
Raw materials for non-textile industries.	3,324,050	3,307,117
Total value of all imports	35,808,800	35,136,555

Summary of Exports.

Articles.	Month ending 30th April	
	1896.	1897.
	£	£
Metals (other than machinery)	2,716,535	2,958,212
Chemicals and medicines	715,256	856,276
Miscellaneous articles	2,723,558	3,057,121
Total value of all exports	18,450,227	19,700,122

Imports of Raw Material for Non-Textile Industries for Month ending 30th April.

Articles.		Quantities.		Values.	
		1896.	1897.	1896.	1897.
				£	£
Bark, Peruvian	Cwt.	4,907	2,165	8,847	4,581
Bristles	Lb.	380,408	361,103	62,219	55,928
Caoutchouc	Cwt.	31,788	30,363	341,940	338,561
Gum :—					
Arabic	"	10,843	5,506	32,019	10,379
Lac, &c.	"	16,911	20,538	82,816	97,327
Gutta-percha	"	3,623	3,442	31,100	30,306
Hides, raw :—					
Dry	"	35,912	68,423	88,601	180,106
Wet	"	42,920	42,237	97,245	94,002
Ivory	"	1,077	1,093	41,371	44,463
Manure :—					
Guano	Tons	225	350	1,405	2,650
Bones	"	5,948	6,111	22,926	22,586
Nitrate of soda	"	11,173	12,958	90,147	101,078
Phosphate of lime	"	35,467	32,930	49,505	48,905
Paraffin	Cwt.	50,528	66,617	51,126	55,608
Linen rags	Tons	1,739	1,873	15,757	19,801
Esparto	"	14,981	10,025	64,779	40,688
Pulp of wood	"	24,197	25,610	118,056	131,091
Rosin	Cwt	196,319	67,541	50,576	16,518
Tallow and stearin	"	200,828	102,008	217,013	99,162
Tar	Barrels	846	2,899	411	1,972
Wood :—					
Hewn	Loads	223,926	231,547	411,389	461,351
Sawn	"	229,436	222,569	543,753	551,452
Staves	"	7,747	8,573	42,241	53,633
Mahogany	Tons	3,987	5,281	34,658	52,853
Other articles	Value £	..	..	824,147	791,516
Total value		..	..	3,324,050	3,307,117

Besides the above, drugs to the value of 67,317*l.* were imported, as against 77,811*l.* in April 1896.

Imports of Metals for Month ending 30th April.

Articles.		Quantities.		Values.	
		1896.	1897.	1896.	1897.
				£	£
Copper :—					
Ore	Tons	3,547	2,432	35,080	24,923
Regulus	"	9,748	6,489	247,468	177,910
Unwrought	"	5,217	3,791	237,968	189,586
Iron :—					
Ore	"	522,637	480,675	340,192	344,593
Bolt, bar, &c.	"	3,608	4,737	28,314	34,487
Steel, unwrought	"	512	6,081	6,754	31,876
Lead, pig and sheet	"	13,148	15,249	146,784	178,457
Pyrites	"	64,701	49,512	110,207	81,877
Quicksilver	Lb.	22,784	1,179,708	2,079	113,813
Silver ore	Value £	..	..	90,422	92,272
Tin	Cwt.	36,518	41,188	109,067	123,235
Zinc	Tons	5,516	0,262	84,267	109,187
Other articles	Value £	..	..	244,753	199,396
Total value of metals		..	..	1,683,325	1,701,612

Imports of Chemicals and Dyestuffs for Month ending 30th April.

Articles.		Quantities.		Values.	
		1896.	1897.	1896.	1897.
				£	£
Alkali	Cwt.	16,742	11,581	7,873	6,372
Bark (tanners', &c.)	"	23,285	27,000	8,407	9,032
Brimstone	"	29,903	29,015	5,003	5,519
Chemicals	Value £	..	..	98,096	103,489
Cochineal	Cwt.	143	165	1,128	959
Cutch and gambier	Tons	2,823	2,158	64,036	39,440
Dyes :—					
Alizarin	Value £	..	..	22,412	16,437
Anilin and other	"	..	..	34,835	39,697
Indigo	Cwt.	4,827	11,575	70,343	210,746
Nitrate of potash	"	39,608	35,258	32,837	24,779
Valonia	Tons	3,319	3,876	33,208	37,680
Other articles	Value £	..	..	150,353	113,929
Total value of chemicals		..	..	528,531	608,029

Imports of Oils for Month ending 30th April.

Articles.		Quantities.		Values.	
		1896.	1897.	1896.	1897.
				£	£
Cocoa-nut	Cwt.	14,861	17,802	19,880	18,949
Olive	Tuns	2,408	898	77,521	30,625
Palm	Cwt.	81,992	63,713	84,345	65,283
Petroleum	Gall.	14,380,459	12,900,838	268,564	236,690
Seed	Tons	3,175	2,230	61,908	63,626
Train, &c.	Tuns	1,977	1,095	26,232	19,907
Turpentine	Cwt.	2,547	12,342	1,984	11,849
Other articles	Value £	..	..	88,271	75,540
Total value of oils		..	..	628,705	522,568

Exports of Drugs and Chemicals for Month ending 30th April

Articles.		Quantities.		Values.	
		1896.	1897.	1896.	1897.
				£	£
Alkali	Cwt.	454,010	477,566	111,739	125,849
Bleaching materials	"	93,619	88,184	31,974	28,076
Chemical manures	Tons	25,448	34,239	130,649	169,683
Medicines	Value £	..	..	88,653	91,615
Other articles	"	..	..	352,241	441,053
Total value		..	..	715,256	856,276

Exports of Metals (other than Machinery) for Month ending 30th April.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Brass............ Cwt.	9,567	7,771	37,409	35,523
Copper:—				
Unwrought.... „	49,516	28,087	113,373	73,199
Wrought...... „	25,954	24,686	72,273	76,414
Mixed metal... „	26,045	18,775	56,959	46,108
Hardware...... Value £	..	..	162,803	185,425
Implements..... „	..	..	108,374	113,532
Iron and steel... Tons	289,333	331,038	1,967,985	2,170,121
Lead............ „	3,002	2,683	39,336	36,801
Plated wares... Value £	..	..	21,215	30,328
Telegraph wires. „	..	..	23,066	75,688
Tin............. Cwt.	9,459	8,628	30,406	27,650
Zinc............ „	18,860	12,092	11,916	9,123
Other articles.. Value £	..	..	71,420	77,297
Total value.......	..	..	2,716,535	2,958,212

Exports of Miscellaneous Articles for Month ending 30th April.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Gunpowder...... Lb.	513,400	633,200	13,780	11,956
Military stores.. Value £	..	..	120,471	154,840
Candles.......... Lb.	1,710,700	1,902,100	26,997	26,354
Caoutchouc..... Value £	..	..	98,234	95,063
Cement.......... Tons	30,150	25,908	49,174	41,817
Products of coal Value £	..	..	162,811	179,135
Earthenware... „	..	..	153,420	171,591
Stoneware...... „	..	..	17,825	20,184
Glass:—				
Plate........... Sq. Ft.	167,563	114,296	7,287	7,770
Flint........... Cwt.	6,599	9,304	16,283	19,650
Bottles......... „	64,488	85,338	30,024	40,511
Other kinds.... „	21,360	25,660	16,286	21,652
Leather:—				
Unwrought.... „	9,908	13,325	92,447	117,719
Wrought..... Value £	..	..	27,601	30,487
Seed oil......... Tons	5,138	5,644	97,098	92,849
Floorcloth ..... Sq. Yds.	1,922,300	2,572,500	79,537	105,435
Painters' materials Val. £	..	..	139,563	138,322
Paper.......... Cwt.	81,130	95,524	129,145	149,227
Rags............ Tons	4,012	4,432	22,312	25,988
Soap........... Cwt.	54,895	65,989	59,041	70,791
Total value..........	..	..	2,723,558	3,057,121

Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—PLANT, APPARATUS, AND MACHINERY.

Applications.

9345. A. Forrett. An incandescent furnace for heating gas retorts. April 13.

9522. E. Edwards.—From C. Schaaf. Improvements in muffle furnaces. April 14.

9523. A. J. Campbell.—From E. Sterne. Machinery or apparatus for charging metal capsules or containers with highly compressed or liquefied gas, and for closing the same when so charged. April 14.

9524. A. J. Campbell.—From E. Sterne. Improvements in means for closing capsules or containers for liquefied or highly compressed gases. April 14.

9582. S. H. Johnson. Improvements in filter presses. Complete Specification. April 15.

9929. G. Buck and E. Strong Torrey. Improvements in apparatus for carburetting air. Complete Specification. April 20.

9941. J. Klein. Improvement in apparatus for cooling, concentrating, evaporating, and graduating liquids. Complete Specification. April 21.

10,202. C. Humfrey. Improvements in condensers. April 23.

10,867. A. Ehrhardt. Improvements in linoleum drying and oxidising apparatus. Complete Specification. May 1.

10,988. F. J. Collin. Apparatus for cooling and washing gases. May 3.

11,181. E. Makin, jun. Improvements in apparatus for the condensation of steam and other vapours, and the cooling of heated liquids. May 5.

11,225. G. B. Ellis.—From La Société des Usines du Rhone, anciennement G. P. Monnet et Cartier. Improvements in means for closing receptacles for highly volatile liquids and for regulating the discharge therefrom. May 5.

11,450. H. McPhail. An improved method of and means for evaporating or drying viscous substances, semi-liquids, and pasty materials. May 7.

11,451. H. McPhail. An improved method of boiling and evaporating liquids, and of digesting materials in liquids. May 7.

11,542. D. Williams and E. R. Davies. An improved gas, smoke, and foul air extractor. May 10.

11,700. E. Simoneton. Improvements in the process of and apparatus for filtering liquids. Complete Specification. May 11.

11,724. G. Williams and R. J. Waddell. Improvements in evaporators for brine or other solutions. May 11.

11,803. J. Williamson. Improvements in filter presses and similar filtering apparatus. May 12.

11,872. J. Hill. Improvements in filter presses. May 13.

Complete Specifications Accepted.*

1896.

8250. T. O'Hara and J. R. and T. Ashmore. Valves. April 21.

9084. J. Longshaw. Vacuum apparatus for drying, distilling, concentrating, and other like purposes, and method and means for obtaining and maintaining vacuum therein. April 21.

10,547. C. H. C. Rudd. Improvements in diaphragm and disc valves for chemical corrosive fluids, gases, &c. May 19.

11,136. E. C. Mills and J. G. Chamberlain. Filters for extracting grease and other impurities from feed water for steam boilers. May 12.

11,633. S. K. Welch. Filtering apparatus. May 5.

12,109. P. Hereng. Carburetting apparatus. May 19.

12,572. S. G. Merrick and H. L. Washburn. Vapour condensers and apparatus for destroying noxious fumes, recovering by-products from distillation, and like purposes. May 5.

14,478. F. Dehaitre. Apparatus for drying and finishing fabrics. May 5.

14,609. M. Stanbrook. Apparatus for cooling air for use in refrigerating. May 12.

14,951. E. J. Duff and J. Brock. Evaporating brine and other solutions, and apparatus therefor. May 5.

14,952. T. Glover and J. Brock. Apparatus for evaporating solutions with multiple effect. May 19.

15,797. E. C. Mills. Apparatus for cooling water for condensing and other purposes. May 19.

25,643. M. H. Smith. Apparatus for cooling purposes and for diffusing heat. May 12.

29,491. J. V. Forns. Improvements in tele thermometers. April 21.

1897.

2900. H. Hirzel. An improved column for condensing, absorbing, heating, distilling, and otherwise treating gases and liquids. April 21.

4762. R. Matchett. Apparatus for reducing the pressure of liquids and gases. May 5.

6916. P. Jensen.—From G. Lunge and L. Rohrmann. Improvements in Glover towers, Gay-Lussac towers, and the like, used, for instance, in the manufacture of sulphuric, muriatic, and nitric acids. May 5.

7127. P. Jensen.—From L. Rohrmann and H. H. Niedenfuhr. Improvements in Glover towers and the like. May 12.

II.—FUEL, GAS, and LIGHT.

Applications.

9294. A. H. Barthez. An improved portable and automatic apparatus for the production of acetylene gas for lighting purposes. Filed April 12. Date applied for Sept. 22, 1896, being date of application in France.

9387. J. A. Sinclair. Improved means for lighting by electricity and incandescence. April 13.

9611. S. Straker. Improved apparatus for the production of an explosive gas by carburetting benzoline and other hydrocarbons. April 15.

9656. W. A. G. v. Heidenstam. Improvements in or connected with the manufacture of charcoal. April 15.

9714. U. Kesselring. An improved apparatus for the manufacture of acetylene gas. Complete Specification. April 15.

9762. J. G. A. Kitchen. Improvements in burners for acetylene. April 17.

9763. J. G. A. Kitchen. Improvements in or connected with portable generators for producing acetylene from calcium carbide. April 17.

9770. J. Walsh. Improvements in gas lighting and in electric lighting for magnifying purposes, and the application of same to lighting in general. April 17.

9857. E. K. Hutton and W. Hutton. Acetylene gas generator for lighting cycle and carriage lamps. April 20.

9953. C. E. Melville and J. Horne. An improved retort to be used in the manufacture of oxygen gas from atmospheric air. April 21.

9996. W. Buddeus. New or improved process for obtaining thorium oxide and for enriching monazite sands. Complete Specification. April 21.

10,103. J. M. M. Rosenberg. Improvements in or relating to apparatus for the production of acetylene gas. April 22.

10,186. D. C. Morency. Improvements in apparatus for generating acetylene gas. Complete Specification. April 23.

10,199. W. P. Thompson.—From The Deutsche Acetylen-gas Gesellschaft mit beschränkter Haftung. Improvements in apparatus for developing acetylene gas. Complete Specification. April 23.

10,249. W. P. Thompson. — From The Deutsche Acetylen-gas Gesellschaft mit beschränkter Haftung. Improvements in or connected with apparatus for the production and consumption of acetylene gas. Complete Specification. April 24.

10,320. M. Sheftel. Improvements in the manufacture and production of bodies for use as filaments in electric incandescent lamps, or for making hoods or mantles for incandescent gas lighting, or for like purposes. April 26.

10,321. P. Dvorkovitz and M. Sheftel. Improvements in the manufacture and production of mantles or hoods and other incandescing bodies for lighting purposes. April 26.

10,371. A. A. Stephenson. An improved method and composition for making gas. Complete Specification. April 26.

10,423. F. J. Clinch-Jones. Improvements in gas producers. April 27.

10,508. E. Evans. Improvements in apparatus for generating acetylene or other gas generated by the addition of a liquid to a solid. April 27.

10,686. J. J. Patterson. Improvements in apparatus for generating acetylene. Filed April 29. Date applied for Dec. 22, 1896, being date of application in United States.

10,796. H. J. Barbier. Improvements in means for the storage of compressed gas. April 30.

10,825. A. S. Bower and G. Bower. *See* Class X.

10,900. H. Hansen. Improvements in the manufacture of fuel blocks from petroleum. May 1.

10,911. A. A. Quentin. Improvements in apparatus for carburetting air. May 1.

11,010. M. Otto. Improvements in the manufacture or production of ozone and in apparatus therefor. May 3.

11,130. A. C. Humphreys and A. G. Glasgow. Improved method and apparatus for making carburetted water-gas. Complete Specification. May 4.

11,131. A. C. Humphreys and A. G. Glasgow. Improved process and apparatus for making carburetted water-gas. Complete Specification. May 4.

11,210. L. de Proft. A process of treating mantles for incandescent gas lighting to render the same flexible or supple. Filed May 5. Date applied for Oct. 5, 1896, being date of application in Belgium.

11,252. F. Van den Abeele. Improved automatic apparatus for supplying incandescent gas-burners with gas under pressure. Complete Specification. May 5.

11,641. H. Satcliffe. Improvements in gas generators. May 11.

11,739. F. R. Foster. Improvements in incandescent mantles for gas-burners. Complete Specification. May 11.

11,838. F. B. Courtenay. Improvements relating to gas-producing apparatus. May 12.

11,873. T. Jamieson. Apparatus for generating acetylene gas from a metallic carbide. May 13.

12,021. H. Freise. Process for manufacturing a smokeless inflammable wick. Complete Specification. May 14.

12,096. L. J. Davies. An improved process for making coke from anthracite and similar coal. Complete Specification. May 15.

12,108. Sir C. S. Forbes, Bart. Improved apparatus for vaporising and burning liquid fuel. May 15.

12,110. G. Trouvé. Improvements in apparatus for the production and storage of acetylene gas. May 15.

Complete Specifications Accepted.

1896.

8384. B. H. Thwaite. Process and plant for generating combustible gases. April 21.

8402. C. D. Jenkins. Preparation of peat for fuel, and apparatus therefor, and for analogous purposes. April 21.

9599. R. Bradshaw. Artificial fuels. May 12.

9630. C. Coppeaux. Method and means or apparatus for facilitating the combustion of acetylene gas. May 12.

10,407. R. Goodwin. A portable lamp standard to produce and burn acetylene gas or acetylene and carbonic acid gas combined. April 21.

10,725. G. Webb, jun., and J. W. Kelly. Apparatus for automatically generating gas. April 28.

11,530. The Economical Gas Apparatus Construction Co., Ltd., and L. L. Merrifield. Apparatus for the manufacture of carburetted water-gas. May 5.

11,632. J. E. Goldschmid and C. Dellwik. Manufacture of water-gas. May 5.

11,951. J. Meikle. Treating peat, coal, and other carbonaceous substances for the obtainment of illuminating gas. May 5.

12,047. M. C. A. Fourchotte. Method of and apparatus for generating acetylene gas. April 21.

12,430. A. Arter. Treatment of gas for illuminating and other purposes. May 19.

12,535. O. Imray.—From F. Brunck and Co. Horizontal coke ovens. May 5.

12,942. T. Thorp and T. G. Marsh. Method and apparatus for treating acetylene prior to combustion. May 19.

13,659. J. F. Duke. Manufacture of mantles or incandescing bodies for incandescent gas lamps. April 28.

14,365. C. Killing. Incandescence bodies for incandescent gas lighting. May 5.

14,845. R. Langhans. Manufacture of incandescing media for use in incandescent gas lighting. May 19.

14,941. H. Strache. The manufacture or preparation of improved odorising substances for artificially imparting odour to gases. May 12.

14,943. H. Strache. Process and apparatus for the removal of sulphuretted hydrogen from gases or gaseous mixtures. May 19.

15,122. F. H. Haviland, A. Holloway, J. B. Collier, and W. H. Murch. Apparatus for generating acetylene gas. April 28.

15,139. G. Isaac. Process for rendering acetylene gas inactive when brought into contact with metals with which it forms explosive compounds. April 21.

1897.

6. T. J. Meek, H. Davison, and J. Southam. An improvement in ascension pipes used in the manufacture of coal-gas. May 19.

885. T. Holliday. Means or apparatus for use in the production, storing, and application of acetylene gas. April 21.

1519. A. Sterza. Apparatus for preparing or rendering acetylene gas fit for heating purposes. May 5.

2194. P. W. von Gehlen. Apparatus for producing and using an incandescent light produced from benzene gas. May 19.

2292. G. de R. de Sales. Apparatus for generating and storing acetylene. May 19.

2976. W. H. Dennis. Acetylene gas lamps and generators. May 12.

5149. A. Roedel. Process and apparatus for removing graphite crusts from the interior of gas retorts. April 21.

5659. J. Kohlndorfer. A process for producing a solid or consistent fuel containing petroleum. April 21.

5756. H. R. Bean and H. Ringwood. An improved automatic generator, condenser, and holder for acetylene and other gases. April 21.

6997. G. Pereire, E. Sorel, and B. Cruvellier. Manufacture of acetylene gas and apparatus therefor. May 5.

7744. E. G. Brewer. — From The Gesellschaft für Acetylen-Gaslicht Basel. Apparatus for generating acetylene gas. May 5.

7782. H. H. Lake.—From F. Ferraccin. Apparatus for automatically generating acetylene gas. May 5.

7920. J. B. de Lery. Method and apparatus for effecting illumination partly by flame and partly by incandescence. May 12.

8779. D. Meyer. Incandescent gas burners. May 19.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Application.

10,577. G. C. Marks.—From La Compagnie pour la Fabrication des Compteurs et Materials d'Usines à Gaz. Triple-action apparatus for the recovery of benzols or other products contained in certain gases. April 28.

Complete Specification Accepted.

1896.

9944. A. C. Thomson. Vertical retorts for the destructive distillation of carbonaceous substances. April 21.

IV.—COLOURING MATTERS and DYES.

Applications.

9268. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of 1-phenyl 2-alkyl-pyrazolones. April 12.

9702. H. H. Lake.—From K. Oehler and Co. Improvements in the manufacture of colouring matters. April 15.

9710. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation. Manufacture of stable diazo-compounds. April 15.

10,208. S. Pitt.—From L. Cassella and Co. Producing black pigment colours. April 24.

10,605. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co. Improvements in the manufacture or production of colouring matters. April 28.

10,810. J. Hauff. A process for the preparation of orthotoluolsulpho acid and of ortho sulpho benzoic acid by oxidation of orthothiocresol. Complete Specification. April 30.

11,572. C. Mills, and Brooke, Simpson, and Spiller, Ltd. A method of preparing basic direct cotton dyeing colours of a blue and blue-black shade. May 10.

11,871. J. Owen. An improved liquid black stain for staining and polishing articles of vulcanite. May 13.

11,997. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis. Manufacture of substantive colouring matters. May 14.

11,998. H. Imray.—From Anilinoel Fabrik A. Wagfing. Improved manufacture of ammonium salts of casein. May 14.

12,011. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of new alizarine dyestuffs. May 14.

Complete Specifications Accepted.

1896.

9477. L. P. Marchlewski, E. S. Wilson, and E. Stewart. Production of dyes. May 5.

10,004. E. Fischer. Manufacture or preparation of derivatives of xanthine from alkylated uric acids. May 5.

11,370. Read, Holliday, and Sons, Ltd., J. Turner, and J. Turner. Production of colouring matters. April 21.

12,142. S. Pitt.—From L. Cassella and Co. Production of polyazo dyestuffs. April 21.

12,556. S. Pitt.—From L. Cassella and Co. Production of dyestuffs suitable for dyeing wool. April 21.

12,922. A. G. Green and A. Wahl. The manufacture and production of new cotton colouring matters. May 12.

14,144. I. Levinstein and Levinstein, Ltd. Azo colouring matters. May 5.

14,563. J. Y. Johnson. From The Badische Anilin und Soda Fabrik. Manufacture and production of mordant-dyeing colouring matters and of intermediate products relating thereto. May 5.

27,775. J. Ville. New red colouring matters of the triphenylmethane series and the method of producing the same. May 12.

1897.

1575. H. Baum. Manufacture of colouring matter of the rhodamine group. May 5.

V.—TEXTILES, COTTON, WOOL, SILK, Etc.

Applications.

9517. R. Thomas and E. Prevost. Improvements in or relating to the process of mercerising vegetable fibres or textile fabrics. Complete Specification. April 14.

10,547. D. McCallum. The reducing of the inflammability of cotton fabrics. April 28.

10,781. H. Imray.—From La Société Anonyme des Blanchiments, Teintures, et Impressions. A process for giving lustre to vegetable fibres and textures. April 30.

10,784. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning. An improved method of mercerising cotton yarns. April 30.

10,914. W. Scheulen and H. Mann. Improvements in the process of finishing bleached and cleaned textile fabrics, yarns, and woven goods of cotton, wool, and tussah silk. May 1.

11,313. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning. Improvements in mercerising vegetable tissues. May 6.

11,804. A. H. Prinz. Improvements in the treatment of jute, bast, china grass, rhea, and like plants for use in textile industries. Complete Specification. May 12.

11,826. Count Hilaire de Chardonnet. Improvements in the manufacture of artificial silk. May 12.

11,917. O. Imray. — From The Farkwerke vormals Meister, Lucius, and Brüning. A process for chlorination of wool. May 13.

Complete Specifications Accepted.

1896.

12,693. S. Bennett. Treatment of wood, paper, fabrics, fibres, and like materials impervious and inalterable by moisture and climate and other influences. May 19.

13,707. T. E. Briggs and E. Webb. Apparatus for polishing and drying yarns, threads, twines, or cords. April 28.

1897.

3832. R. W. Strehlenert. Method of spinning artificial silk and apparatus therefor. April 21.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

Applications.

9392. C. E. Gassmann. A new process of fixation of colours basic and phenolic in printing. April 13.

9499. J. Grossmann. A new indigo vat. April 14.

9804. P. Dosne. Process for imitating on vegetable fibres, fabrics woven in colours. April 17.

10,213. E. Bentz. Improvements in the production of white and coloured patterns on aniline black grounds in calico printing. April 24.

10,787. C. D. Abel.—From F. Petersen and Co. Process for dyeing fast black. April 30.

10,901. J. Imray. — From La Société Anonyme A. Roudillon et Cie. A process for fixing interference colours on paper or other surface. May 1.

10,933. G. Wendler. A new or improved [illegible] adapted for use in dyeing wool, in bread or pastry, and other purposes. May 3.

11,110. C. W. Fulton. Method of producing patterns upon textile fabrics. May 4.

11,983. C. W. Fulton. Improvements on methods of producing patterns on textile fabrics. May 14.

Complete Specification Accepted.

1896.

10,936. A. F. B. Gomess. Bleaching certain vegetable fibres, more especially those of the urtica family. April 21.

VII.—ACIDS, ALKALIS, and SALTS.

Applications.

9418. C. M. White. — From Smith, Powers, Simson Geenens, and Co., Ltd. Improvements in the manufacture of chloride of baryta and oxide of iron. April 13.

10,183. G. B. Ellis.—From La Société Chimique des Usines du Rhone, anciennement G. P. Monnet et Cartier. Improvements in the manufacture of aldehydo-benzoic acid and intermediate products. April 23.

10,361. C. Masson and C. Tilliere. Improvements in the preparation or manufacture of borax. April 26.

10,400. A. Crosbie. *See* Class X.

10,501. A. Feldmann. A process for removing carbonic acid and hydrogen sulphide from ammoniacal liquor. Complete Specification. April 27.

10,509. T. T. Sill. *See* Class X.

11,240. W. Shedlock. Improvements in apparatus for obtaining salt from brine and for similar purposes. May 5.

11,353. J. R. Wylde, J. W. Kynaston, and the United Alkali Co., Ltd. Improvements in the manufacture of hydrochloric acid free from arsenic. May 6.

Complete Specifications Accepted.

1896.

13,244. A. Crossley and H. A. Allport. Manufacture of ferric oxides. April 21.

13,844. J. Brock and F. Hurter. Recovering sulphur from sulphuretted hydrogen. April 21.

23,245. J. Pattison. Improved ammoniacal compounds. April 28.

1897.

1179. A. J. Boult.—From S. Ganelin and J. Block. Method of making chloride of lead. May 19.

6916. P. Jensen.—From G. Lunge and L. Rohrmann. *See* Class I.

VIII.—GLASS, POTTERY, and ENAMELS.

Applications.

9351. H. Emery. Improvements in the form of saggars for potters' use. April 13.

10,430. H. de Wit. Manufacture of enamelled ceramics with a slaggy biscuit covered over with coloured enamels. Complete Specification. April 27.

10,691. P. Delorme, fils. Improvements in the manufacture of hollow castings of glass and the like, and apparatus therefor. Complete Specification. April 29.

11,343. R. F. Ayre and C. Donnill and Co., Ltd. Improvements in pottery kilns or ovens. May 6.

11,630. C. C. Schirm and O. Lessing. Process for the ceramic production of grinding agents. Complete Specification. May 10.

11,735. G. Tasche and F. Sporer. Improvements in glass cooling or annealing ovens. Complete Specification. May 11.

Complete Specifications Accepted.

1896.

11,522. H. Hayward. System of glazing. April 21.

13,823. W. Macrae. Production of decorative stained glass or imitations thereof. April 28.

22,t-C. W. F. Mason. Kilns for bricks, pottery, and the like. May 5.

1897.

1995. T. Pfister and E. Barthels.—From A. Navarein. Cloisonne work. May 5.

4141. T. W. Horn. Window glass. April 21.

4527. C. Paulus and H. Heim. Manufacture of ceramic ware and machinery or apparatus for the same. April 21.

453-. C. C. Schirm and O. Lessing. Process for producing ceramic masses. May 12.

-104. H. Heckert. Glass hollow-ware. May 12.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Applications.

9315. C. Richardson. Improvements in asphaltic pavements and the like. Complete Specification. April 12.

9316. L. S. Kasson. Apparatus for treating and assembling the ingredients for the manufacture of paving compositions. Complete Specification. April 12.

10,317. W. F. Dyson. Improvements in artificial stone. April 26.

11,016. Kuma, Ltd.—From P. W. Wierdsma and J. Kuipers. *See* Class XIII.

11,153. J. N. Santha. Improvements in or relating to the preparation of asphalt for pavements and the like. May 4.

11,162. J. Davie and J. Baird. Improvements in manufacturing blocks for causeying streets, pawing footpaths, and for other constructive purposes. May 5.

11,403. J. Brock. Improvement in the composition of matter for bricks. May 7.

11,925. H. Coward. An improvement in the construction of fire-proof floors. May 13.

12,024. J. C. Rombach and E. S. Restieaux. Improvements in cements for resisting the action of acids, acid vapours, and heat. May 15.

Complete Specifications Accepted.

1896.

11,817. J. E. Brindley. Manufacture of tiles and quarries. April 21.

12,368. W. Owen. Manufacture of artificial stone, marble, and the like. April 21.

12,693. S. Bennett. *See* Class X.

25,880. A. H. van der Vygh, H. van der Vygh, and G. van der Vygh. An improved fire-proof artificial stone. April 21.

1897.

3571. A. H. van der Vygh, H. van der Vygh, and G. van der Vygh. Construction of fire-proof buildings. April 21.

5439. W. S. Wilkinson. Manufacture of paving blocks, tiles, and the like. April 21.

X.—METALLURGY, MINING, Etc.

Applications.

9403. G. W. Gesner. A new and useful alloy of iron and hydrogen, termed Gesner metal, and means for producing the same. Complete Specification. April 13.

9409. H. R. Angel. Improvements in the treatment and reduction of sulphide ores containing zinc or otherwise. April 13.

9717. F. Ellershausen. Improvements in the treatment of complex sulphide ores. April 15.

9840. H. H. Eames. Improvements in apparatus for amalgamating and concentrating precious metals. April 20.

10,118. A. James. Improvements in the treatment of gold-zinc slimes from the cyanide process. April 23.

10,173. C. P. Shrewsbury. Improvements in means or apparatus for recovering gold from the residue resulting from the treating of tailings by the cyanide process. April 23.

10,400. A. Crosbie. Improvements in the method of preparing iron oxides from waste acids or pickle from tinning and galvanising works. April 27.

10,403. J. Colley. Improvements in linings of metallurgical vessels. April 27.

10,459. A. J. Boult.—From A. Lismann. A new or improved method of coating copper or copper alloys with patina. April 27.

10,509. T. T. Sill. Improvements in the manufacture of red oxide from the waste liquors from galvanising works, tin-plate works, wet copper process, or other residual liquors containing iron in the form of chloride. April 27.

10,634. H. C. Bull, C. P. Shrewsbury, and F. L. Marshall. A new or improved process for extracting gold and other metals from sea water. April 29.

10,715. J. L. Dobell. Improvements in the manufacture of cupels and crucibles to be employed in the refining of metals. April 30.

10,721. G. P. Royston. The annealing of cast iron without melting of the same, by heating at that temperature at which the cast iron, when fused, solidifies. April 30.

10,785. B. H. Thwaite and H. V. Holden. A process for decarburating steel or other compounds of iron and carbon, and apparatus for that purpose. April 30.

10,825. A. S. Bower and G. Bower. A combined process for the protection of iron and steel surfaces from rust, and for the manufacture of combustible gas. May 1.

10,829. J. Swinburne. Improvements in treatment of sulphide ores. May 1.

11,081. J. M. Spink. Improvements in annealing metals during manufacture, and in the means or apparatus employed therein. May 4.

11,151. G. H. Blenkinsop. Improvements in and relating to the roasting and calcining of minerals and the like substances. May 4.

11,410. R. S. Lovelace. An improved method of toughening and strengthening metals. May 7.

11,518. C. P. Shrewsbury. Improvements in means or apparatus for extracting gold and other metals from solutions containing such metals. May 8.

11,602. E. E. von der Linde. Treatment of tin oxide in the form of powder, sludge, or paste for the smelting thereof. Complete Specification. May 10.

11,659. W. Blackmore. The treatment of slags containing zinc and silver. May 11.

11,695. J. C. Mewburn.—From La Société Anonyme de Commentry-Fourchambault. Improvements in the manufacture of alloys of or containing iron and nickel. May 11.

11,996. G. Deer. An improvement in the smelting of copper, and apparatus for that purpose. Complete Specification. May 14.

12,074. F. B. Aspinall and E. C. Ekstromer. Improvements in apparatus for extracting gold from its ore. May 15.

Complete Specifications Accepted.

1896.

3051. J. Robertson. Improvements in shaping, forging, drawing, solidifying, and squirting metals, and in means and apparatus therefor. May 19.

8051. T. Huntingdon and F. Heberlein. Treatment of sulphide ores of lead, zinc, or antimony, preparatory to smelting the same, and incidentally in obtaining sulphurous acid. April 21.

8882. A. Tracy. Method of combining hard metal and soft metal or other material to form non-slipping and non-extensible floor covering and stair nosing. May 5.

9524. J. A. Bruce-Fraser. Process for extracting gold and other metals from their ores, especially applicable to the treatment of refractory ores. May 5.

9656. The Improved Electric Glow Lamp Syndicate, Ltd.—From C. Schwabe. Process for depositing metals on silver coatings. May 12.

11,833. R. A. Hadfield. Method of toughening or softening manganese steel castings. April 21.

12,255. M. B. Zerener. Precipitation of precious metals from their cyanide solutions. April 21.

12,287. W. Van Wart, F. W. Popp, and J. J. Bradley. A certain new or improved metal or admixture of metals. May 19.

12,328. J. C. Bull. Alloys. April 28.

13,111. E. A. G. Street. Manufacture of chromium and similar alloys. May 19.

13,371. O. Frolich. Process for extracting metals from pyritic ore by the treatment of, with chlorine, and for recovering the chlorine employed. April 21.

13,451. L. Pszczolka. Improvements in Bessemer or Thomas process for the treatment of low qualities of crude iron. May 5.

14,006. C. Parlacott. Manufacture of metallic alloy. May 12.

14,622. A. Wolski. Blast furnaces. May 19.

16,756. Elkington and Co., Ltd., and H. T. Fellows. Muffles for annealing metals and for other like purposes. May 12.

1897.

4202. A. F. M. V. Baron. Manufacture of an alloy of copper and iron. May 19.

5926. J. de Moya. Manufacture of steel. May 19.

8873. W. Noad and W. Agate. Improvements in the obtainment of copper from ores and other copper-bearing substances. May 19.

XI.—ELECTRO-CHEMISTRY and ELECTRO-METALLURGY.

Applications.

9276. W. Rowbotham. Improvements in primary batteries. April 12.

9317. Sir C. S. Forbes, Bart. Improvements in plates for secondary batteries. April 12.

9456. J. H. McLean and C. D. Burnet. Improvements in secondary batteries. April 13.

9563. H. T. Cheswright. Improvements in or relating to plates for secondary batteries or accumulators. Complete Specification. April 14.

9631. J. Dunn. A new or improved plate for accumulators or electric storage batteries. April 15.

9803. H. E. de Rufz de Lavison. A voltaic battery having its depolarisation effected by heat. April 17.

9913. E. Marckwald. A manufacture of electrodes for electric accumulators. Complete Specification. April 20.

9914. E. Marckwald. A manufacture of electrodes for electric accumulators. Complete Specification. April 20.

10,251. W. A. Boese. An improved process for the manufacture of accumulator plates. Complete Specification. April 24.

10,279. A. E. Ann.—From C. W. Hertel. Improvements in galvanic batteries. April 24.

10,439. C. E. Lee. Improvements in secondary batteries. Complete Specification. April 27.

10,451. F. S. Brown and L. J. Davies. Improvements in separating stable salts by means of diffusion, and generating electricity direct out of chemical energy. April 27.

10,481. J. L. Dobell. Improvements in or connected with electric batteries. April 27.

10,705. L. Epstein. An improvement in plates for secondary voltaic batteries. April 29.

10,719. W. Rowbotham. Improvements in primary batteries. April 30.

10,822. W. Evans and J. W. Smith. Certain improvements in apparatus for the electro-deposition of metals. April 30.

10,872. J. Sime.—From R. J. Browne and J. G. Dobbie. Improvements in and relating to electric batteries. May 1.

11,190. F. M. Lyte. Improvements in the electrolysis of fused zinc chloride. May 5.

11,231. E. Giglio. Improvements in dry batteries. May 5.

11,236. D. Reynolds. An improved process and means for electrically purifying liquids. May 5.

11,402. W. S. Romme. A process of and apparatus for electrically decomposing solid substances. May 7.

11,578. F. J. Gillibrand. Improvements in or relating to carbon plates, rods, or the like for galvanic batteries. May 10.

11,603. E. Riley. An improvement in plates for secondary voltaic batteries. May 10.

11,800. S. de la Precilla. Improvements in and connected with the formation of galvanic batteries. May 12.

11,844. G. C. Allingham and W. Fennell. Improvements in voltaic batteries and electrolytic decomposition cells. May 12.

11,861. O. Lindner. Improvements in or relating to electric accumulators or storage batteries. May 13.

12,028. R. W. Atkinson and S. F. Walker. Sulphuring carbon plates for galvanic batteries. May 15.

12,073. H. W. Headland and Headland's Patent Electric Storage Battery Co., Ltd. Improvements in secondary battery plates and in moulds for making them. May 15.

Complete Specifications Accepted.

1896.

8906. L. B. Atkinson and F. G. Treharne. Generation of electricity. May 19.

11,338. E. A. G. Street. Electrodes for electrolytic purposes. May 19.

13,200. A. G. Stromberg. Charging accumulators. May 5.

16,256. L. M. Bullier. Electric furnaces. May 19.

16,257. L. M. Bullier. Electric furnaces. May 19.

16,852. G. Poore. Electrolysis. May 5.

22,069. D. Young. From La Société Anonyme "Accumulateur Eclair." Electric accumulators or storage batteries. May 19.

25,369A. F. H. Snyder. Improvements relating to the coating or covering of metals and other substances with metals and other materials by the aid of the electric arc. May 5.

1897.

6818. J. Entwisle. From C. T. Barret. Improved storage battery. April 21.

8131. S. V. B. Essick. Primary electric batteries or generators. May 12.

XII.—FATS, OILS, and SOAP.

Applications.

9314. F. Rapp-Rosenthal. A process for the production of oil from peat. Complete Specification. April 12.

9529. S. Rosenblum, S. Rideal, and The Commercial Ozone Syndicate, Ltd. Improvements in the oxidising of oils and in apparatus for the purpose. April 14.

9799. R. W. English. An improved method of and apparatus for bleaching linseed oil. April 17.

10,526. J. R. Whiting and W. A. Lawrence. Improvements in the process of and apparatus for deodorising oil. Complete Specification. April 27.

10,576. J. E. Bedford and C. S. Bedford. An improved method for obtaining solid products from linseed oil. April 28.

Complete Specifications Accepted.

1897.

1355. E. A. Ruch. Method of concentrating glycerin. May 19.

1554. J. C. F. Muller. Process and manufacture of a cleaning and caustic agent. May 12.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

9288. G. W. Young. A new or improved waterproofing compound. April 12.

9405. A. G. Haehre. Improvements in the manufacture of plastic materials. April 13.

9423. C. M. White.—From Smith, Powers, Simson, Geenens, and Co., Ltd. Improvements in the manufacture of red oxide of iron. April 13.

9447. J. W. Blake and S. J. Blake. Improvements in compositions or paints for preserving structures. April 13.

9461. E. Hornung and S. Hansel. Improvements in the treatment of india-rubber, gutta-percha, and their compounds. Complete Specification. April 13.

9538. F. King. Improvements in or connected with the manufacture of articles wholly or partly of rubber or other gum and the like dissolved in a solvent. April 14.

9775. J. S. MacArthur. Improvements in making white lead. April 17.

11,016. Kuma, Ltd.—From P. W. Wierdsma and J. Kuipers. The manufacture of a new or improved substance or product suitable for use as a substitute for vulcanite, hard woods, or other hard materials, or for other uses or purposes for which same may be applicable. May 3.

11,168. E. Garnier and S. J. Prescott. An improvement in india-rubber compounds and in gutta-percha compounds. May 5.

11,500. T. Grantham and J. J. Grantham. A new or improved boot varnish. Complete Specification. May 8.

11,920. E. J. Soutar-Arnold and W. Baines. A method of extraction or production of caoutchouc. May 13.

Complete Specifications Accepted.

1896.

2677. H. Birkbeck.—From P. W. Wierdsma and J. Kuipers. Manufacture of a new or improved substance or product suitable for use as a substitute for vulcanite, hard woods, or other hard materials, or for other uses or purposes for which same may be applicable. May 12.

10,462. A. H. Sandiland. Manufacture of white lead. May 19.

14,267. D. Marcus. Paints and varnishes. April 28.

14,428. H. C. Webster and R. Crawford. Manufacture of white lead. May 19.

14,884. W. Mitchell.—From G. L. Ball. A liquid for removing varnish. April 21.

1897.

7399. E. O. S. Gentele and H. R. Gentele. Manufacture of pigments, printing inks, or paint mixtures. May 12.

XIV.—TANNING, LEATHER, GLUE, and SIZE.

Applications.

9969. J. Biddulph and E. A. Haggard.—From R. Acklom. Improvements in or relating to the treatment of hides. April 21.

11,111. E. O. Daniel. Improvements in the preparation for and tanning of hides and skins. Complete Specification. May 4.

11,597. G. Levinstein. Improvements in and relating to the tanning of hides and skins. May 10.

12,018. A. MacFarlane and H. W. Stanbury. Process of treating gelatine for the production of imitations of various other substances. May 14.

12,099. C. S. Dolley. Improvements in processes of tanning. Complete Specification. May 15.

Complete Specifications Accepted.

1896.

16,420. E. S. Cook. Process for treating raw hide for use in the construction of pneumatic tyres. May 12.

1897.

3073. J. Howden.—From J. Tyciak. Glue compounds, and applying same to barrels and the like. May 5.

XV.—AGRICULTURE and MANURES, Etc.

Applications.

9369. A. Gladstone. Improvements in and relating to artificial manures or fertilisers. April 13.

9704. C. R. Thompson. The manufacture of improved fertilised and fertilising materials. Complete Specification. April 15.

10,369. A. Quaet-faslem. Improved method of and process for working and treating nitrate deposits for the extraction of the nitrates therefrom, and apparatus for that purpose. April 26.

Complete Specification Accepted.

1896.

17,883. E. van De Griendt. Treatment of moss-litter manure for obtaining products therefrom. May 12.

XVI.—SUGARS, STARCHES, GUMS, Etc.

Applications.

9455. W. Feld. Improved process and apparatus for desaccharising molasses by means of barium hydroxysulphide and for regaining the by-products. April 13.

10,395. H. W. Aitken. Improvements in apparatus for defecating and purifying saccharine juice or liquid. April 27.

11,842. Z. Lubinski and R. Krajewski. Improvements in and relating to centrifugal machines for use in the manufacture of sugar. Complete Specification. May 12.

COMPLETE SPECIFICATION ACCEPTED.

1896.

11,790. G. Ranson. Process for the complete refining of sugar. May 19.

XVII.—BREWING, WINES, SPIRITS, Etc.

APPLICATIONS.

10,180. M. van Look. New or improved process for the preparation of mead. Complete Specification. April 23.

10,899. R. Willcox.—From The Actien-Maschinenbau-Anstalt vormals Venuleth and Ellenberger. Improvements in and apparatus for the preparatory treatment of cereals for use in brewing and distilling. May 1.

11,515. J. H. Folkerts. Improvements relating to the production of yeast and to apparatus therefor. May 8.

11,600. A. C. Briet. Improvements in diffusion apparatus for the manufacture of cider. May 10.

COMPLETE SPECIFICATIONS ACCEPTED.

1896.

28,713. C. Killing. Process and apparatus for purifying crude alcohol and other alcoholic mixtures. April 28.

1897.

1455. A. Kinder. Malt kilns. May 5.

XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

APPLICATIONS.

A.—Foods.

9911. E. B. Watson. Employment of diastase as a preservative of oily or fatty vegetable food-matter. April 20.

10,871. J. Hogarth. Improvements in and relating to alimentary substance and beverages. May 1.

10,873. W. T. Murray. An improved process of preserving milk. Complete Specification. May 1.

10,926. J. A. Cope. An improved preservative for eggs and other articles of food. May 3.

11,513. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of albuminous products soluble in water from seeds of plants. May 8.

11,629. H. Higgins. An improved method of treating "separated" milk for alimentary purposes. May 10.

B.—Sanitation.

9558. R. H. Reeves. Improvements in or relating to the treatment of sewage. April 14.

9858. J. B. Petrie. Improvements in filtering and oxidising sewage and other foul waters after the same may have been treated by precipitants. April 20.

COMPLETE SPECIFICATIONS ACCEPTED.

A.—Foods.

1896.

8232. H. Higgins. Preservation of foods and other perishable goods. April 21.

11,878. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of new albuminous compounds. May 12.

1897.

6064. H. Humbser. Process for preserving substances. May 12.

B.—Sanitation.

1896.

14,124. A. Angell and F. Candy. Preparations for use as precipitants in the treatment of sewage and other polluted water. May 19.

14,125. A. Angell and F. Candy. Manufacture of materials for use in the filtration and purification of impure water and other liquids. May 19.

1897.

5793. P. Altmann. Process for rendering drinking water free from germs. May 12.

XIX.—PAPER, PASTEBOARD, Etc.

COMPLETE SPECIFICATION ACCEPTED.

1896.

12,693. S. Bennett. *See* Class V.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

APPLICATIONS.

9578. J. Hauff. A process for the preparation of sulphinide of benzoic acid (saccharine). Complete Specification. April 14.

9664. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Improvements in the production of a pharmaceutical compound. April 15.

9701. E. Dolle. The manufacture of improved preparations of iron for medicinal purposes. Complete Specification. April 15.

9827. F. Valentiner. A process for the production of aromatic fluoro-hydrocarbons in aqueous solution. Complete Specification. April 17.

9898. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of a new chemical compound. April 20.

10,535. J. W. Mackenzie.—From C. Schmid. A new method for producing an artificial scent or musk-like substance. April 27.

10,594. G. B. Ellis.—From La Société Chimique des Usines du Rhone, anciennement G. P. Monnet et Cartier. Improvements in preparations for producing local anæsthesia. April 28.

10,790. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of new pharmaceutical compounds. April 30.

11,228. A. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Schering. The production of sterile iodoform. May 6.

11,344. W. L. Wise.—From Knoll and Co. Manufacture of compounds of ichthyol. May 6.

11,596. J. Wetter.—From J. D. Riedel. Process for the manufacture of salicylo-acetic acid. May 10.

COMPLETE SPECIFICATIONS ACCEPTED.

1896.

11,458. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Manufacture of pharmaceutical compounds. April 21.

12,434. W. P. Thompson.—From Ludwig, Sell, and Co. Process for making a compound of good keeping property of salicin, saligenin, glucose, and free hydrochloric acid. April 28.

14,552. O. Imray.—From The Fabriques de Produits Chimiques de Thann et de Mulhouse. Manufacture of artificial musk. May 5.

1897.

6220. J. C. Fell.—From W. J. Matheson and Co., Ltd. Process of producing acetanilid or acetoluids. April 21.

XXI.—PHOTOGRAPHY.

Applications.

11,133. G. Selle. Improvements in photographic dark backs for successive exposures for the purpose of colour photography. May 4.

11,612. J Joly. Improvements in or relating to the art of producing photographs in natural colours. May 10.

12,017. J. Wheeler. Improvements in the production of films and in apparatus therefor. May 14.

Complete Specifications Accepted.

1897.

2305. H. H. Lake.—From H. C. Fairchild. Producing photographs in relief. April 21.

3121. G. J. Sershall. Producing photographs with coloured effects. May 19.

XXII.—EXPLOSIVES, MATCHES, Etc.

Applications.

9496. L. Gathmann. Improvements in safety fuses for high explosive shells. Complete Specification. April 13.

9535. T. Teyley. Improvements in explosives. April 14.

9970. E. A. G. Street. Improvements in or relating to explosives. Filed April 21. Date applied for March 23, 1897, being date of application in France.

10,362. O. F. Carlson. Improvements in explosives. Complete Specification. April 26.

11,655. H. Humphreys. A fog signal with three chambers, each fitted with explosive, and guaranteed to explode without the aid of nipples or percussion caps. May 11.

Complete Specifications Accepted.

1896.

9574. H. Hohendahl. Safety fuses. May 12.

11,842. G. G. Andre and C. H. Curtis. Manufacture of explosives. April 21.

15,022. G. B. Ellis.—From La Société Chimique des Usines du Rhone, anciennement G. P. Monnet et Cartier. Manufacture of explosive compounds. May 19.

15,351. A. F. Hargreaves. Explosives. May 12.

1897.

6461. M. van Look. Explosives.

Printed and Published by Eyre and Spottiswoode, East Harding Street, London E.C., for the Society of Chemical Industry.

THE JOURNAL

OF THE

Society of Chemical Industry.

A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

No. 6.—Vol. XVI.] JUNE 30, 1897. [Non-Members 30/- per annum; Members 21/- per Set of extra or back numbers; Single Copies (Members only) 2/6.

The Society of Chemical Industry.

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THE JOURNAL.

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NOTICES.

In accordance with the provisions of Rule 18 of the Bye-laws, notice is hereby given that those Members whose names are placed in italics in the list of Council will retire from their respective offices at the forthcoming Annual General Meeting.

Prof. F. Clowes, D.Sc., has been nominated to the office of President; Dr. Edward Schunck, F.R.S., has been nominated Vice-President under Rule 11; Mr. A. Gordon Salamon and Dr. Wm. Jay Schieffelin have been nominated Vice-Presidents under Rule 24; and Mr. D. Howard, Mr. Ivan Levinstein, and Mr. Wm. Thorp have been nominated Vice-Presidents under Rule 8.

The Hon. Treasurer and Hon. Foreign Secretary have been nominated for re-election to their respective offices.

Mr. John Pattinson and Mr. Walter F. Reid have been nominated under Rule 18, and Sir David Gamble, Bart., C.B., and Prof. J. J. Hummel, under Rule 19, to fill four vacancies among the Ordinary Members of Council.

STATEMENT OF REVENUE AND EXPENDITURE FOR THE YEAR 1896.

REVENUE.

	£	s.	d.	£	s.	d.
Annual Subscriptions for 1896:—						
1 subscription received in 1894	1	5	0			
105 subscriptions received in 1895	131	0	1			
2,588 subscriptions received in 1896	3,235	0	5			
33 subscriptions received in 1897	41	5	0			
(Sundry balances of subscriptions)	0	12	0	3,409	2	6
2,727						
214 Entrance Fees	224	14	0			
4 Life Composition Fees	80	0	0			
Subscriptions received on account of the "Collective Index"	..			347	5	0
Investments:—						
Interest on Metropolitan Consolidated Stock	129	16	4			
Interest on North British Railway Consolidated Lien Stock	31	9	2			
Interest on Midland Railway 4 per cent. Preference Stock	17	15	8			
Interest on Gas Light and Coke Company's 6 per cent. Debenture Stock	17	8	0			
Interest on Deposit Account at Bank	19	4	9	215	13	11
Journal:—						
Advertisements	600	0	0			
Sales	329	5	8	929	5	8
				£4,901	7	1

EXPENDITURE.

	£	s.	d.	£	s.	d.
Journal Expenses:—						
Publishing	1,478	11	3			
Insurance of Stock	3	15	0			
Editorial	1,168	15	0	2,651	1	3
Printing Sundries				16	14	0
Sectional Expenses				231	18	6
Secretary's Salary (Portion not included in Journal Editorial Expenses)				300	0	0
Expenses in connection with "Annual Meeting"				159	12	6
Honorary Treasurer's Assistant				52	10	0
Office Expenses				118	9	5
Library (Binding Books)				5	2	5
Stationery				29	13	10
Auditors' Fee				10	10	0
"Collective Index"				102	12	0
Bank Charges				3	16	1
Donation to "Pasteur Memorial"				10	10	0
Medals				103	18	6
Secretary's Petty Cash				47	12	7
Treasurer's Petty Cash				16	2	1
Balance of Receipts over Expenditure				1,011	3	11
				£4,901	7	1

THE TREASURER—IN ACCOUNT WITH THE SOCIETY OF CHEMICAL INDUSTRY FOR THE YEAR 1896.

Dr.

	Stock £ s. d.	£	s.	d.	£	s.	d.
To Cash on Deposit (1st January 1896)		300	0	0			
Balance at Bank (1st January 1896)		322	13	10			
Balance in Secretary's hands (1st January 1896)		6	16	5	629	10	3
Annual Subscriptions:—							
1 subscription for the year 1893		1	5	0			
8 subscriptions for the year 1894		10	0	0			
40 subscriptions for the year 1895		49	19	0			
2,588 subscriptions for the year 1896		3,235	0	5			
76 subscriptions for the year 1897		95	0	9			
1 subscription for the year 1898		1	5	0			
2,714							
Sundry Amounts received on account of Subscriptions for 1895, 1896, 1897, and 1898		3	19	1	3,396	9	3
Entrance Fees (214 at 1*l.* 1*s.*, less 6*s.* short paid)		..			224	8	0
Subscriptions on account of the "Collective Index"		..			347	5	0
Life Composition Fees (4 at 20*l.*)		..			80	0	0
Interest on Investments:—							
Metropolitan 3 per cent. Consolidated Stock	(4,476 9 2)	129	16	4			
Midland Railway 4 per cent. Preference Stock	(460 0 0)	17	15	8			
North British Railway 4 per cent. Lien Stock (converted into 3 per cent. Consolidated Lien Stock)	(813 10 0) (1,084 13 4)	31	9	2			
Gas Light and Coke Company's 6 per cent. Debenture Stock	(300 0 0)	17	8	0			
Deposit Account at Bank		19	4	9	215	13	11
Journal:—							
Advertisements		600	0	0			
Sales		340	7	4	940	7	4
					£5,833	13	9

Cr.

	£	s.	d.	£	s.	d.
By Journal Expenses:—						
Publishing	1,467	9	1			
Editorial	1,174	3	3			
Insurance of Stock	3	15	0	2,645	7	4
Sundries, Printing, &c.				45	11	0
Sectional Expenses				268	16	0
Secretary's Salary				300	0	0
Honorary Treasurer's Assistant (for work in 1895)				52	10	0
Stationery				33	3	8
Expenses in connection with "Annual Meeting"				159	12	6
Office Expenses				118	9	5
Library (Binding Books)				5	2	5
Auditors' Fee				10	10	0
Donation to "Pasteur Memorial"				10	10	0
Medals:—						
Cost of Design and Copyright	45	0	0			
Cost of Dies, and supplying Medals, Engraving ditto	58	18	6	103	18	6
Purchase of 654*l.* Great Eastern Railway 4 per cent. Guaranteed Stock				1,000	1	11
Amount paid on account of the compilation of the "Collective Index"				92	2	0
Bank Charges				3	16	1
Treasurer's Petty Cash				16	2	1
Secretary's Petty Cash				47	12	7
Cash on Deposit (31st December 1896)	750	0	0			
Balance at Bank (31st December 1896)	162	17	5			
Balance in Secretary's hands (31st December 1896)	7	10	10	920	8	3
				£5,833	13	9

There are now the following Investments:—Metropolitan 3 per cent. Consolidated Stock, 4,476*l.* 9*s.* 2*d.*; North British Railway Company 4 per cent. Consolidated Lien Stock, 813*l.* 10*s.*; Midland Railway Company 4 per cent. Preference Stock, 460*l.*; Gas Light and Coke Company 6 per cent. Debenture Stock, 300*l.*; Great Eastern Railway Company 4 per cent. Guaranteed Stock, 654*l.*

We have compared the above Statement with the Vouchers, Counterfoils of the Receipts issued, and Books of the Society and certify it to be correct. The amount of the Metropolitan 3 per cent. Consolidated Stock has been confirmed by the Chief Accountant to the Bank of England. Certificates for the remaining investments have been inspected, and the Bankers have verified the Balances upon the Deposit and Current Accounts.

(Signed) MIALL, WILKINS, RANDALL, & Co.,
Chartered Accountants.

23, St. Swithin's Lane, London, E.C.,
19th March 1897.

A Ballot List and Member's Ticket are enclosed herewith.

The Annual General Meeting will be held in Manchester on the 14th, 15th, 16th, and 17th July next.

For Application Forms for Tickets, which should be sent in at once, see May number of the Journal. The Society's headquarters will be the Chemical Club, Victoria Hotel.

Jubilee Honour List.

Her Majesty the Queen has been pleased to confer the dignity of a baronetcy on David Gamble, Esq., C.B.

Her Majesty has also been pleased to confer the honour of knighthood on Prof. Wm. Crookes, F.R.S.

Her Majesty has also approved for promotion to the rank of Knight Commander in the Most Honourable Order of the Bath (Civil Division), Edward Frankland, Esq., F.R.S., M.D.

Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, part of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows:—

To Members (see Rules 25 and 27) and Past Members (see Rule 30); Libraries, Corporations, and Exchanges on the Society's List — Each copy 10*s.*

To Subscribers .... " 12*s.* 6*d.*

To others.......................... " 15*s.*

LIST OF MEMBERS ELECTED 28th JUNE 1897.

Beadle, Alec A., Beadonwell, Belvedere, Kent, Electro Chemist.

Benfey, Dr. Hans, 123, Plymouth Grove, Manchester, Manufacturing Chemist.

Buck, Chas. A., The Bethlehem Iron Co., South Bethlehem, Pa., U.S.A., Chief Chemist.

Enequist, Erik W., 136, Liberty Street, New York, U.S.A., Chemist.

Granger, Dr. J. Darnell, 2, Burlington Lane, Chiswick, W., Analytical Chemist.

Hambly, Fred. J., Gordon's College, Aberdeen, N.B., Lecturer on Chemistry.

Hyde, Benj. T. B., c/o B. F. Babbitt, 82, Washington Street, New York, U.S.A., Soap Manufacturer.

King, Joshua, Clarewood, Camberley, Surrey, Indian Civil Service (retired).

Lloyd, Thos. H., The Laboratory, Penygraig, near Pontypridd, Analytical Chemist.

Motion, Jno., c/o John Ellis and Co., Edgewater, N.J., U.S.A., Oil Refinery Chemist.

Pellew, Chas. E., Columbia University, New York City, U.S.A., Adjunct Professor of Chemistry.

Rae, Roderic H., Edison-Swan United Electric Light Works, Ponder's End, N., Engineer.

Robertson, Jno. R. H., c/o The Darien G.M. Co., Ltd., Messrs. Gabrois and Sons, Cana, *via* Panama, U.S. Colombia, Chemist.

Tucker, Samuel A., Columbia University, New York City, U.S.A., Tutor in Industrial Chemistry.

CHANGES OF ADDRESS.

Banks, A. J., l/o Chancery Lane; The Brewery, Blenheim Road, Hornsey Rise, N.

Bernel, L. A., l/o McCall P.O.; Luling P.O., Louisiana, U.S.A.

Beveridge, Jas., l/o Gravesend; Burnside, Wycombe Marsh, High Wycombe, Bucks.

Bloxam, A. G.; all communications to 16, Bolingbroke Road, West Kensington, W.

Charlier, A. C. J., l/o Glasgow; Glenpark House, Eldon Street, Greenock.

Clayton, R. H., l/o Manchester; 12, Park Avenue, Southport.

Clemes, J. H., l/o Chyan Bryn; Vista, Mount Wise, Newquay, Cornwall.

Cochrane, Jno., l/o Springfield; Watford Bridge, New Mills, *via* Stockport, Calico Printer.

Constable, W. H., l/o Liscard; Australian Alum Works, Runcorn.

Corcoran, Jas., l/o London; 122, Brook Street, Chester.

Exley, A., l/o Meanwood; 13, Woodbine Terrace, Headingley, Leeds.

Foster, R. Le Neve, l/o Droylsden; The Firs, Clayton, Manchester.

Hall, J. W., l/o Bombay; B. B. & C. I. Railway, Sabarmati, near Ahmedabad, India.

Heywood, Jos. H., l/o Manchester Road; 231, Drake Street, Rochdale.

Hibbert, W., l/o 14; 101, Goldhurst Terrace, South Hampstead, N.W.

James, Alf., l/o Glasgow; MacArthur and James, 56, New Broad Street, London, E.C.

Kershaw, J. B. C., l/o Pimlico; 3, Wavertree Road, Streatham Hill, S.W.

Kitchen, Theo., l/o Queen Victoria Street; subscriptions to c/o John H. Kitchen, 60, Watling Street, E.C.

Lucas, B. R., l/o Sandbach; 3, Dyar Terrace, Winnington, Northwich.

MacArthur, J. S., l/o Hope Street; MacArthur and James, 45, Renfield Street, Glasgow.

McCrae, Dr. Jno., l/o Birmingham; 7, Kirklee Gardens, Glasgow, W.

Miller, J. Hopkins, l/o McAslin Street; 5, Catherine Street, Parliamentary Road, Glasgow.

Mitchell, C. A., l/o Chelsea; c/o Beaufoy and Co., South Lambeth Road, S.W.

Moffat-Johnston, J., l/o Midcalder; 57, Dick Place, Edinburgh.

Nelson, H. W., l/o Rue St. Urbain; c/o R. A. Ross, 17, St. John Street, Montreal, P.Q., Canada.

Ormerod, Jno., l/o Littleboro'; Woodland Cottage, Heywood, Lancashire.

O'Sullivan, Jas., l/o Ashby Road; High Bank, Burton-on-Trent.

Parker, C. E., l/o East Orange; 150, Alden Street, Orange, N.J., U.S.A.

Richardson, D. B., l/o Streatham; Tiroran, Pennygbael, Isle of Mull, N.B.

Royal-Dawson, H., l/o New South Wales; 3, Kenilworth Road, Ealing, W.

Sims, Thos. H., l/o Manchester; 61, Earlham Road, Norwich.

Thomson, G. Carruthers, l/o Hillhead; 1, Thornwood Terrace, Partick, Glasgow.

Williams, W. J., l/o Camden; Station F., 2215, Bridge Street, Frankford, Philadelphia, Pa., U.S.A.

Wingham, A., l/o London; Livermead House, Torquay.

MEMBER OMITTED FROM LIST.

Vandenbergh, Dr. Frank P., Erie County Bank Building, Buffalo, N.Y., U.S.A., Professor of Chemistry.

CHANGE OF ADDRESS REQUIRED.

Holland, Jos., l o 70, Higher Ardwick, Manchester.

Deaths.

Harris, Dr. Sydney J., School of Science, Rossington Street, Leeds.

Hart, Peter, Gransmoor Avenue, Fairfield, Manchester. May 30.

London Section.

THE CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE, W.

Chairman: R. Messel.
Vice-Chairman: Boverton Redwood.
Committee:

B. Blount.	H. de Mosenthal.
C. F. Cross.	B. E. R. Newlands.
W. J. Dibdin.	*S. Rideal.*
R. J. Friswell.	*F. G. Adair Roberts.*
S. Hall.	F. Napier Sutton.
D. Howard.	Wm. Thorp.
E. Grant Hooper.	T. Tyrer.

Hon. Local Secretary:
John Heron, 110, Fenchurch Street, E.C.

The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Committee*: E. J. Bevan, Otto Hehner, J. B. Knight, A. R. Ling, and F. B. Power.

SESSION 1896–97.

Meeting held on Monday, June 14th, 1897.

DR. R. MESSEL IN THE CHAIR.

NOTE ON A POSSIBLE SOURCE OF DANGER OF FIRE DURING THE TRANSPORT OF BARIUM PEROXIDE.

BY A. DUPRÉ, PH.D., F.R.S.

Two years ago I had the honour of bringing to the notice of this Section the danger of fire involved in the transport of sodium peroxide, owing to its violent action on combustible substances in the presence of water. To-day I wish to bring before you a danger of fire which may be involved in the transport of barium peroxide.

A short time ago I was consulted by the Solicitors' Department of the Board of Trade as to the probable cause of a fire which had broken out on board the steamship "Rialto," an iron cargo steamer, which led to the loss of the vessel. On looking over the list of articles comprising the cargo I did not find any materials which were known to be liable to spontaneous ignition.

The cargo consisted chiefly of caustic soda, old rope, soda crystals, firebricks, cement, soda ash, hyposulphite of soda, bleaching powder, and 5 tons 12 cwt. of so-called barium. The vessel left the Tyne on February 21st, and almost at once encountered very bad weather. At 6 a.m., March 2nd, two men went into the 'tween decks, above the hold in which the fire subsequently broke out, to secure something which had broken loose. They had no naked light and struck no match while they were below, and noticed nothing unusual. At 5 a.m. on March 3rd signs of fire in the fore part of the ship were noticed, at 9.30 a violent explosion took place, and on March 5th the ship was abandoned. Nearly all the time a heavy gale had been blowing. At first I was inclined to suspect the bleaching powder, as this material had not only been present in several previous cases of fire in which the cause of the outbreak had remained undiscovered, but several accidents ascribed to the action of bleaching powder have been brought to the notice of the Explosives Department of the Home Office, in one of which the coming together of bleaching powder and methylated spirit caused a fire. Careful inquiry, as well as experiments, have, however, led me to the conclusion that, in the present case, at any rate, the bleaching powder had nothing to do with the fire.

I should add that, while convinced that bleaching powder by itself would not be liable to cause a fire, the accident mentioned above shows that there may be danger of its causing fire if it becomes mixed with other substances. In the present case no such substances were present.

My attention was next given to the so-called barium, which, on inquiry, I ascertained to have been barium peroxide. Remembering my former experience with sodium peroxide, I started with some experiments similar to those made in the former case, but soon found that—at any rate, at ordinary temperature—water had no sensible effect on mixtures of barium peroxide with combustible substances. I next subjected such mixtures to percussion and friction, and found that a mixture of wood meal and barium peroxide could be set on fire either when placed on a steel anvil and a steel weight of two pounds allowed to fall upon it from a height of 40 inches, or, when placed on wood, it was struck a glancing blow with a broomstick. To such a blow the mixture proved indeed far more sensitive than any explosive at present licensed, or likely to be licensed. Proceeding with the experiment it was found that in order to cause fire it was not necessary to mix the peroxide and combustible, but that, by merely placing the peroxide on a deal board and striking it a glancing blow with a broomstick the surface of the wood could be set on fire. Finally it was found that on putting some of the peroxide on a piece of wood and drawing another piece of wood rapidly across it under moderate pressure, one stroke was frequently sufficient to set the surface of the wood on fire. With small portions of peroxide the wood takes fire only along the line of friction, and the flame is soon extinguished; when, however, the peroxide is in larger quantity, and particularly when some of it has been rubbed into the grain of wood, the fire not unfrequently assumes considerable proportions, and, under favourable conditions, would undoubtedly spread readily. Here, then, we have a serious danger, not, I believe, previously recognised, involved in the transport of barium peroxide. It is obviously necessary to pack and stow the peroxide in such a manner as to render it practically impossible for it to come into contact with combustible substances under conditions in which it would be liable to friction.

I should add that sodium peroxide is subject to the same danger, but that I have not succeeded, with the primitive tools mentioned, to set fire to wood by using lead peroxide, red lead, or manganese peroxide in place of the barium peroxide.

Since writing the above I have attended the inquiry into the cause of the fire on board the "Rialto," held at Hull from May 27th to June 1st. From what I learned then it is but fair to state at once that it was, in the first place, clearly proved that the barium peroxide on board the vessel, like all the peroxide shipped for export by that firm, was packed in new well-made oak casks, at any rate strong enough to bear all ordinary risks; and in the second place it was by no means clearly established that barium peroxide was stowed in the hold, in which, almost certainly, the fire originated. This particular fire, therefore, cannot be said to have been traced to the presence of the barium peroxide, but, nevertheless, the possibility of a fire originating from the presence of this material amongst a cargo, owing to its extreme sensitiveness to friction on wood, remains of course unaffected.

During the inquiry Dr. Attfield and Prof. Lewes gave evidence on behalf of the manufacturers of the barium

peroxide, with much, though not all, of which I heartily agree. I must, however, take this earliest opportunity of recording my most emphatic dissent from a portion of it. Both these gentlemen stated that, in their opinion, no danger, or, as subsequently somewhat qualified by Dr. Attfield, no practical danger, would arise even should some of the peroxide escape from the casks and become scattered on the deck, between the casks, or between the wood used for dunnage. I am, on the contrary, most strongly of opinion that should any of the peroxide escape from the casks a fire is extremely likely to break out when the vessel is labouring in a gale. As long, however, as the peroxide remains inside the casks it is a perfectly harmless material.

In conclusion I should like to be allowed to make two suggestions. In the first place, this Section might, I think, do a considerable service to the shipping interest of the country by devoting one or two of its meetings to a discussion of the probable cause of fires on board ships and the best means for their prevention. As the causes are frequently due to some chemical action, this Society would be highly competent to undertake such a discussion. In the second place, it seems to me that the way in which expert evidence is dealt with in our law courts and other places is, to say the least, not satisfactory, and I have often thought that if the evidence given could afterwards be discussed by impartial and competent persons much good might be done in checking, what I may perhaps call, the exuberance of fancy indulged in by some witnesses. The Society of Chemical Industry is large and powerful enough to undertake such a task without fear of consequences; and I hope my suggestion may be taken into serious consideration.

Discussion.

Mr. T. Tyrer had had considerable experience with barium peroxide, but had never had or heard of a conflagration caused by this body, and he was by no means clear that the accident which had given rise to Dr. Dupré's paper was due to it. If his opinion had been asked as to the safety of barium peroxide for export, he would have said that it was safer than coals, and, proper conditions being observed, he would even now express the same opinion. Referring to the wider question touched on by the author, and the position which he suggested the Society should take with respect to it, he would point out that the utmost care should be exercised, in any discussion which might consequently arise, not to unduly increase the fears of carriers of chemicals. The keenness of present-day competition with our foreign neighbours was to a great extent due to the fact that foreigners had greater latitude than their British competitors. Chemicals could be shipped from Hamburg without question which in England would not be received without great difficulty or a guarantee of safety; and there was a daily growing indisposition on the part of shippers to accept the word of even experienced manufacturers as to the safety of chemical exports. There was no doubt that this difficulty was increased by the action of insurance companies and Government departments, taken no doubt with the best intentions in the interest of the public.

Dr. J. Attfield admitted the correctness of Dr. Dupré's statements and the views he had expressed as to the liability of barium peroxide to promote combustion. So far as he understood the properties of this body and the precautions taken by those who shipped it, he still considered that there was very slight practical danger even if the powder did leak out in transit. Dr. Dupré had performed an experiment in which he used a weapon partly consisting of hard wood which had been a good deal scored at the end. This weapon he had rubbed sharply across a rough piece of wood in such a way that the grains of the two woods were at right angles to each other, the barium peroxide between them being in a very dry condition. He did not think that these conditions would be realised under ordinary circumstances, and even under such conditions he had never himself succeeded—and he questioned whether Dr. Dupré had succeeded—in producing a fairly continuous fire such as would be likely to lead to a conflagration on board ship. In all else that Dr. Dupré had said respecting barium peroxide he entirely concurred. He could not help thinking, however, that the suggestion which the author had made respecting expert witnesses generally, if acted on, might lead to very voluminous discussions; nevertheless he agreed that much good might result in some cases from turning the Society into a sort of court of appeal with respect to scientific evidence. He was inclined to go even further than Dr. Dupré with respect to regulations for the packing and transmission of dangerous goods, though he would not do anything that should interfere to any considerable extent with trade, especially with a trade which was in the hands of a small number of men who were well acquainted with its collateral relationships. He was of opinion that the present regulations with respect to "dangerous" goods on board ship created more danger than they prevented, and he would urge that there should be either more stringent regulations or none at all. For his own part he would be in favour of an Act, if it were acceptable to manufacturers and ship-owners as well as shippers, under which certain questionable goods should be labelled C, F, E, or D, according as they were combustibles, fire-feeders, explosives, or generally dangerous.

Mr. E. J. Millard inquired what the actual temperature was at which ignition of barium peroxide occurred.

Mr. E. J. Bevan said that he had made some experiments on the ignition of barium peroxide by heating that body in a powdered state, together with disintegrated wood pulp, in a test tube. The mixture, when heated slightly above the boiling point of water, ignited and burned fiercely.

Dr. Dupré, in reply to Mr. Millard's question, said that it was known that wood began to char at about 400° F., and as he could not produce any charring effect in this way it was obvious that the friction produced in his experiment did not reach that point. He would point out to Mr. Tyrer that he had expressly stated in his paper that he did *not* trace the fire referred to to barium peroxide; but that did not do away with the possibility of danger, which he thought he had proved to exist. Whenever proposals were made to deal with public risks, objections were raised such as Mr. Tyrer suggested. In his view, however, no such objection should stand in the way. It was the unknown danger which led to disaster; a known danger could generally be guarded against. He would not be true to the position he held if he failed to state publicly that he considered the matter dealt with in his paper a source of serious danger. The fact that no accident had yet been proved to be due to barium peroxide was not a very powerful argument; for the same thing might have been said some years ago about picric acid, which was manufactured on a large scale for over half a century and was shipped to every part of the world in utter ignorance of its highly explosive character: now, after several very serious accidents, it was known as one of our most powerful explosives. Referring to the remarks which had been made about the regulations under the Explosives Act, he would point out that whereas, in England, the authorities were required to prove their case in an open court of law before they could take any action against manufacturers or shippers, in Germany, where there were fewer regulations, the police had the power to go into a factory and, on their own authority, order goods with which they were dissatisfied to be summarily destroyed. Surely even strict regulations, carefully guarded against abuse, were better than such arbitrary power. When the Explosives Act was first introduced there was a great outcry among the manufacturers that it would ruin the trade. The trade, however, flourished more than ever, and now, he believed, most manufacturers gladly acknowledged the benefits conferred by the Act.

Prof. Attfield was quite mistaken in saying that the friction must be across the grain; he could quite easily set fire to the wood and barium peroxide by friction along the grain. (*Experiment shown.*)

The Chairman thought that, considering the responsible position he held, Dr. Dupré had by no means overstated his case. On the other hand, it was not surprising that manufacturers, menaced with foreign competition and harassed by legislative restrictions, should make a stand in their own interest. The suggestion made by Dr. Dupré was on all fours with the very first paper read before the Society by himself. He thought it very appropriate that matters which

caused dissension in the commercial world should be discussed impartially by practical members of the Society, and he felt certain that if this course were taken up in the spirit suggested by Dr. Dupré, it would open a great field of usefulness to the Society, and be advantageous both to manufacturers and to the public officials who had to deal with such matters.

ON THE VALUATION OF COMMERCIAL NITRATE OF SODA.

BY DR. H. PAULI.

Considering the large sums of money spent in the purchase and sale of nitrate of soda, it is unintelligible that an antiquated method for its analysis is still used to a considerable extent. Instead of estimating directly the most important constituent, nitric acid, the impurities, chiefly water and compounds of chlorine (calculated as sodium chloride), insoluble matter, calcium, magnesium, and sulphuric acid, are determined, and their sum subtracted from 100, the resulting difference being regarded as the correct expression for the percentage of sodium nitrate. This is, to say the least of it, a very unreliable process. Chemical industry requires, in these days of competition, exact methods of analysis, and ought only to admit such a method of refraction, or estimation by difference, when direct estimation is quite impossible. The consideration that all the inevitable errors in the other estimations, influence the percentage of sodium nitrate found by difference alone serves to justify this contention. Above all, the presence of potassium nitrate in Chili nitrate of soda, in varying quantities (in one case about 6 per cent. was found), renders it highly desirable that the percentage of nitric acid shall be determined by experiment, and not by such indirect calculation as that just referred to.

If a sample of Chili nitrate of soda contain potassium nitrate, and not sodium nitrate only, as is assumed in the method of difference, it is clear that, corresponding to the higher atomic weight of potassium, proportionately less nitric acid should be found present than the result obtained by difference would indicate. Thus the method of refraction is deceptive, and the loss falls upon the purchaser.

The percentage of potassium ought to be determined directly by repeatedly evaporating a weighed quantity of the nitrate to dryness with concentrated hydrochloric acid, and subsequently precipitating an aliquot part with platinic chloride.

Agreed as to the urgent desirability of abandoning the method of "refraction" in favour of a direct method, the remaining question is, What method is most suitable for the direct estimation of nitric acid?

For very frequent determinations, the use of Lunge's nitrometer for commercial nitrate of soda may be employed, but in this case the apparatus must always be in order, and even then much depends on the individual skill of the worker. The quartz method of Reich is considered more suitable. According to this method, in which the nitrate of soda, first thoroughly dried at 130° C., is heated to dull redness with excess of pure silica in a platinum crucible, the nitric acid is expelled and determined by the loss in weight. We, in the Hoechst laboratory, have found by repeated titrations, before and after equally long heating, that the chlorides present, suffer no appreciable volatilisation. Latterly perchloric acid has been found in nitrate of soda, probably present as potassium perchlorate, and in one case upwards of 6 per cent. Since then we have often determined the perchloric acid quantitatively, and have found sometimes none and sometimes from 0·1 to 1·3 per cent.

It is only necessary to make two chlorine estimations of the same sample of nitrate before heating, and after heating with a little caustic soda, by Volhard's method of titration, and to calculate the difference as perchloric acid, subtracting the loss in weight, due to the evolution of oxygen from the perchlorate, from the loss of weight obtained by heating with quartz. By the present refraction method of analysis all perchlorates are estimated as nitrate of soda.

From the above, it is evident that analytical chemists are quite able to determine the percentage of nitric acid in commercial nitrate of soda directly and with accuracy in all cases, and they should no longer hesitate to reject the old false method of determination of sodium nitrate by difference as being unworthy of the present state of chemical science.

(Fresenius, in his classical work on Quantitative Analysis, expresses the opinion that Reich's silica method is the simplest for the purpose, besides possessing all the necessary accuracy. We (Meister, Lucius, and Co.) simultaneously employed this method some years ago, in an analytical dispute, for a third party, in which Fresenius and we were referred to as independent experts.)

The presence of potassium nitrate has no influence on the accuracy of the results obtained by the silica method, but the contrary is the case with the indirect method of refraction.

Assuming that we had a nitrate containing 3 per cent. of potassium nitrate, and that 4·3 per cent. of other ordinary impurities had been found, we should obtain the erroneous result of 95·7 per cent. by the indirect method, whilst the correct result of the silica method gives 95·21 per cent. when calculated as sodium nitrate, *i.e.*, almost ½ per cent. less.

As regards the influence of potassium perchlorate on the correctness of the results in the case of the indirect method, the result obtained is too high by the percentage of perchlorate present, whilst in the case of the direct method the result is too high by the amount of oxygen evolved from the perchlorate on ignition, *i.e.*, a smaller error than that of the indirect method. If 0·4 per cent. of perchlorate of potassium be present, only 0·28 per cent. too much nitrate of soda is found by the silica method, if one does not (as is now always done), subtract the loss in weight due to evolution of oxygen, calculated from the perchlorate estimation, from the total loss in weight, thus excluding the whole error. [In order to prove that the chlorine compound present was perchloric and not chloric acid in combination, we precipitated the chlorides in commercial nitrate of soda with the quantity of silver nitrate calculated from the chlorine titration, added sufficient concentrated sulphuric acid to the filtrate from the silver chloride, and passed sulphuretted hydrogen into the liquid, which was kept as concentrated as possible from the beginning, until all the nitric acid was decomposed. Chloric acid would, in such a case, be reduced to hydrochloric acid, which, after the removal of the excess of sulphuretted hydrogen with ferric sulphate, would give a precipitate of silver chloride on addition of silver nitrate. This, however, was not the case, but the perchloric acid, which is unchanged by sulphuretted hydrogen, gave, after treatment with excess of caustic soda and subsequent ignition, a precipitate of silver chloride with silver solution.]

See also this Journal, 1897, 163—164, *Detection and Estimation of Perchlorates in Chili Nitrate of Soda.* Also, *ibid.* 358, *Estimation of Perchlorate in Saltpetre.* Also Lunge's *Sulphuric Acid and Alkali* (1891), Vol. I., 78.

Discussion.

Mr. Otto Hehner said that the name of Dr. Fresenius having been mentioned, he would not like this occasion to pass without expressing his very deep regret at his death, which had occurred a few days ago. Fresenius had always been to him an excellent friend, and it was perhaps only right that he, as his old pupil, should remind his fellow-members of what they owed to his memory, and the immense influence which his work had had upon the progress of manufacturing chemistry. If Fresenius was not associated with any of the great discoveries which had revolutionised physical chemistry during the last and present generation, it was nevertheless true that no name was more of a household word than was his, not only on account of the books he had written, but of the numerous exact and beautiful methods which he had devised, and which were now in daily use, and without which they could hardly conceive the carrying on of ordinary analytical work. Referring to the paper, he could heartily agree with Dr. Pauli. The accepted method of valuing nitrate of soda was simply an anachronism. It might have been justifiable years ago, when methods for the direct determination of nitric acid

presented difficulties, but it seemed to him absurd at the present time to take into account everything else except the very thing which one was searching for. Anachronisms proverbially died hard, and it would be perhaps most fitting that the last nail in the coffin of this particular one should be driven in by the Society of Chemical Industry.

Mr. T. Tyrer wished to support Mr. Hehner in his admiration of the work of his old master, the late Prof. Fresenius. With regard to the subject of Dr. Pauli's paper, he could quite agree with Mr. Hehner that it was absurd to call upon people to pay for what they did not get. If manufacturers paid for nitric acid, they should get it. If the nitrate contained impurities which affected its value in nitric acid, a method of analysis should be used which would give the facts. He was speaking with special regard to the use of nitrate of soda in the enormous industry of sulphuric acid manufacture, and, in that light, Dr. Pauli's paper was one of great importance. If the Chairman saw fit to bring the matter before the Council, he would have great pleasure in supporting him.

Mr. J. Hughes had expected to hear evidence of the existence of dissatisfaction with the present method of valuation of nitrate of soda, but absolutely no figures had been put before them to prove that that was the case. The Permanent Nitrate Committee was known to be an energetic body, and if it were shown that they were giving nitrate of potash under the name of nitrate of soda they would immediately alter their procedure. He had had occasion to examine a great number of samples, and had never had any complaint of the method used, which, though an indirect one, had the advantage of giving the respective proportions of the impurities in the form of chlorides and sulphates of soda, the exact figures for which were of great importance for manufacturing purposes.

Mr. W. Crowder observed that statements had been made that quantities of iodine were sometimes found in shipments of nitrate of soda, either in the form of iodide of sodium or iodide of potassium, and asked whether this was a fact.

Mr. W. F. Reid wished to support the Chairman's suggestion that the subject of the paper should be referred to the Council, with a view to action in the matter being taken. He regarded the Society as an eminently fit and proper body to take such action, as in it both sides were represented. He would like to inquire also whether Dr. Pauli had made experiments with nitrate of soda containing sulphates as impurities. Part of the sulphuric acid would probably be volatilised on heating with silica.

The Chairman reminded Mr. Hughes that the fact that many papers had already appeared on this subject, and with the same object, might be taken as *prima facie* evidence of the existence of dissatisfaction with the present method of valuation. Something should be done, and he thought the proper course was to ask the Council to appoint a committee to consider the matter in an unbiassed manner. If, therefore, he had the consent of the meeting, he would put the matter before the Council in that sense.

The Chairman's motion, seconded by Mr. Tyrer, was then put to the meeting, and was carried *nem. con.*

SOME RECENT IMPROVEMENTS IN SMOKELESS POWDER COMPOUNDS AND IN PROCESSES OF MANUFACTURE.

BY HUDSON MAXIM.

Explosive compounds are burned in two ways: one by combustion from surfaces exposed to a consuming flame, the other by what is called detonation, by which the explosive is consumed nearly simultaneously throughout its mass by a wave action. Combustion from surfaces, requiring an appreciable time for the consumption of the explosive body, adapts it to use as a gunpowder. The detonative form of explosion adapts a compound to disruptive or shattering purposes, and unfits it as a gunpowder. However, the function of an explosive, whether as a combustive or a detonative compound, is very largely determined by its physical condition, temperature, and the conditions of confinement under which it is fired. The equivalent of these conditions is produced when a sufficiently large quantity is fired, and also by the force and temperature brought to bear upon a less sensitive compound by another and highly detonative explosive.

It is well known that guncotton can be ignited in considerable quantity, and burned quietly away without detonation. Nitroglycerin in small quantities may be burned like an oil, and even fulminate of mercury may be burned in a vacuum without detonation. Nitrogelatin, the most powerful commercial high explosive known, may even be used as a propellant or gunpowder, in small quantities and under light confinement.

Fibrous guncotton, which consists of an infinite number of small tubes, whose walls, although of a dense colloid, are very thin, owes the facility with which it can be detonated to the fine state of division of this colloid. If guncotton be dissolved in acetone, poured on a plate of glass, and dried, it is well known how the hard and horn-like product resists detonative influences. However, if reduced to a fine powder, its susceptibility to detonation is restored, or if redissolved in acetone, and poured into water in a fine stream, a fluffy, fibrous material is formed, resembling very closely the original fibrous guncotton, and which will detonate with equal ease and violence.

As practically all high explosives may, under suitable conditions, be consumed by surface combustion, similarly may all explosives, under suitable conditions, be detonated. The harder and more coherent the body, the less rapidly it will burn by surface combustion, and the more difficult it is of detonation.

However, the hard and dense colloid of guncotton burns much too slowly, and through too small a thickness of material in large guns under service conditions, to yield a sufficient quantity of gases to produce requisite velocities, without such fine granulation as to expose such a large amount of initial burning area as to exceed permissible pressures, thereby preventing the use of full charges. For this reason, no pure guncotton powder, except in the form of the Maxim-Schüpphaus multi-perforated grains, has yet been produced which has been successful in guns of more than 4- or 5-in. calibre. However, for rifles, shot-guns, and small quick-firing guns, very good results have been obtained, although still higher results are readily obtainable in small cannon powders by multi-perforating the grains.

By our system of multi-perforated grains, a pure guncotton powder may be made which will produce exceedingly high ballistics in guns of the largest calibre.

Another method of meeting the difficulty is to add some other substance which will quicken the combustion of the compound, such as nitroglycerin, which softens the guncotton colloid and causes it to burn with much greater rapidity, effecting the consumption through a sufficient thickness of material to permit of such coarse granulation, with such lessened initial combustion area, as to enable the use of full charges. The best representative of such high percentage nitroglycerin powders is British cordite.

Nevertheless, if the cordite compound be made into grains or strips of the proper size and shape, and suitably multi-perforated by our system, its ballistics are correspondingly improved. We have conducted a large number of experiments with this and similar high-percentage nitroglycerin compounds, with the result that the improved ballistics, above the usual form of solid rods, corresponded with and verified our calculations of the advantages of the perforations.

The exceedingly high temperature of the products of combustion of nitroglycerin is an important factor in the production of the ballistics attainable with high-percentage nitroglycerin powders, but the excessive heating and erosive action on the gun of these powders, together with other objections too well-known to require discussion here, makes it preferable to employ a pure guncotton colloid, or one containing a small percentage of nitroglycerin, provided it can be put into such physical shape as to produce equally high ballistic results.

The hard colloid of guncotton is also modified and caused to burn with somewhat greater rapidity, by digesting it with the smallest possible quantity of solvent, to render

it sufficiently plastic to be stuffed through a die or rolled into sheets. The product, although resembling very closely the colloid formed by a complete solution of the guncotton, still is not so glassy and hard, and will burn through a greater thickness under like conditions. There is another method for effecting a similar result by forming a compound consisting of undissolved fibrous trinitrocellulose agglutinated by a collodion made from a more soluble variety of pyroxylin.

A method patented by us for producing a smokeless powder of this character is substantially as follows:—Trinitrocellulose is made into paper or a thin pulp board, which is subjected to a bath made of soluble pyroxlin dissolved in a solvent which is not a solvent of the trinitrocellulose. In other words, the guncotton paper is sized in a collodion bath, which bath may or may not contain nitroglycerin; the excess of the bath being removed, sheets of the material are placed upon one another and pressed together, whereby a body of any desired thickness is built up. The mass is then placed in a suitable die or mould and multi-perforated in a manner explained further on. The hardness and density of the product may be varied within wide limits by varying the consistence of the bath or the amount of size left in the paper, and also by varying the compression. The material may then be cut into strips approximating in length the powder chamber of the gun, or any length desired. Before being perforated, a sheet of unnitrated paper may be affixed to two surfaces of the body, whereby they will be, to a great extent, protected from ignition during the early stages of combustion in the gun, thus causing a greater degree of combustion to take place within the perforations. Or, the body of material may be multi-perforated and dried, and then coated with any suitable substance which will delay ignition of the surface.

There is one thing which we know to a certainty, and that is, that the shape of an explosive grain is not as it should be when it presents the maximum of combustion area to the flame of ignition, and is consumed with a constantly decreasing burning surface. The configuration of the grain should be such as to present an initial area which, with a given density of loading and weight of projectile, shall produce a desired pressure, and burn with such increase of surface and evolution of gases as shall maintain the pressure equally behind the projectile in its flight throughout the entire length of the gun. In other words, the burning surface should be regulated to the demands upon it for the evolution of gases. The control of its combustion is the great desideratum in a gunpowder. The others are smokelessness, stability, minimum effect upon the gun and gunners, and practicability and cost of its manufacture on a large scale.

The old black and brown powders, being but compressed mechanical mixtures, were unsuited to that special form of grain consistent with the production of the highest ballistic results. With the introduction of smokeless powders made of a dense and tenacious colloid of guncotton, or of guncotton and nitroglycerin, it became possible, by a special system of multi-perforations, to produce a grain which will burn in the desired manner, although this result has not been easy of attainment. Trinitrocellulose requires a large quantity of solvent, not only to dissolve it, but even to render it plastic; and rods or grains formed of it are exceedingly difficult to dry without warping and cracking to pieces; and it is difficult to make it take and retain the exact shape of the forming die. Consequently, unless a very large percentage of nitroglycerin be employed, a composition must be produced which allows working in a much drier state than ever heretofore attempted, in order that the plastic mass shall retain the shape given it by the die in all its geometrical details. All this calls for special tools, special processes, and a special compound.

There are two smokeless powder compounds employed by us, the one consisting of a compound of nitroglycerin and mixed guncottons, the other of mixed guncottons without nitroglycerin. The first is made by mixing together, in a kneading machine, at a temperature of about 120° F., 80 lb. of trinitrocellulose of about $13\frac{4}{10}$ per cent. nitrogen, with 8 lb. of gelatin-pyroxylin of 12 per cent. nitrogen (and soluble in nitroglycerin below 100° F.), 12 lb. of nitroglycerin, and 35 lb of pure anhydrous acetone, adding thereto 1 lb. of pure urea dissolved in pure methyl alcohol. The mixture is worked in the kneading machine for about an hour at a temperature of about 120° F., heated by means of a water-bath. The mealy mass is then passed between cold rolls and formed into rough sheets, which are then converted into smooth sheets by passing between slightly warmed rolls. The proper consistence for the mass is easily recognised by the experienced workman by the sense of touch, but the amount of solvent retained may also be easily ascertained by weighing from time to time. Only from 15 to 20 per cent. of solvent should be retained when the compound is ready for stuffing or moulding into grains.

The smooth sheets are compactly wound about one another to form a roll of the diameter of the press cylinder, the ends preferably being trimmed. The press is kept warm by means of a water jacket, having a temperature of about 120° F. The press filled, the cylinder is preferably exhausted of the air within the spaces unfilled by the powder. This process, though not absolutely essential, is advisable. The mass is then pressed hard against the head to solidify it. The forming die is now affixed, and the compound forced through it at a pressure varying from 3,000 to 4,000 lb. to the square inch, emerging in the shape of multi-perforated cylinders. The smaller the grain, the softer should be the material and the higher the pressure to force it out. The rods are cut into grains about 3 diameters long, and placed on shelves in a drying room. When partially dried, the drying is finished in a vacuum.

Several years of experience have shown that, after this treatment, a certain trace of solvent is retained, but it is a constant for each size of grain, and no further drying or other change whatever takes place under service conditions.

It hardly needs mention that the pyroxylins and nitroglycerin should possess the highest stability, and that all the other materials entering into the composition should be of the highest standards of purity.

The nitroglycerin may be entirely dispensed with, as I have said, and a pure guncotton composition be manufactured after essentially the same method. In this case, we prefer to use about 80 parts trinitrocellulose, and $19\frac{1}{2}$ parts gelatin-pyroxylin, and $\frac{1}{2}$ per cent. urea. Owing to the absence of nitroglycerin, which facilitates the moulding operation, slightly more solvent is left in the material before pressing. As I have said, it is impossible to evaporate the last trace of solvent from the compound, but, as the quantity remains for ever constant, and never escapes, it does no harm.

We placed 100 lb. of this powder in a room heated to a temperature of 125° F. for two weeks, without showing the least loss of weight. Some of this powder was then pulverised and exposed to the same temperature, when the remaining solvent was driven off. Whatever theories and preconceived opinions may be, the true test of a cannon powder is the test of time and exposure under service conditions.

At the Sandy Hook proving grounds, in the United States, a quantity of this powder was thrown loose upon the floor in an open room, and there exposed, for a period of three years, to the heat of summer and the cold of winter. At the same time, an equal quantity was filled into cartridges and kept sealed up for an equal period. Some of the loose powder was then filled into cartridges and fired side by side, in alternating shots, with that which had been kept sealed, with the result that the ballistics of that which had been exposed and that which had been sealed up were identical, proving that the powder had undergone no change whatever.

These powders can be produced on a large scale, speedily, economically, and, as the experience of years has proved, they can be produced of uniform composition. The machinery, tools, and processes have all proved practicable and workable commercially.

In view of the peculiar treatment to which our powder is subjected in the process of manufacture, we prefer to add a small quantity of urea—the ideal substance to counteract any possible slight local decomposition, such as has been quite recently again pointed out by Mr. Guttmann

in his able paper on the stability of nitro-compounds (this Journal, 1897, 284).

The peculiar influence of urea is well illustrated by its use in the manufacture of transparent celluloid, such as photographic films, which—in case they are cut from a block of compressed material, and not made by flowing a comparatively thin solution on glass plates, or by an equivalent method—could not be produced without the use of this substance, as the temperature to which it is necessary to subject the material causes such slight decomposition as to discolour the product. Urea counteracts this by neutralising the nitrous acid as fast as formed. Urea has the further advantage above any other neutralising substance, in that it is decomposed by nitrous acid into carbonic acid gas, water, and nitrogen, leaving no solid product in the material, while it is not an active alkali and its presence has no effect whatever upon the nitro-compounds with which it is combined.

When soluble pyroxylin is combined with trinitrocellulose in suitable proportions, it has the peculiar effect of rendering the compound plastic, and enabling it to be moulded with very much less solvent, under the influence of an elevated temperature. This peculiar effect resembles that produced by camphor combined with celluloid pyroxylin. It furthermore renders the final product more tenacious and less likely to crack.

I will now call your attention to our various forms of multi-perforated grains. The figures shown in the drawing are *four times* natural size. Fig. 1 represents a bar or strip of the powder compound transversely perforated with

Fig. 1.

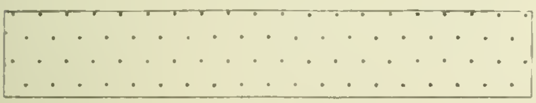
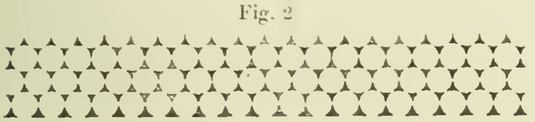


Fig. 2



round holes. Fig. 2 shows the same strip nearly consumed, and illustrates the increased burning area produced by the enlargement of the perforations. It will be observed that at the instant of interjunction of the perforations, there remains a portion of the powder unburned. Figs. 3, 4, 5, 6, 7, and 8 represent strips of powder perforated with

Fig. 3.

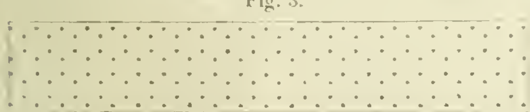
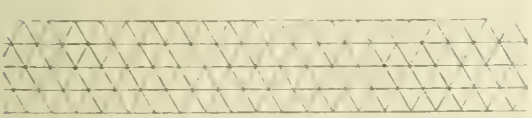


Fig. 4



angular holes of such shape, and so arranged with respect to one another, as to effect the complete consumption of the entire body of explosive material simultaneously with the interjunction of the perforations. These rods or strips may be made of any desired length—as, for example, the full length of the powder chamber of the gun.

Fig. 5.

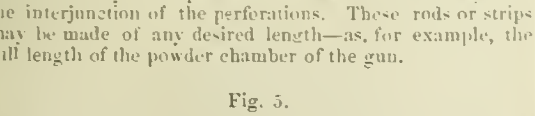


Fig. 6.

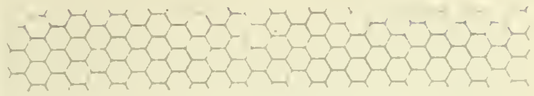


Fig. 7.

Fig. 8.

The remaining figures represent rods or grains of powder longitudinally perforated with round and with various angular perforations, whereby a similar result is effected as that just explained with reference to the other figures.

Fig. 9, perforated with round holes, leaves a small body of unconsumed material, shown in Fig. 10, representing

Fig. 9.

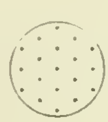


Fig. 10.

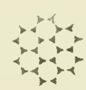
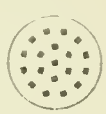


Fig. 11.



the same grain nearly consumed. In the remaining figures the powder grain is wholly consumed simultaneously with

Fig. 12.

Fig. 13.

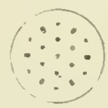


Fig. 14.

the interjunction of the perforations with one another, illustrated by the nearly consumed grains shown in Figs. 12 and 14.

A peculiarity of smokeless powder in its combustion is that the character or shape of a perforation is maintained. This is due to the more rapid combustion in the corners or angles than upon the surfaces lying farther apart, owing to the closer confinement of the gases there evolved.

I think I was the first to invent a powder grain having angular perforations for producing unequal combustion, whereby complete combustion of the grain is effected simultaneously with the interjunction of the perforations.

Fig. 11 represents a grain which is probably the preferable form of perforation for longitudinally perforated powder. This particular form of grain was independently invented by Mr. Fred. H. McGhaie, of Brooklyn, New York, and by myself.

To verify the claims for the particular form of combustion secured by these methods of perforating the powder, I have samples which I shall be glad to show, at the close of these remarks, to anyone wishing to examine them. They exhibit grains of powder at different stages of consumption, demonstrating that our theories are warranted in practice.

We have conducted a larger number of experiments to prevent or retard ignition of exposed surfaces of grains and strips of powder, in order to confine, as far as practicable, the combustion within the perforations. This may be effected by covering the powder with a layer of unnitrated

paper, coating the powder with varnish, or by denitrating the surface of the material, or by coating the material with a layer of non-explosive celluloid. We have found that that form of coating works best which retards ignition sufficiently to give combustion within the perforations a good start of combustion upon the other surfaces, but which coating will be consumed a little in advance of the interjunction of the perforations.

We are aware that a multi-perforated grain of powder is not novel; but that form of perforated grain whereby the highest ballistic results are secured is novel, and a rod or strip of powder having transverse perforations as an article of manufacture is entirely new.

To produce a grain of powder consisting mainly of guncotton which will take and retain the shape given it by the die in all its geometrical details, it must be moulded while in a condition quite hard and stiff, and should be worked at an elevated temperature so as to rapidly stiffen by lowering the temperature as soon as formed. When making longitudinally perforated grains, the perforations must be small and the mandrels of the die to produce them must be light and slender, and to prevent distortion they must lie exactly in the path of flow of the material, and the material must therefore be made to pass through spaces between the mandrels at their base. This calls for dies of special construction.

For a fuller description of the processes and apparatus employed by us, mentioned in this paper, I beg to refer you to the following British patents of Dr. Robert C. Schüpphaus and myself:—

No. 22,382 of 1893 (this Journal, 1894, 174); No. 18,682 of 1894 (*ibid.*, 1894, 1219); No. 18,683 of 1894 (*ibid.*, 1894, 1220); No. 8569 of 1895 (*ibid.*, 1895, 820); No. 11,299 of 1895 (not published); No. 14,311 of 1895 (this Journal, 1897, 65); No. 16,861 of 1895 (*ibid.*, 1896, 51); No. 16,862 of 1895 (*ibid.*, 1897, 161); No. 3002 of 1897 and No. 7178 of 1897 (not published).

Discussion.

Mr. W. F. Reid said there were a few thoretical points upon which he would like to have further information, for example, Was it found that the perforated pellets showed any tendency to break up in the barrel of the gun? Such pellets of black powder had been used, but it was generally found that they broke down under the enormous strain of the explosion. If the prisms failed to retain their form under all circumstances, the disadvantage of an irregular increase of surface would naturally result. The amount of surface exposed to ignition in the first instance was not the only means of modifying the explosive force; but he had heard nothing from the author of other means to that end, such as a difference in the density of the grain, rendering it more easily inflammable on the outside and harder towards the centre or *vice versâ*. He had adopted that method himself in the case of the E.C. powder, and found it successful. He agreed with the principle advanced by the author, that gas pressures should not be greater as they advanced towards the muzzle. The experiments made in the United States seemed to prove that one could subject the powder to very varying conditions, and the solvent remained at a fixed percentage. But under service conditions in this country it was found that the varying proportion of the remaining solvent was a serious difficulty. Whether that solvent took part in the explosive action was a very material point in the problem. It must be recollected that explosives manufactured for the English Government had to meet the conditions of various climates, some of them very humid, and therefore the presence of a solvent that might evaporate at different temperatures might have a very serious effect on the ballistics of a powder.

Mr. W. MacNab observed that Mr. Maxwell Lyte had about 1868–69 introduced a smokeless powder in France which closely resembled the form described by Mr. Maxim.

Dr. A. Dupré had listened with great interest to the paper, which showed a large amount of mechanical ingenuity. There was, however, one chemical point dealt with, *viz.*, the use of urea. That was a practice which he had always set his face against; it was equivalent to adding, say, boracic acid to milk in order to mask want of cleanliness or of honesty in the milk trade. If a powder were well manufactured it ought to stand without urea or any other masking body. The remarks about making a powder so that it should keep up the same pressure up to the muzzle sounded very well, but practically the idea was an impossible one, for guns could not be made so as to stand such a pressure throughout their length. The pressure should reach its maximum in the chamber, and so give a greater ratio of power for a given weight of explosive. If the guns were as strong at the muzzle as they were at the breech they would be clumsy and unwieldy. Mr. Reid had, if he would allow him to say so, put the cart before the horse. Mr. Maxim, it appeared to him, wanted to produce a powder that burnt more rapidly as combustion proceeded; Mr. Reid desired the reverse. The greater the pressure the more rapid was the combustion, and no peculiar shape of the pellet was needed to ensure this. It was practically impossible to make a powder so that it should just have burned completely as the projectile reached the muzzle and keep up a uniform pressure as well. One might get a greater effect from such a powder, but it would be at the expense of very costly material; and under present circumstances the question of expense was a very important factor.

Mr. Hudson Maxim, in reply, said that smokeless powder was entirely different in this respect to the old black and brown prismatic powders; but even in smokeless powder, if the rods were long and longitudinally perforated, they would be useless, as they would burst in the centre. They were therefore, when perforated lengthwise, made in comparatively short lengths of about three diameters, and when made in long strips they were pierced with transverse perforations of such a length as not to lead to disruption of the material. With regard to rapidity of combustion, the problem was to perforate in such a way as to produce a pressure as nearly as possible uniform up to the muzzle. Other patents were mentioned in the paper, but as the paper had not been read in its entirety they were passed over. He intended to refer only to his own firm's recent improvements; those of others which were already published could hardly be considered new, and if they were not published, he, of course, could not refer to them. With respect to the production of smokeless powder, it should be made with as little solvent as possible and moulded into grains at an elevated temperature, so as to harden by the lowering of the temperature as soon as formed. If not, it would collapse, shrink, and ultimately crack to pieces. They, by the use of gelatin pyroxylin and trinitrocellulose in certain proportions, worked at an elevated temperature, used but little solvent, and thereby overcame these difficulties. He did not claim the pyroxylin paper as an invention, though the patent covered improvements in treating it, building up and covering it. One of his brother's early patents mentioned the making of guncotton into paper, but it was not claimed for lack of novelty. Dr. Dupré had referred to the disadvantages of foreign matter in a powder. For his own part, he did not know of any powder that was free from it. His own firm added about 1 per cent. of urea for safety's sake; if the materials were pure it did no harm, and if the materials should happen to be impure it made the powder stable. The Government put in 5 per cent. of vaselin—a paraffin which had no action on the other ingredients. He must take decided exception to Dr. Dupré's conclusions with regard to the advantages of a powder made to burn with increasing rapidity with consequent maintenance of pressure behind the projectile to the muzzle of the gun. Such a powder certainly had great advantages above one which burnt with a constantly decreasing rapidity. Captain Stewart, of the United States Army, in reporting to the Chief of Ordnance on the Maxim-Schüpphaus powder, said that in this multi-perforated powder the dream of artillerist had been realised. He thought Dr. Dupré had misunderstood Mr. Reid, for there could be no advantage in a powder which would burn more rapidly at first and then more slowly. Any of the present multi-perforated powders burnt more slowly as the pressure fell off in the gun, as all powders burnt with a rapidity in

proportion to the pressure. He could see no disadvantage in maintaining the pressure right up to the end, since with a less initial pressure one got as high a velocity. In the trials by the United States Government, they had obtained a muzzle velocity as high as 2,800 feet per second in a 45-pounder gun, 37 calibres long, with less than 35,000 lb. per sq. in. pressure. Unless one used a nitro-glycerin compound or added some oxygen-bearing salt which produced smoke, one must multi-perforate the powder; otherwise it had to be granulated too finely, and full charges could not be employed without too high pressures.

COMPARATIVE EXPERIMENTS ON THE ESTIMATION OF PHOSPHORIC ACID.

BY ALEXANDER CAMERON, F.C.S.

I HAVE carried out a number of experiments with the view of ascertaining the extent of the discrepancy between the two principal methods of determining phosphoric acid, *viz.*, the molybdate and the citrate methods, and between certain modifications of them. These methods are sketched with some detail at the end of the paper.

The basis of the experiments was tribasic phosphate of lime prepared by adding to a solution of phosphate of ammonia a solution of calcium chloride, the salts being present in their combining proportions. After thorough washing, the precipitated phosphate was dissolved in hydrochloric acid and reprecipitated by the addition of ammonia in slight excess. This precipitate was again filtered, thoroughly washed, dried, separated from the filter paper, and gently ignited. The purity of this phosphate of lime was established by a careful determination of the lime contained in it, it being useless to determine the phosphoric acid present, as the method of estimating that substance was itself under consideration. The estimation of lime was done by precipitating the calcium as calcium oxalate, collecting it on a weighed filter, drying, and weighing as $CaC_2O_4 + H_2O$. Calculating to CaO, in two determinations 53·94 per cent. and 54·03 per cent., or an average of 53·99 per cent., was obtained. In other words, about 99·6 per cent. of the CaO contained in the phosphate, reckoned as $Ca_3P_2O_8$, was recovered; the theoretical amount of lime, as CaO, contained in $Ca_3P_2O_8$ being 54·19 per cent. Now it has been proved when estimating the lime in pure calcium carbonate by the above method of weighing as oxalate, that about 99·5 per cent. of the theoretical quantity of $CaCO_3$ is obtained, which is almost identically proportional to the results obtained in the phosphate of lime here prepared. There is every reason therefore for believing that the calcium phosphate operated upon is pure tribasic phosphate of lime, as represented by the formula $Ca_3P_2O_8$, and the following experiments were based on the purity of this compound. The experiments were also conducted on different samples of superphosphate of lime and mineral phosphates as representing artificial manures. The $Ca_3P_2O_8$ reported is calculated from the P_2O_5 soluble in water in the case of the superphosphates, and from the total P_2O_5 in the case of the other materials.

Estimation by the Citrate Method.

(*a.*) *The Effect of the Addition of Varying Amounts of Citric Acid:—*

In pure $Ca_3P_2O_8$, 0 5-grm. sample.

Citric acid added............ Grm.	0·25	0·50	1·00
$Ca_3P_2O_8$ recovered......... Per cent.	97·82	97·12	96·56

In superphosphate of lime, 1-grm. sample.

Citric acid added .... Grm.	0·50	1·00	2·00
$Ca_3P_2O_8$ recovered. Sample A. Per cent.	25·06	24·71	24·64
$Ca_3P_2O_8$ recovered. Sample B. Per cent.	24·58	24·49	24·49

These results show that the larger the excess of citric acid employed, the smaller is the weight of the precipitate obtained. I am always in the habit of weighing out the quantity of citric acid added, having determined, by previous experiment, what the average minimum quantity is, that is required for the various classes of substances.

On a system suggested by Mr. F. J. Lloyd, and on figures with which I have been supplied, I have drawn out the following table, the adoption of which I recommend. The list may of course be indefinitely extended on the same lines:—

TABLE OF PHOSPHATE FACTORS.

Name of Phosphate.	Composition per Cent.			Amount of Reagents when using 2 grms. of Sample.		
	P_2O_5	Ca O.	$Fe_2O_3 + Al_2O_3$	Mg Mixture.*	Am. Ox.	Citric Acid.
				C.c.	Grm.	Grm.
Algerian	25·03	51·99	0·55			
"	26·95	51·24	0·47	35	3·0	0·3
"	29·51	51·27	0·48			
Belgian	18·37	50·59	1·29			
"	21·06	49·89	1·42	30	2·7	0·5
"	21·61	49·84	1·38			
South Carolina	25·26	42·00	2·71			
"	25·61	42·92	1·77	35	2·5	0·7
"	26·79	43·96	2·22			
French	19·94	53·84	0·36	25		
"	26·41	53·11	0·77	35	3·0	0·5
"	29·22	45·44	4·16			
Florida, pebble	32·39	47·09	4·00	40	2·7	
" "	33·19	48·30	3·12			
" rock	35·38	48·44	3·76	45	2·8	2·0
" "	36·59	51·88	1·77			
" Peace River	27·69	41·36	2·40	35	2·5	

* For composition, see end of paper.

(*b.*) *The Effect of Iron.*—The larger the amount of iron present the more phosphoric acid will be carried down as phosphate of iron (and alumina, if that substance be present) in the lime precipitates. In the pure salt the amount of phosphoric acid found in the lime precipitates was usually but little over 0·1 per cent., while the addition of 3, 6, and 9 per cent. of ferric oxide to a similar sample yielded 0·5, 0·6, and 0·7 per cent. respectively of phosphoric acid in the lime precipitates; hence the necessity of determining the amount of phosphoric acid thus carried down. One-third the weight of such precipitates equals the phosphoric acid present. This I have frequently shown to be the case, as the following results, which are the averages of many closely agreeing experiments, prove.

—	In Pure Salt.	In Super-phosphate.	In Belgian Phosphate.
	Per Cent.	Per Cent.	Per Cent.
Phosphate precipitate......	0·12	0·10	1·03
P_2O_5, as one-third of precipitate.	0·14	0·03	0·34
P_2O_5, by actual determination.	0·14	Trace.	0·32

The addition of 3 per cent. of ferric oxide, other conditions being similar, had no effect on the amount of phosphoric acid obtained by the citrate method, while the addition of 6 and 9 per cent. increased the weight of the total $Mg_2P_2O_7$ precipitate slightly, the results being in the proportion of 100 : 100·39 : 100·79, respectively.

It can safely be concluded that, except in the presence of large amounts of iron (when the molybdate-citrate method should be employed, as described later), the determination of phosphoric acid by the citrate method can be accurately performed.

(*c.*) *The Volume of Solution before Precipitation.*—This consideration is also very important. As a rule, it may be laid down that the greater the volume of the liquid the less will the precipitate recovered weigh. After separation of the lime the liquid should bulk 200 c.c. as nearly as possible in a 250-c.c. beaker. This figure is easily kept constant by the use of a certain sized beaker, the capacity of which is known to be 200 c.c at a certain point. Experiments have proved that the loss due to diluting the solution to double the volume recommended amounts to $2\frac{3}{4}$ per cent. of the total as nearly as possible.

(*d.*) *The Influence of Time.*—Sometimes when phosphoric acid determinations are asked for immediately, the time usually allowed for settling the precipitates obtained with magnesia mixture is a severe tax on the patience of the operator. On several occasions the effect of rapid determinations was tried, the soluble phosphoric acid being reported in about $1\frac{1}{2}$ hours after the commencement of the estimation. The results came out very close to what was obtained in a similar determination conducted by the usual longer method of allowing the precipitate about six hours to settle out. In the short method the magnesia mixture was slowly run in, and the whole stirred constantly for about ten minutes, and then filtered. The filtrates, on being allowed to stand, showed in some cases no further precipitate, while in others a very slight trace came down, equal to about 1 mgrm., when about 2 grms. of the sample were operated upon. Therefore, if the short method is adopted, and 1 mgrm. is added to the weight of the precipitate obtained, the result will be quite accurate.

Estimation by the Molybdate Method.

When the yellow phospho-molybdate precipitate was dissolved in ammonia and the phosphoric acid reprecipitated by the addition of magnesia mixture, without the addition of any other reagent, high results were obtained in the case of the pure salt, the results being 101·51 per cent., and 101·38 per cent. of $Ca_3P_2O_8$ in two determinations, or an average of 101·45 per cent. In these determinations the precipitates were not blowpiped, but ignited over a good Bunsen flame for about half a minute after the glow had passed over the surface, the primary part of the ignition being performed over an Argand burner.

Blowpiping the precipitate till constant in weight is commonly adopted, and, if this is done, the results obtained will be low. My expreiments on artificial manures confirm the method of correction as recommended by Neubauer (this Journal, 1894, 1092), but as the results obtained by the molybdate-citrate method described below, without blowpiping, are practically the same as those obtained after blowpiping and making the correction, I can see no reason why the blowpipe should be applied at all.

Estimation by the Molybdate-Citrate Method.

After precipitating with molybdate solution in the usual manner, filtering, washing, and dissolving the precipitate in ammonia, 0·1 grm. of citric acid is added to the ammoniacal solution, and dissolved in it before adding excess of magnesia mixture. This is what is termed here the molybdate-citrate method. Duplicate results by this method on the pure salt yielded 99·70 and 99·83 per cent. of $Ca_3P_2O_8$, or an average of 99·76 per cent.

In these determinations, as in those by the citrate and molybdate methods, and indeed in all comparative experiments on any substance, exactly the same amount of magnesia mixture was added, and in the same manner, so that no error could accrue from this source.

Excess of citric acid is to be avoided, as tending to give low results. It has been recommended that citric acid be added till the cloudiness, often noticeable in the ammoniacal solution of the phospho-molybdate precipitate, disappears. As a large quantity is often required for this purpose, the results so obtained will certainly be too low. 0·1 grm. has been found to be the best amount to add, provided the phospho-molybdate precipitate has been properly brought down.

Different Methods of Extracting Soluble Phosphoric Acid, with Various Methods of Precipitation compared.

1*st Method.*—This method consists in taking about 2 grms. of the sample, rubbing it up thoroughly in a tall 100-c.c. beaker with about 20 c.c. to 30 c.c. of cold water on two successive occasions, the solid matter being allowed to settle out each time, after which the clear supernatant liquid is filtered off. The third and final extraction is performed by adding a similar amount of hot water, heating to boiling, allowing the insoluble matter to settle for a moment, and then filtering, the residue being washed on the filter with hot water.

2*nd Method.*—To determine the effect of the washing with hot water in the first method, experiments were nowt conducted exactly as before and in exactly the same time, the tests being done side by side, *cold* water, however, in this case being used throughout.

3*rd Method.*—This consists in taking about 7 grms. of the sample, rubbing up thoroughly with about 100 c.c. to 150 c.c. of cold water in a beaker or test glass, allowing to settle over night, filtering, washing with cold water, and making up the filtrate to 500 c.c., 50 c.c. of the resulting solution being taken for the determination. In this case the estimation was done by the citrate method, as in the first two cases.

4*th Method.*—The ordinary molybdate method was used in this case, taking 50 c.c. of the solution prepared for the third method.

5*th Method.*—The molybdate-citrate method. This was conducted exactly in the same manner as the fourth method, adding, however, 0·1 grm. of citric acid to the ammoniacal solution of the phospho-molybdate precipitate before precipitating with magnesia mixture.

6*th Method.*—Reprecipitation. This method was conducted in the same manner as the fourth; the magnesium ammonium phosphate precipitate, however, was dissolved in dilute nitric acid and reprecipitated with excess of ammonia.

The results obtained on different samples of superphosphate may be seen on reference to the accompanying table.

The difference between the results of the first and second methods in Table A. is not great, but when we compare the third with the first method the difference is apparent, both cold methods failing to give so thorough an extraction of the phosphoric acid as the hot method.

When the molybdate method, No. 4, is compared with the first (the hot extraction) method a considerable difference is found, the molybdate method being the higher of the two. By adding a little citric acid to the ammoniacal solution of the phospho-molybdate precipitate—the fifth method—a slightly lower result is obtained than if no citric acid had been added, while the sixth method (re-precipitating the magnesium ammonium phosphate precipitate) seems to give irregular and undoubtedly low results.

These remarks apply to Table A. Table B. shows the comparative values of the citrate and molybdate-citrate methods applied to the estimation of the total phosphoric acid in rock phosphates.

Table A.—Comparison of Results.

Percentage of $Ca_3P_2O_8$ calculated from the Soluble Phosphates in Superphosphates.

Description of Method.	Method No.	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.	Sample No. 5.	Sample No. 6.	Sample No. 7.	Sample No. 8.	Sample No. 9.
Citrate method, hot extraction ...	1	32·72	28·41	23·25	25·28	24·99	23·95	33·12	27·13	..
" + $\frac{1}{50}$*............	..	..	..	..	25·78	25·49	24·43	33·78	..	..
" cold extraction...	2	32·33	28·36	22·86	25·13	24·97	23·84	33·16	..	..
" in ½ of 7 grms...	3	..	28·38	22·83	25·04	24·86	23·75	32·96	26·87	26·35
" + $\frac{1}{50}$*............	..	..	..	..	25·54	25·36	24·23	33·62	..	26·88
Ordinary molybdate method, in solution of "3."	4	..	..	23·95	26·24	25·96	24·76	34·12	27·64	27·72
Molybdate-citrate method, in solution of "3."	5	..	..	..	25·85	25·34	24·45	33·92	..	27·33
Molybdate reprecipitation method, in solution of "3."	6	..	..	..	25·04	24·45	24·05	33·16	..	26·52

* This addition is referred to later on.

Table B.

Percentage of Total $Ca_3P_2O_8$ in Rock Phosphates, calculated from the $Mg_2P_2O_7$ Precipitates.

Method.	Belgian. Sample No. 10.	Belgian. Sample No. 11.	Spanish. Sample No. 12.	Aruba. Sample No. 13.
Citrate....................	40·82*	41·57*	61·19*	73·07*
" + $\frac{1}{50}$............	(41·63)	(42·40)	(62·41)	(74·53)
Molybdate-citrate.......	41·63	42·46	62·43	74·46

* These results include the phosphoric acid obtained from the lime precipitates.

The Comparative Values.—So many different results being obtained by so many different processes, one is naturally led to inquire which of these methods of extraction and determination approaches most nearly the truth. In order to discuss this question, reference must be made to the results already described, obtained on the pure calcium phosphate. In this salt the $Ca_3P_2O_8$ found by the ordinary molybdate method averaged 101·45 per cent., while, by using the citrate modification of this molybdate process, an average of 99·76 per cent. was obtained, while again in the citrate method 97·82 per cent. was more than once obtained when only a small excess of citric acid was added. Thus:—

Table C.

	$Ca_3P_2O_8$ per Cent obtained in Pure Salt.
Citrate method (same as method No. 1)	97·8
Ordinary molybdate method (same as method No. 4)	101·45
Molybdate-citrate method (same as method No. 5)	99·76

Now, if the results obtained by the molybdate-citrate method be taken as the standard of accuracy, seeing that the results obtained are nearest 100·00 per cent., and if the results obtained by the same method in the samples of superphosphate be also taken as correct, and as equal to 99·76 per cent. of the total $Ca_3P_2O_8$, let the other results obtained on the superphosphates be calculated, for the sake of comparison, in proportion, and they will appear as follows:—

Table D.

$Ca_3P_2O_8$ in Samples of Superphosphate, calculated on the Basis of the Molybdate-Citrate Results.

Method.	Sample No. 4.	Sample No. 5.	Sample No. 6.	Sample No. 7.
Citrate (No. 3)..........	96·63	97·87	96·91	96·94
Molybdate	101·28	102·16	101·01	100·34
Molybdate-citrate.......	99·76	99·76	99·76	99·76

The citrate method (No. 3) already referred to is selected here, seeing that the solution employed in this is the same as that used for the two following cases, and therefore strictly comparable.

Table E.

Percentage of $Ca_3P_2O_8$ in Samples of Rock Phosphates calculated on the Basis of the Molybdate-Citrate Results.

Method.	Sample No. 10.	Sample No. 11.	Sample No. 12.	Sample No. 13.
Citrate....................	97·82	97·67	97·78	97·89
Molybdate-citrate.......	99·76	99·76	99·76	99·76

Taking the average, the results are, including those on the pure salt:—

Average of $Ca_3P_2O_8$ per Cent.

Method.	Pure $Ca_3P_2O_8$.	Superphosphates.	Rock Phosphates.
Citrate (No. 3)........	97·82	97·09	97·79
Molybdate............	101·45	101·20	..
Molybdate-citrate.....	99·76	99·76	99·76

It will be seen that these average results on the superphosphates and rock phosphates vary in proportion, and are very similar to the results found in the pure salt; and the same conclusion may be arrived at here as in the case of the results there obtained, *viz.*, that the citrate modification of the molybdate process appears to give the truest results, and should be applied to all determinations of phosphoric acid where the greatest accuracy is required.

However, as the citrate method is much quicker and more economical than the molybdate modification, it commends itself in many cases. Nor need the difference pointed out in the average results above be so great if the *hot* method of extraction be adopted, the average result in samples No. 4 to No. 7 in the table being:—

	$Ca_3P_2O_8$ average per Cent. from Table A.
Citrate method, hot extraction............	26·84
" " + $\frac{1}{50}$......................	(27·38)
Molybdate-citrate method................	27·39

Or, calculating the results by the molybdate-citrate method as equal to 99·76 per cent., as found in the pure salt:—

	$Ca_3P_2O_8$ per Cent.
Citrate method, hot extraction...........	97·76
" " + $\frac{1}{50}$......................	(99·72)
Molybdate-citrate method................	99·76

Thus it will be seen that the results on the superphosphate of lime are in exactly the same ratio to one another as those in the pure salt (Table C.), and therefore the inference based on one may be equally applied to the other.

Frequently a correction is made for the deficiency resulting from the use of the citrate method. While some recommend an addition of as much as 0·3 per cent. to the amount found, others state that no correction is required.

Now it will be seen, on reference to the above results, that if one-fiftieth of the phosphoric acid, or its equivalent of phosphate of lime ($Ca_3P_2O_8$), be added to the results found, the same result will be obtained, on an average, as found by the molybdate-citrate method. The author therefore recommends that if the citrate method be adopted, the addition of one-fiftieth of the percentage of phosphoric acid or phosphate of lime should be made to the result actually obtained by calculation from the weight of the precipitate, and the results taken as accurate. This method of correction, provided the conditions are adhered to, will be found much more convenient than taking into account considerations recommended by other writers. Thus, for example, if 25 per cent. of phosphate of lime be found by actual determination by the citrate method, 25·5 per cent. would be reported as present; and, if 50 per cent. be found, 51·0 per cent. of phosphate of lime would be reported.

This correction does not of course hold good if an excessive amount of citric acid be used in the citrate method, as this, in itself, vitiates the result. This may be seen by referring to sample A. already noted in the beginning of the paper. In this sample 25·60 per cent. of phosphate of lime was obtained by the molybdate-citrate method, and, as stated, 25·06 per cent. by the citrate method, when 0·5 grm. of citric acid (which was sufficient) was added. Now 25·06 + 0·50 (= $\frac{1}{50}$) = 25·56 per cent., which is very close to the standard molybdate-citrate result. If an addition of $\frac{1}{50}$ be made to the results obtained after the addition of 1 and 2 grms. of citric acid, it is seen that the correction does not hold good.

The presence of a moderate amount of iron does not, in itself, in any way influence the value of the correction.

Reagents used in the above Experiments.

Molybdenum Solution.—80 grms. of molybdic acid are dissolved in 80 c.c. of ammonium hydrate (0·880) *plus* 120 c.c. of water, then slowly poured into 300 c.c. of nitric acid (1·42) *plus* 300 c.c. of water, and the whole diluted to one litre with distilled water.

100 c.c. of this solution will precipitate 0·27 grm. P_2O_5.

Magnesia Mixture.—75 grms. ammonium chloride, with 62 grms. magnesium chloride (cryst.), and 200 c.c. of ammonium hydrate (0·880), to 1 litre with distilled water. 10 c.c. of this solution will precipitate 0·18 grm. P_2O_5.

Nitric Acid (dilute).—5 c.c. (sp. gr. 1·42) to 100 c.c. with distilled water.

Ammonium Hydrate (dilute).—25 c.c. (sp. gr. 0·880) to 100 c.c. with distilled water.

Ammonium Hydrate (sp. gr. 0·880).—20 c.c. added to each experiment before running in magnesia mixture.

Citric Acid and *Ammonium Oxalate* in fine powder.

The Processes used.

The Molybdate Method.—To the solution of the sample is added 20 c.c. of NH_4HO (0·880), then HNO_3 (1·42) till in excess by five drops, and then excess of molybdenum solution, slowly and with stirring, the solution of the sample being at 55° to 60° C. After settling clear in a warm place, filter, wash thoroughly with 5 per cent. HNO_3, dissolve in stream of 25 per cent. NH_4HO through filter till 200 c.c. have passed through, and add a fair excess of magnesia mixture. Filter next morning, wash, dry, ignite (not blowpipe), and weigh.

The Molybdate-Citrate Method.—As above, but in addition add 0·1 grm. of citric acid to the ammoniacal solution of the yellow precipitate before adding magnesia mixture.

The Citrate Method.—To the solution add the given amount of citric acid; boil, add excess of ammonia, then excess of acetic acid, then ammonium oxalate; boil, filter, and wash (volume now 200 c.c.). Add 20 c.c. NH_4HO and fair excess of magnesia mixture. Filter next morning, dissolve precipitate in dilute HCl, add 0·1 grm. of citric acid, dilute to 100 c.c., add 20 c.c. NH_4HO (0·880), stir, settle one hour, filter, wash twice with dilute NH_4HO, dry, ignite (not blowpipe), and weigh.

Discussion.

Mr. Otto Hehner said that the subject-matter of the paper seemed familiar. No doubt the correct estimation of phosphoric acid was important; but the amount of literature already devoted to it was so considerable as to constitute a scientific library in itself. Certainly much of Mr. Cameron's paper would be found in published works.

Mr. Cameron, in reply, said it was generally admitted that if no correction were made there was a great liability to error. In analytical work he had made that principle the basis of his work and experiments.

THE STRENGTH OF COMMERCIAL FORMALDEHYDE SOLUTIONS.

BY WILLIAM A. DAVIS.

Owing to improvements that have been made in the past three or four years in the methods of removing methyl alcohol from commercial formaldehyde solution, the table given by H. Lüttke, in Fischer's Jahresbericht (1893, 512), for the specific gravities of solutions of formaldehyde of various strengths, is no longer correct. This

Specific Gravity at 60° F.	Percentage Formaldehyde by Weight.	Formerly given.	Difference in Percentage.	Percentage Formaldehyde by Volume.
1·0025	1	1·25	−0·25	1·0
*1·005	2	2·3	−0·3	2·0
1·0075	3	3·5	−0·5	3·0
*1·0100	4	4·3	−0·3	4·0
1·0125	5	4·7	+0·3	5·0
*1·015	6	5·0	+1·0	6·1
1·0175	7	6·25	+0·75	7·1
*1·020	8	8·0	0·0	8·2
1·0225	9	8·8	−0·2	9·2
*1·025	10	10·0	0·0	10·25
1·0275	11	11·5	−0·5	11·30
*1·030	12	12·5	−0·5	12·4
1·0325	13	13·75	−0·75	13·4
1·035	14	14·7	−0·7	14·5
*1·038	15	15·7	−0·7	15·6
1·041	16	17·0	−1·0	16·6
1·044	17	18·5	−1·5	17·75
1·047	18	19·7	−1·7	18·8
1·050	19	20·3	−1·3	19·9
*1·053	20	21·3	−1·3	21·1
1·056	21	22·3	−1·3	22·2
1·059	22	23·3	−1·3	23·3
1·062	23	24·3	−1·3	24·4
*1·064	24	25·0	−1·0	25·5
1·067	25	26·5	−1·5	26·7
1·070	26	28·0	−2·0	27·8
1·073	27	29·5	−2·5	29·0
*1·076	28	32·0	−4·0	30·1
1·079	29	34·5	5·5	31·3
*1·083	30	37·0	−7·0	32·5
*1·087	31	40·0	−9·0	33·7
*1·092	32	..	..	34·9
1·096	33	..	..	36·2
*1·100	34	..	..	37·4
1·104	35	..	..	38·6
*1·109	36	..	..	39·9
1·113	37	..	..	41·2
*1·117	38	..	..	42·5
*1·121	39	..	..	43·7
1·125	40	..	..	45·0

becomes of importance, owing to the fact that manufacturers often test the strength of their product only by taking its specific gravity, before its consignment to the public.

The above table, giving a comparison of the corrected values with those formerly given, shows that in the case of the stronger solutions, an error as great as 25 per cent. of the correct strength can arise in this way; indeed, it was through such errors arising that I was induced to draw up a corrected table of specific gravities for solutions of formaldehyde up to 40 per cent. in strength.

The results were obtained with solutions of commercial formaldehyde, supplied by C. A. F. Kahlbaum, freed as far as possible from methyl alcohol. The analyses of the solutions were carried out mostly by means of the iodometric method recently given by Romijn (Zeits. ang. Chem. 36, 18—24), which is at once rapid and exact. Many of the analyses were, however, repeated, and confirmed by the method originally given by Legler (Ber. XVI. 1333).

The results marked with an asterisk were the results obtained as the mean of several experiments; the rest were obtained by interpolation. The specific gravities were taken at 60° F. The percentages by volume give the number of grms. of formaldehyde in 100 c.c. of the solution.

Discussion.

The Chairman said that it was evident that formaldehyde solutions should be valued by analysis and not by specific gravity, which, on account of the ever varying impurities, both as to quality and quantity, could not be reliable. A specific gravity table referring to poor solutions, if the author would draw it up, would possess a permanent value.

Meeting held on Monday, January 4th, 1897.

THE SMELTING AND REFINING OF CYANIDE BULLION.

BY ARTHUR CALDECOTT, B.A.

(*This Journal*, 1897, 3.)

Discussion.

Mr. A. Caldecott sends the following reply to the points raised in discussion:—

Mr. A. C. Claudet's suggestion to use pure zinc in the extractor boxes is impracticable, since precipitation in such a case is very imperfect. This is a fact well known to practical cyanide men, and also that the presence of a small percentage of lead or other metallic impurity in the zinc, whereby a couple is formed, is a positive advantage.

Bullion obtained by smelting slimes which have simply been roasted is, in actual practice, lower than 800 fine—and not 900 fine, as stated by Dr. A. F. Fuerst. The bullion obtained by the method mentioned in the paper was 814 fine. The matter of sampling and assaying cyanide bullion has recently been fully discussed at meetings of the Chemical and Metallurgical Society of South Africa.

Bisulphate of potash may be used with advantage instead of sulphuric acid when the acid treatment is practised. It is a cheap by-product in the manufacture of dynamite, and, whilst containing 36 per cent. available sulphuric acid, presents considerable advantages in transport and handling.

To lessen the gold contents of the slag, a mould to receive the contents of the crucible may be used which has a small hole bored in the side a couple of inches from the bottom. Whilst pouring, this hole is plugged up with clay, and a few minutes afterwards, when the surface of the slag has solidified, an iron rod is thrust through, whereupon the still molten slag inside runs out. During the interval after pouring, any shots of metal contained in the slag settle to the bottom, or are caught on the partially chilled layer next the sides of the mould. Hence the outflowing slag is, as experiment shows, nearly gold-free. That portion of the slag which remains as a shell in the mould can be returned to the crucible when the next charge is melted.

Manchester Section.

ROOMS OF THE CHEMICAL CLUB, VICTORIA HOTEL.

Chairman: George E. Davis.
Vice-Chairman: Peter Hart.
Committee:

F. H. Bowman.	J. M. Irving.
R. Forbes Carpenter.	*M. J. Langdon.*
R. S. Dale.	J. Lewkowitsch.
R. Le Neve Foster.	E. Schunck.
B. W. Gerland.	*W. Thomson.*
H. Grimshaw.	D. Watson.
J. Grossmann.	

Hon. Local Secretary:
J. Carter Bell,
Bank House, The Cliff, Higher Broughton, Manchester.

The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Committee:* E. Knecht, I. Levinstein, A. Liebmann, A. Ree, and C. Truby.

SESSION 1896–97.

Meeting held on Friday, May 7th, 1897.

THE PRESIDENT IN THE CHAIR.

CONTRIBUTIONS TO THE ANALYSIS OF FATS. VIII.—THE ACETYL VALUE.

BY DR. J. LEWKOWITSCH.

The acetyl value, introduced by Benedikt into the analysis of fats as a chemical constant, furnishes a measure of the proportion of hydroxylated fatty acids in fatty oils or fats.

The method of determining the acetyl value as proposed by Benedikt is based on the principle that hydroxy acids, on being heated with acetic anhydride, exchange the hydrogen atom of their alcoholic hydroxyl group or groups for the radicle of acetic acid according to the following equation, taking ricinoleic acid as an example:—

$$\underset{\text{Ricinoleic acid.}}{C_{17}H_{32}(OH)COOH} + \underset{\text{Acetic anhydride.}}{(C_2H_3O)_2O} =$$

$$\underset{\text{Acetylricinoleic acid.}}{C_{17}H_{32}(OC_2H_3O)COOH} + \underset{\text{Acetic acid.}}{C_2H_4O_2}$$

The acetylation process and the determination of the acetyl value was carried out, according to Benedikt and Ulzer, in the following manner:—The free fatty acids were prepared in the usual manner and heated together with acetic anhydride. The mass was next boiled with water and washed until the acetic acid was entirely removed. The acetylated fatty acids were then filtered off, and both their acid and saponification values determined in the well-known manner. The difference between the two values thus found was termed "acetyl value."

The neutralisation and saponification of the acetylated fatty acids was supposed to take place in two stages, according to the following equations:—

I.

$$\underset{\text{Acetylricinoleic acid.}}{C_{17}H_{32}(OC_2H_3O).COOH} + KOH =$$

$$\underset{\text{Potassium acetylricinoleate.}}{C_{17}H_{32}(OC_2H_3O)COOK} + H_2O$$

II.

$$\underset{\text{Potassium acetylricinoleate.}}{C_{17}H_{32}(OC_2H_3O).COOK} + KOH =$$

$$\underset{\text{Potassium ricinoleate.}}{C_{17}H_{32}(OH)COOK} + \underset{\text{Potassium acetate.}}{C_2H_3O_2K}$$

Accordingly, the acetyl value would indicate the number of milligrammes of KOH required for the neutralisation of the acetic acid obtained on saponifying 1 grm. of the acetylated insoluble fatty acids.

The hydroxyl in the carboxyl group of the fatty acids was therefore, in the light of the explanations given, not affected during the acetylating process. Hence fatty acids containing no alcoholic hydroxyl group, such as stearic, oleic, &c., should not yield an acetyl value, and the acid and saponification values should be identical in these cases.

I have, however, shown (this Journal, 1890, 660) that pure capric, lauric, palmitic, stearic, cerotic, and oleic acids gave very considerable acetyl values when treated by Benedikt and Ulzer's process. This result could only be explained by the fact that the fatty acids named had been converted into their anhydrides, the acetic anhydride acting in the manner explained by the following equation:—

$$+ \underset{\text{Palmitic acid.}}{C_{15}H_{31}COOH} + \underset{\text{Acetic anhydride.}}{\begin{matrix} C_2H_3O \\ C_2H_3O \end{matrix}\!\!>O} =$$

$$\underset{\text{Palmitic anhydride.}}{\begin{matrix} C_{15}H_{31}CO \\ C_{15}H_{31}CO \end{matrix}\!\!>O} + \underset{\text{Acetic acid.}}{2C_2H_3O.OH}$$

When the anhydrides were dissolved in cold alcohol for the purpose of titrating, on adding the potash hydrolysis (saponification) of the anhydrides took place at once to a certain extent, and as the acid combined with a certain quantity of potash an (apparent) acid value of the substance was obtained. When, however, the anhydrides were shaken up with water, the first drop of potash gave a pink coloration (in the presence of phenolphthalein), which disappeared but slowly. Thus in the alcoholic solution partial hydrolysis of the anhydrides took place, hydrolysis ceasing when an equilibrium was established in the solution. Under these conditions apparent acetyl values were obtained, being, in truth, nothing but fictitious values.

Hydroxylated fatty acids are certainly acetylated when boiled with acetic anhydride, but simultaneously the acetylated acids are converted into their anhydrides in consequence of the dehydrating action of the acetic anhydride used in excess. On being boiled subsequently with water to remove acetic acid, a portion of the anhydrides may or may not become hydrolised, yielding possibly a mixture of free acetylated acids and acetylated anhydrides. When this is dissolved in alcohol and titrated with caustic potash, after neutralisation of the free acids, if any, partial hydrolysis will set in, as explained above, and thus an acid value will be obtained (Benedikt's "acetyl acid value"). But such acid value will be lower than the true acid value of the acetylated fatty acids, the anhydrides remaining unacted on to a certain extent.

Consequently the saponification value of the acetylated product (Benedikt's "acetyl saponification value") will be found too high, the not-hydrolised anhydrides being then saponified by the boiling alcoholic potash. The difference between the saponification and acid values, supposed to be the acetyl value of the acids, will therefore, be devoid of any quantitative meaning.

The same argument holds, of course, of mixtures of ordinary fatty acids of the acetic and oleic series and hydroxylated acids.

No ready method being available to ascertain whether complete hydrolysis of the anhydrides has taken place after boiling out with water (the anhydrides of the higher fatty acids proving themselves very stable indeed, even on boiling with water), I rejected Benedikt's method altogether and proposed to determine the true acetyl value by actually titrating the amount of acetic acid assimilated by the hydroxylated acid in the form of acetyl, C_2H_3O, and given up, on saponification, as acetic acid to the alkali (this Journal, 1890, 846).

This is done by boiling the acetylated product with alcoholic potash and estimating the acetic acid formed in a similar fashion, to that adopted in the determination of volatile acids by Reichert's distillation process. There is, however, this important difference that the *total* amount of volatile acids, in the present case, acetic acid, is driven off, so that the distillation is carried on until practically no acidity is shown by the distillates. This occurs, as a rule, when about 500 or 700 c.c. have been distilled over. The distilled liquors are then titrated with standardised potash, phenolphthalein being the indicator; the number of c.c. of normal potash used per gramme of the acetylated product multiplied by 56·1 represents the acetyl value of the insoluble fatty acids.

On repeating my experiments I proved that my method yielded correct results. But at best the process is a somewhat tedious one. Furthermore, it has that important drawback, that the acetyl value refers to the insoluble fatty acids rather than to the oils and fats themselves. This naturally tends to obliterate at the outset important differences existing between the various glycerides, for in the process of preparing the fatty acids volatile fatty acids are washed away and the characteristic differences of such fats as, *e.g.*, butter and tallow, may entirely disappear. There is the further drawback that during the operations entailed in liberating and drying the free fatty acids, oxidation may set in with possible formation of hydroxy acids, resulting in an increased acetyl value.

It appears, therefore, preferable to work with the original fats and thus to bring this important constant into line with the other constants, such as saponification value, iodine value, &c. Since the basic radical, glyceryl, is in common to all the glycerides it is evident that any difference between the mixed fatty acids of the various oils and fats must find its corresponding expression in the different behaviour of the oils and fats themselves. This is, indeed, borne out by various reactions, such as the action of sulphuric acid on the fatty acids on the one hand and the oils and fats on the other, due allowance being made for the constitutional difference between acids and their glyceryl ethers (esters).

It was then found that the oils and fats containing hydroxy acids assimilate acetyl radicles. In their case, however, no complication can arise through formation of anhydrides, so that from neutral oils and fats neutral—though acetylated—esters were obtained. The small quantity of free fatty acids naturally occurring in oils and fats may be left out of consideration here, as not appreciably affecting the result.

It would thus appear that by taking the difference of the saponification values of the acetylated esters and the original esters, or, to use less scientific parlance, of the acetylated oils and fats and the original oils and fats the acetyl values would be obtained. If it could be agreed amongst chemists that this difference should be looked upon as the true acetyl value the matter might end there. But, obviously, it would not be a very rational proceeding, for the acetylated products naturally have a higher molecular weight than the original oils and fats. Thus the two substances are not strictly comparable, unless, indeed, we reduce the weight of the acetylated substance by calculation to the corresponding amount of original substance.

I therefore prefer to ascertain the acetyl value by determining direct the amount of acetic acid formed on saponification. I proceed as follows:—

10 grms. of an oil or fat (or any other convenient number of grms.) are boiled with an equal volume of acetic anhydride for two hours in a round-bottomed flask attached to an inverted condenser. The mixture is then transferred to a large beaker, mixed with several hundred c.c. of water and boiled for half an hour. A slow current of carbonic dioxide is conveniently passed into the liquor through a finely drawn out tube reaching nearly to the bottom of the beaker; this is done to prevent bumping. The mixture is then allowed to separate into two layers, the water is siphoned off and the oily layer again boiled out in the same manner until the last trace of acetic acid is removed. This is ascertained by testing with litmus paper. The acetylated product is then freed from water and finally filtered through filter paper in a drying oven.

This operation may be carried out quantitatively, and in that case the washing is best done on a weighed filter. On weighing the acetylated oil or fat, an increase of weight would prove that assimilation of acetyl groups

has taken place. This method may be found useful to ascertain preliminarily whether a notable amount of hydroxylated acids is present in the sample under examination.

2 to 4 grms. of the acetylated substance are then saponified by means of alcoholic potash solution as in the well-known determination of the saponification value. If the "*distillation process*" be adopted it is not necessary to work with an accurately measured quantity of standardised alcoholic potash. In case the "*filtration process*" be used, the alcoholic potash must be measured exactly. (It is, however, advisable to employ in either case a known volume of standard akali as one is then enabled to determine the saponification value of the acetylated oil or fat.) Next the alcohol is evaporated and the soap dissolved in water. From this stage the determination is carried out either by (*a.*) the "distillation process" or (*b.*) "filtration process."

(*a.*) *Distillation Process.*—Add dilute sulphuric acid (1:10) more than to saturate the potash used, and distil the liquid as is usual in Reichert's distillation process. Since several 100 c.c. of water must be distilled off, either a current of steam is blown through the suspended fatty acids or water is run into the distilling flask, from time to time, through a stoppered funnel fixed in the cork, or any other convenient device is adopted. It will be found quite sufficient to distil over 500 to 700 c.c., as the last 100 c.c. practically contain no acid. Then filter the distillates to remove any insoluble acids carried over by the steam, and titrate the filtrate with decinormal potash, phenolphthalein being the indicator. Multiply the number of c.c by 5·61, and divide the product by the weight of substance taken. This gives the *acetyl value*.

(*b.*) *Filtration Process.*—Add to the soap solution a quantity of standardised sulphuric acid *exactly* corresponding to the amount of alcoholic potash employed, and warm gently, when the fatty acids will readily collect on the top as an oily layer. (If the saponification value has been determined, it is, of course, necessary to take into account the volume of acid used for titrating back the excess of potash.) Filter off the liberated fatty acids, wash with boiling water until the washings are no longer acid, and titrate the filtrate with decinormal potash, using phenolphthalein as indicator. The acetyl value is calculated in the manner shown above.

It goes without saying that both methods must give identical results. A glance at the table will show that this is the case. The method (*b.*) will be found shorter and more convenient than (*a.*).

We now arrive at the definition:—

The acetyl value indicates the number of milligrammes of KOH required for the neutralisation of the acetic acid obtained on saponifying 1 grm. of the acetylated oil or fat.

No.	1. Kind of Oil or Fat.	2. Acetylated Oil or Fat.			3. Neutralised Filtrate from III. acidified and distilled. Acetyl Value calculated.	4. Insoluble Fatty Acids from 3 distilled. Used for neutrelising Distillate Decinormal KOH.
		Saponification Value.	Acetyl Value by Distillation Process.	Acetyl Value by Filtration Process.		
		I.	II.	III.		C.c.
1	Castor oil, I.	311·2	150·5	149·6	..	..
	„ „ II.	310·3	149·9	149·4	..	0·0
	„ „ III.	312·1	..	146·7	..	0·1
2	Cotton-seed oil, I.	213·3	24·76	25·1	..	..
	„ „ II.	216·5	..	21·1	..	..
	„ „ III.	214·7	..	21·9	..	..
3	Maize oil, I.	201·5	8·75	8·25	..	0·0
	„ „ I.	..	..	8·21	..	..
	„ „ II.	200·9	7·81	7·9	7·62	0·0
4	Colza oil	192·9	17·2	16·6	..	..
5	Olive oil, I.	203·1	12·78	13·48	13·48	..
	„ „ II.	204·7	..	13·62	13·44	..
6	Linseed oil, I.	208·5	6·85	6·92	6·92	..
	„ „ II.	210	7·03	..	..	..
7	Shark-liver oil	..	..	17·83	..	..
8	Animal oil	221	22·04	22·38	..	..
9	Horse's-foot oil	214	..	14·40	..	..
10	Tallow (South America)	202·4	9·52	9·82	..	..
11	Beef marrow	203·6	6·63	6·64	..	..
12*	Croton oil, I.	236	40·68*	41·09*	..	..
	„ „ I.	237·1	40·85*	40·91*	..	..
	„ „ II.	210·4	..	53·55*	..	..
13*	Cocoanut oil	..	57·29*	..	..	..
14*	Butter fat	..	45·23*	..	..	..

* Should be accepted with reserve.

To further check the correctness of the process (*b.*), in some cases the neutralised filtrate was acidified and the acetic acid distilled off again, when the same number as that given in col. 2 III. was found. The insoluble fatty acids on being subjected to distillation in a current of steam proved themselves free from volatile fatty acids, as shown in col. 4.

The oils and fats given in the table under No. 1–11 contain practically no volatile fatty acids, as evidenced by their extremely low Reichert values. It is clear that in the case of those glycerides that possess notable Reichert values, such as croton oil, cocoanut oil, and butter fat—No. 12–14 of the table—a complication arises, and it might appear that just for this very reason it would be advisable to work on the insoluble fatty acids after elimination of the volatile acids, or, at all events, of the bulk of them, and then determine their acetyl value. This objection, however, is of no importance, and I shall show in a subsequent paper how the true acetyl value is determined. Meanwhile the acetyl values given for No. 12, 13, and 14 should be considered as representing a measure of both volatile and hydroxylated acid, and should therefore be accepted with reserve.

I confine myself at this occasion to proposing these processes as readily workable ones for the determination of the acetyl value without discussing the conclusions to be drawn from the numbers given. These numbers deviate considerably from those published by Benedikt and Ulzer (cpr. Lewkowitsch, Chemical Analysis of Oils, Fats, Waxes, &c., p. 251), and their figures must therefore be abandoned. Acetyl numbers published during the last years by several chemists fall under the same stricture, and their revision is therefore necessary.

It should be noted that if the oil or fat under examination contains *free alcohols*—phytesterol, cholesterol—the acetyl value will be a measure of both the hydroxy acids and the free alcohols.

If the free alcohol is isolated, its acetyl value (*see* below) should be determined as well. The difference between the acetyl value of the original oil or fat and the acetyl

number proportionate to the amount of free alcohol present, will be the true measure of the proportion of hydroxy acids.

The determination of the acetyl value of a *free alcohol* offers no difficulty. On acetylating the alcohol, its acetate is formed; the saponification value of the latter is also its acetyl value, the alcohol having, of course, no saponification value.

The examination of natural waxes, characterised by the occurrence of free alcohols is in hand, and their acetyl values will be given in a subsequent paper.

In conclusion I wish to express my thanks to my assistant, Mr. C. D. Robertshaw, to whom I am indebted for the analytical results stated in the table.

Newcastle Section.

SESSION 1896–97.

Meeting held at the Durham College of Science, Newcastle-on-Tyne, on Thursday, May 27th, 1897.

MR. F. S. NEWALL IN THE CHAIR.

MICRO-CHEMICAL EXAMINATION OF LEAD-ANTIMONY, TIN-ANTIMONY, TIN-ARSENIC ALLOYS, &c.—PART II.

BY JOHN E. STEAD, F.I.C.

In reviewing the work of other investigators in my first paper, by an oversight no notice was given of the very valuable paper "On the Structure of Metals: its Origin and Changes," by Mr. F. Osmond and Prof. Roberts-Austen, read before the Royal Society last year. This paper treats on the mechanical properties and micro-structure of gold containing about $\frac{2}{10}$ths per cent. of other metals. They classify them into classes according to the nature and thickness of the joints, as follows:—

"1st. Pure gold alloys with Zr, Al, Rh, Pd, Zn; joints which are usually non-crystalline, of a thickness which equals 0 to 2 μ ($\mu = 0{\cdot}001$ mm.); the mathematical joints, or very fine ones are dominant.

"2nd. Alloy of lithium: the thickness of the joints equals 0 to $3{\cdot}5\ \mu$; the thick joints are crystalline, but are rare and disconnected; the mathematical joints, or very fine joints, are dominant.

"3rd. Alloys with Sb, Se: crystalline joints, which are often broken; the thickness of these equals 1 μ to $3{\cdot}5\ \mu$, or a mean thickness of 2 μ.

"4th. The alloys of In, K: crystalline continuous joints, with a thickness of 1 to 4 μ, or a mean thickness of 2 to $2{\cdot}5\ \mu$.

"5th. Alloys with Tl, Bi: crystalline joints, which are continuous, often passing into fissures; the thickness equals 1 μ to 5 μ, or a mean thickness of $2{\cdot}5\ \mu$. Generally speaking the condition of these joints appears to be closely related to the mechanical properties of the alloy, as will be seen by the following table, published in the Philosophical Transactions of the Royal Society, Vol. 179, 1888, A., p. 339."

Table showing the Properties of Gold when Alloyed with 0·20 per Cent. of various Elements.

Name of Added Element.	Tensile Strength.	Elongation Per Cent. on Three Inches.	Impurity Per Cent.	Atomic Volume of	Reduction of Area at Fracture per Cent.
	Tons per Sq. In.				
Potassium	Less than 0·5	Not perceptible	Less than 0·2	45·1	Nil.
Bismuth	0·5 (about)	"	0·210	20·9	"
Tellurium	3·88	"	0·186	20·5	"
Lead	4·17	4·9	0·240	18·0	Very slight.
Thallium	6·21	8·6	0·193	17·2	15
Tin	6·21	12·8	0·196	16·2	Not measured.
Antimony	6·0 (about)	Qy.	0·203	17·9	54
Cadium	6·88	44·0	0·202	12·9	*
None. Pure gold	7·00	30·8	None	..	*
Silver	7·10	33·3	0·200	10·1	*
Palladium	7·10	32·6	0·205	9·4	75
Zinc	7·54	28·4	0·205	9·1	74
Rhodium	7·76	25·0	0·21 (about)	8·4	*
Manganese	7·99	29·7	0·207	6·8	60
Indium	7·99	26·5	0·290	15·3	72
Copper	8·22	43·5	0·193	7·0	*
Lithium	8·87	21·0	0·201	11·8	60
Aluminium	8·87	25·5	0·186	10·45	46

* These test pieces drew out after the manner of pitch; that is, as a viscous solid.

They also treat, in a very interesting chapter, on some phenomena of annealing, and prove that the large grains of the melted metal became subdivided after heating for five minutes to between 200° to 250° C. into a number of small polyhedral grains, when bismuth, thallium, antimony, and aluminium were present, whereas pure gold and the alloys with K, Pd, Zn, Rh, Li, Se, and Zr did not undergo any change; and conclude that, generally speaking, it seems that bismuth, thallium, antimony, and aluminium, when present in the proportion of about 0·2 per cent., behave in respect to gold in the same way as carbon does to steel, but at a much lower temperature.

Since I last lectured before this Section I have received the March Bulletin of the Société d'Encouragement pour l'Industrie Nationale, which contains a most interesting communication by Mr. M. G. Charpy, on the Microscopic Study of Metallic Alloys. In this work Mr. Charpy has fully described the structure of some of the alloys described in my paper, and it is satisfactory to note that our conclusions do not disagree—at least, to any important extent.

The alloys studied and described by Mr. Charpy are as follows:—

Lead and antimony.
Lead and tin.
Zinc and aluminium.
Bismuth and tin.

Copper and antimony.
Bismuth and antimony.
Copper and tin.
Antimony and tin.
Antimony and silver.
Copper and zinc.

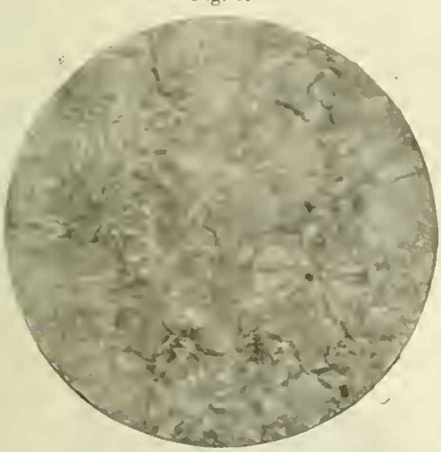
It will be seen from this list that Mr. Charpy has investigated three of the series of alloys I described in my first lecture, *viz.*, the lead-antimony, tin-antimony, and copper-tin series.

So far as our microscopic work is concerned, our results agree closely, but Mr. Charpy has more fully studied the structures of the lead-antimony alloys containing less than 13 per cent. antimony than I had done at that time; and I, on the other hand, had supplemented the microscopic investigation with a chemical research, with the object of determining the composition of the separated crystals.

Lead and Antimony.—Mr. Charpy had great difficulty in polishing the alloys, containing less than 13 per cent. of antimony, and it will be remembered that I experienced the same thing, and resorted to the examination of the surface of the solidified eutectic of the lead-antimony alloys instead of the polished and etched surfaces. On further attempts to develop the structure by etching this eutectic, and of the alloys containing less than 13 per cent. antimony, I have recently been more successful. After long-continued action of dilute nitric acid on the eutectic, it was observed that a thick adherent layer, almost black, covered the polished surface. A portion of this was removed, and was, after well washing with water, tested for lead, and as it was found to be absent we may conclude it was pure antimony.

After rubbing away the dark layer by gentle friction under running water, the metal below presented a most characteristic microscopic appearance. At fairly equable distances apart, what appeared to be laminæ radiated from nuclei, and continued in right lines until met by similar radiations from other nuclei. The accompanying photograph clearly illustrates this.

Fig. 1.



Lead-Antimony Eutectic. × 50 diameters.

The appearance is similar to that of nodules of pyrites, with radial structure or spherulites in obsidian.

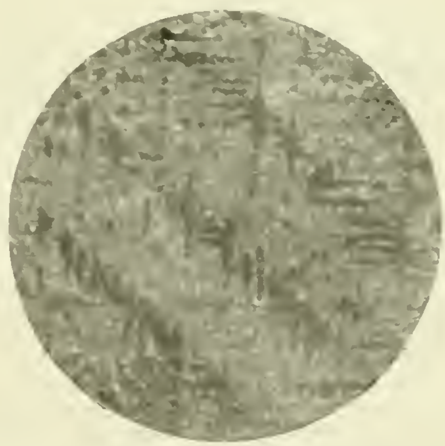
On examining the dark crust after gently crushing so as to separate the individual particles of antimony, it was found that it consisted of very fine, thin plates, as the microscopic appearance of the etched specimen would lead us to suppose; and these, on further pressing, appeared to split up into excessively fine rod-like bodies, which, but for their absolute opacity, might be mistaken for certain forms of bacteria.

Every metal I have examined, and many alloys, appear to be built up of laminæ, and such laminæ are themselves built up of crystals of definite form. The lead eutectic is no exception to the rule, and it appears certain that it is constituted of alternate laminæ of lead and antimony, as Mr. Charpy suggests. The composition of this eutectic is, as before pointed out, that of the atomic proportion of Pb_4Sb; but there can be no doubt, judging from the facts just referred to, in the solid state they are not in chemical union.

Whether this eutectic is analogous to a cryohydrate, or at its melting point when fluid is a chemical compound which afterwards, simultaneously with solidification, splits up into its elements, remains to be determined. The fact that it has a distinct tendency to crystallise in hexagonal forms, whereas both constituents separately at the solidification point give skeleton octahedra, strengthens the latter hypothesis. Fluid grey iron simultaneously solidifies and splits up into iron and graphite, and may be an instance analgous to what takes place in the eutectic.

It has already been shown by the critical points on cooling, that in the alloys containing less than 12·7 per cent. antimony, the lead crystallises in the eutectic. Mr. Charpy has demonstrated that this is so by the microscope, and I have recently obtained an excellent illustration of this from an alloy containing 10 per cent. antimony and 90 per cent. lead, in which the dendritic crystals of lead are clearly developed in an eutectic matrix, thus confirming Charpy's results.

Fig. 2.



Lead, 90 per cent. Antimony, 10 per cent.
Polished and etched with acid. × 50 diameters.

The structures, or surface markings of small ingots of alloys after solidification, without any preparation, frequently are similar and sometimes identical with those obtained by the more laborious methods of grinding and polishing. This is the case with some of the alloys of tin and copper, tin and arsenic, tin and lead, lead and antimony. The surface of an ingot or small cake of a lead-antimony alloy containing 10 per cent. antimony, has upon it the skeleton octahedron lead crystals and the spherulitic structures of the eutectic side by side. If the antimony present does not exceed 6 per cent., the eutectic sinks below the mass of lead crystals, which solidify first, leaving the latter in strong relief; it is therefore impossible, in such cases, to obtain on the surface indications of the structure of the eutectic.

As the eutectic on digestion in nitric acid has all of its lead dissolved away, leaving the antimony intact, and in a mass of the same bulk as the original alloy, and also fairly coherent, it was believed that when there were free fir-tree crystals of lead in the solid eutectic, they would be dissolved away by acid, leaving cavities where they originally existed, and that on breaking the coherent and porous mass after drying it, these cavities would be visible on the fractured surfaces. On making the experiment this was proved to be the case; the empty areas where at one time lead had crystallised were clearly visible.

The solid eutectic of lead and tin consists of similar masses of spherulites. They can be easily seen on the surface of an ingot carefully cooled, and if, when the alloy is partially solidified and is poured out suddenly upon an iron plate, they are readily detected, for they stand out as

little nobs above the surface of what was, at the time of pouring, liquid metal. The spherulites being solid, the still liquid metal falls away, leaving the nobs referred to.

When alloys of antimony and lead with between 15 per cent. and 40 per cent. are caused to solidify very slowly, the crystals of antimony do not separate in the mass and then float to the surface, but begin to form at the surface and slowly grow downwards, and the crystals branch out. and are more or less perfectly joined together, and the ends terminate in the eutectic below.

Antimony and Tin.—Since reading the first part of this paper, the study of these alloys has been further investigated. It would not appear that Mr. Charpy has fixed the composition at which cubic crystals cease to be formed on cooling. This point, according to my work, appears to be reached when the antimony approximates to 7·5 per cent. When this alloy is perfectly polished, the joints of the grains appear light on a slightly darker ground, and, it would seem, contain a compound harder than that of the main body of the grains. After slightly etching with very dilute nitric acid, these brighter parts develop to a darker shade than the centres. In fact, the appearance is exactly reversed. When the section is bent, fracture commences at the joints.

All these considerations lead us to the conclusion that there is a fusible eutectic in the joints of the grains, richer in antimony than that in the main mass.

On taking the lower part of an alloy containing 10 per cent. antimony which had been so slowly cooled as to permit of the hard cubic crystals floating to the surface, this also had the same appearance. On digesting this portion of the alloy for several days in weak hydrochloric acid, a dark-coloured, soft, and easily detached layer remained adhering to the surface. It apparently consisted of an amorphous structureless powder which soiled the fingers like lampblack. No structure or form could be detected by the highest power of the microscope. The small quantity available was analysed, and a considerable amount of tin was proved qualitatively to be present. A larger quantity is in process of being separated, which, when obtained, will be more thoroughly examined.

In the alloy containing 4 per cent. antimony, the structure appeared to be similar to the last.

All the specimens containing between 7·5 per cent. and 0·5 per cent. antimony crystallised in the same forms as pure tin, and the crystalline markings of octahedral skeletons were almost identical.

Mr. Charpy believes that he detected needles of pure tin in a 5 per cent. antimony alloy; but it is doubtful whether pure tin could be detected, for every polished section, even of alloys containing very little antimony, after treatment for a short time with dilute nitric acid or hydrochloric acid, was darkened by an amorphous black deposit slightly darker and most pronounced at the joints of the grains, and the highest power could not resolve any separate constituent.

The photograph given by Mr. Charpy resembles an appearance which can be obtained readily enough by the action of very dilute acid for a few seconds, and is exactly what I have before described, and proves that there is a larger proportion of antimony at the joints than in the mass. When, however, the etching action is continued, the whole surface is veiled over, those portions at the joints still taking the lead in relative darkness. To the naked eye the etched surface assumes the same appearance as bright copper assumes when it has received a coating of arsenic in the Reinsch test.

Perfectly pure tin, prepared by electrolysis, after fusing and polishing, gave no bright junction lines, and on slightly etching, developed a granulation identical with the alloys containing a little antimony—with this exception, that the joints remained relatively brighter than the mass, and after long etching no dark antimonial veil could be obtained. We conclude therefore that tin and antimony, or tin and a compound of tin and antimony, solidify together, either as an amorphous mixture, or solidified solution; and that, if the tin in such alloys is in the free state, it is in such close contact and so intermixed with the antimonial compound or free antimony as to be unrecognisable under the highest power of the microscope.

With regard to the composition of the cubic crystals which separate from alloys containing above 7·5 per cent. antimony, I proved in my first communication, those from the 25 per cent. alloy had the composition of SnSb, but after what we have learnt with regard to the crystals in copper-tin, to which we shall presently draw attention, it will be necessary to separate them from several alloys containing different quantities of antimony, and analyse each separately, before we can conclude that they are or are not of that constitution. The same precaution is necessary with regard to the crystals in tin-arsenic and tin-phosphorus compounds.

I have shown that the melted and solidified cubic crystals containing equal atomic proportions of antimony and tin, split up into the eutectic alloy and a compound containing a relatively larger proportion of antimony. After very slow cooling such an alloy, I have obtained much better results, and have demonstrated that all of the white plates are relatively more highly charged with antimony in the centre than at the sides, and that some have a clearly defined, separate, hard constituent in the centre.

In general, the results of Mr. Charpy and my own agree most closely. The slight difference observed in the structures of the 25 per cent. antimony-tin alloy would be most readily accounted for by differences in the rate at which the alloys were cooled.

I have myself obtained the same differences by varying the conditions of cooling.

It is in the alloys containing little antimony (5 per cent.) where we differ more seriously, but here, even, it is not important. Mr. Charpy believes he can see the separate independent tin crystals. I maintain this is impossible.

Tin-Copper Alloys.—To chemically examine all the alloys of copper and tin is the work of time. I have not fully examined other than a few of the alloys containing an excess of tin; but as the results are rather different from those obtained by other investigators, it becomes of interest to give them at once, without waiting to complete an exhaustive research.

Microscopic results of tin-copper compounds obtained in my laboratory were exhibited on the screen at my last lecture, in February, but were not given in my printed paper, as the research was so incomplete.

The alloys examined contained between 0·1 per cent. and 40·0 per cent. of copper.

On polishing the slowly cooled alloys and etching with very dilute nitric acid, it was possible to detect, even in the alloy with only 0·1 per cent. copper, fine bright needles; and these increased in size, both in length and diameter, with each addition of copper. In the alloy in which the proportion of copper amounted to 2 per cent., the needle crystals were completely separated by long digestion in dilute nitric acid (1 to 10), followed by treatment with dilute hydrochloric acid, which readily dissolved the hydrated

Fig. 3.

Copper, 30 per cent. Tin, 97 per cent.
× 50 diameters.

metastannic oxide formed by the nitric acid. The crystals so obtained were well formed, the edges perfectly angular, and the sides bright, smooth, and metallic, having the colour and brightness of polished nickel. In form they were apparently square prisms, and occasionally several of them were found together to form a more or less plate-like structure. With 3 per cent. copper the alloy gave similar crystals, but the tendency to grow into plates was greater than in the others. With 5 per cent. copper the alloy yielded crystals, the greater proportion of which were in plates which crystallised with bright, smooth surfaces. With 10 per cent. the separated crystals were badly formed, and were nearly all in the form of plates with most irregular, granulated, uneven surfaces, but occasionally well-formed needle crystals protruded from the ends of the plates.

From this alloy up to that of the composition of SnCu, containing 34·7 per cent. copper, all the separated compounds were similar in appearance, and, excepting at the ends of some of the plates, there were no clearly definite crystalline forms present.

All the crops of crystals were analysed, but before doing so they were examined under the microscope, and proved to be free from oxide of tin and from surface oxidation. They were simply tested for copper, the tin being taken by difference, excepting in two cases, when the tin was determined, and the results obtained agreed closely with the difference between the copper present and 100 parts.

The following are the results :—

Alloy.	Crystals separated.	
	Copper.	Tin.
	Per Cent.	Per Cent.
98 per cent. tin, 2 per cent. copper ...	34·58	65·42 SnCu
97 " " 3 " " ...	36·50	63·50
95 " " 5 " " ...	39·80	60·20
90 " " 10 " " ...	44·60	55·40 Sn_2Cu_3
85 " " 15 " " ...	47·20	52·80
80 " " 20 " " ...	53·00	47·00
65·3 " " 34·7 " " ...	56·12	43·88 $SnCu_2$

Mr. Charpy states that from similar alloys H. le Chatelier and M. Laurie obtained crystals quite pure, having the exact formula of $SnCu_3$. Now $SnCu_3$ contains :—

	Per Cent.
Copper	61·6
Tin	38·4

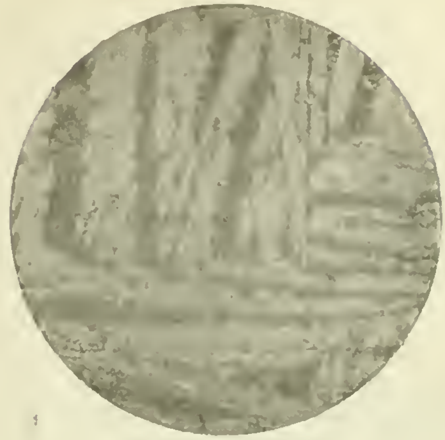
The crystals obtained from the 2 per cent., 10 per cent., and 34·7 per cent. copper alloys approximate to the formulæ SnCu, Sn_2Cu_3, and $SnCu_2$ respectively, but none of them approach to $SnCu_3$.

In my trials, the first crystals obtained were from the 2 per cent. alloy, and as these appeared to be of definite chemical composition, a large quantity of an alloy was made of the composition SnCu, and the soluble eutectic was then dissolved and the separated compounds analysed. This proved to have the composition approximating to $SnCu_2$. The micro-structure showed the presence of the edges of thick, bright plates and a compound readily dissolved away, which left a darkened etched surface which was practically pure tin. It is evident that such a compound SnCu could not be melted by itself without suffering change, and is another instance of the same thing which occurs when carbide of iron, antimonide, arsenide, and phosphide of tin are melted by themselves.

It is possible that all the various separate compounds, containing more copper than sufficient to make SnCu, may be mixtures of molecules of that composition, with other molecules containing a larger atomic proportion of copper. The fact that needle crystals were detected at the ends of some of the plates, and that certain retardations in cooling were observed by Prof. Roberts-Austen and Mr. Stansfield, rather strengthen the view that they may contain more than one chemical constituent.

The facts are undisputable, however, that the copper steadily increases in the separated crystals with each increment of copper in the alloy.

Fig. 1.



Copper, 40 per cent. Tin, 60 per cent.
× 30 diameters.

When the proportion of tin is 60 per cent. the fusible tin eutectic is most readily noticed, but when 56 per cent. copper and 44 per cent. tin are present it is absent, and it is difficult to develop the structure. I have not, however, thoroughly studied the alloys higher in copper than 40 per cent.

Eutectics.—About 18 months ago, I demonstrated and Mr. Charpy has also shown that there is some relation in structure between the pearly constituent discovered by Dr. Sorby in steels and that of eutectics in alloys. The pearly effect in steel after etching, as is well known, is caused by parallel laminæ, often very much curved, of a hard constituent of the composition of Fe_3C, projecting in relief on the polished surface of steel, the alternative laminæ of soft iron having been either worn away by polishing or dissolved away by acid. The eutectics of 23 per cent. copper and 77 per cent. antimony, and of bismuth and tin, give fine pearly structures. The eutectic of tin and antimony does not give any pearly structure, for the simple reason that the hard and soft constituents are not separate in the form of lamular plates. The eutectic of lead and antimony consists of alternate plates of soft and hard constituents, and theoretically should give a pearly structure; but I have not succeeded in obtaining it.

The eutectic of tin and lead, under high-power magnification, although crystallising in gross spherulites, appears to consist of two constituents, worm-like in structure; but I have failed to obtain any perlite appearance.

The eutectic of lead, antimony, and tin is brilliantly pearly, and so also are the eutectics of several of the ternary alloys; and under high power in all such cases they are resolved into comparatively gross alternate layers of a hard and soft constituent.

The structure of the eutectic of an alloy containing 80 per cent. lead, 5 per cent. tin, and 15 per cent. antimony is a marked instance of this, and also indicates a very beautiful crystallite formation.

In concluding this second paper, I must apologise for its incompleteness: but I am continuing the investigation, and shall, if agreeable to the Society, continue to bring before it the results of my future work.

Errata for Part I.

No. 2 and 3 diagrams are reversed. No. 2 should be No. 3, and No. 3 should be No. 2 (this Journal, 1897, 202).

The description of diagram No. 9 should have been: Tin, 99·75 per cent.; phosphorus, 0·25 per cent. (*ibid.* 206).

Note.—Prof. Behrens has given a good account of both copper-tin and tin-copper alloys in his work, "*Das Mikroskopische Gefüge der Metalle und Legierungen.*"

Nottingham Section.

UNIVERSITY COLLEGE, NOTTINGHAM.

SESSION 1896–97.

Meeting held in Nottingham, on Wednesday, May 26th, 1897.

MR. F. J. R. CARULLA IN THE CHAIR.

ON A PURE CULTIVATION OF A BACILLUS FERMENTING BRAN INFUSIONS.

BY J. T. WOOD AND W. H. WILLCOX, B.SC. (LOND.)

Isolation of Pure Culture.—In our previous communication on bran fermentation as applied in the manufacture of light leathers (this Journal, 1893, 422), we gave an account of the actual fermentation and its products, together with the mode of action on the bran and on the skins for which this fermentation is used, reserving to the present paper an account of the products of a pure cultivation of the bacteria causing the fermentation.

The cultivation used in the first experiments for this purpose was one isolated in 1889, and used in the cellulose and starch experiments described in the above-mentioned paper.

This cultivation had not been obtained from a single colony from gelatine, and in order to make quite sure that the cultures used were pure, it was decided to make another attempt to isolate the bacillus by plate cultivation. Previous attempts to do this had failed, bate organisms and gelatine liquefying bacilli developing in such numbers that the plates were spoiled before the organism, which caused the fermentation, had time to develop; besides which the organism, as obtained direct from the drenches, grew with difficulty in the ordinary nutrient gelatine. A special gelatine was therefore prepared of the following composition:—

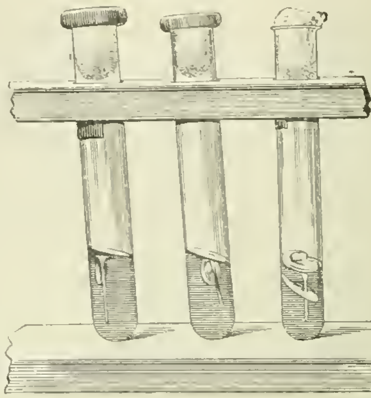
Gelatine	100 grms.
Glucose	30 „
*Salt solution	200 c.c.
Water	800 „

Plates of this gelatine in Petri dishes were prepared from the previously used supposed pure cultures which had been preserved in sealed tubes. These were found to be dead. A modification of the method previously described by one of us (this Journal, 1890, 28) was adopted.

A solution of nutrient glucose was inoculated from a working drench, and as soon as the liquid was observed to become cloudy, a tube of the solid glucose gelatine was inoculated from it by plunging in a platinum needle. In two days the bacteria developed along the needle track. Fig. 1 shows the appearance of the tube four days after inoculation, a bubble of gas being formed in the solid gelatine. On the following day the tube was broken, and from the portion where gas was given off most vigorously other tubes of solid and liquid media were inoculated. Acid was quickly formed in the nutrient glucose solutions. In the gelatine tubes, the bacteria developed well in the depth.

* Potassium phosphate, 1 grm.; magnesium sulphate, 0·2 grm.; calcium chloride, 0·1; water, 1,000 c.c. *Vide* Frankland and Frew, Trans. 1892, 255.

Fig. 1.



Cultures of and in Glucose Gelatine, showing Bubbles of Gas.

The now purified culture was passed through three more glucose gelatine tubes, each time also a glucose tube being inoculated. From the last of these tubes a very minute quantity was taken 12 hours after inoculation on the point of a platinum needle, and a streak culture made on glucose gelatine. In 24 hours a growth could be seen on the surface of the gelatine in the form of minute dots perfectly separated one from another.

From one of these dots a tube was inoculated and from this several plate cultivations were made. The colonies which developed on these plates were of two kinds, the majority being round, yellowish and of small size, a smaller number spreading out on the surface of the gelatine and slightly iridescent. These surface expansion colonies when examined with a low power appear like a milky drop, with very fine granular contents, the whole surrounded by wavy lines which follow exactly the irregular contour of the expansion. The small round colonies growing in the depth occur in the proportion of about 3 to 1 of the surface expansion colonies. The microscopic appearance of the bacteria composing the two kinds of colonies, is almost exactly similar, they are extremely small and regular in size, $0 \cdot 75\,\mu \times 0 \cdot 5\,\mu$ to $0 \cdot 7\,\mu \times 1\,\mu$. When spread upon a slide they are not readily miscible with water, and appear greasy. Both colonies inoculated into glucose tubes produced acid. The existence of these two organisms was confirmed in the following way:—A glucose tube was inoculated from a drench in active fermentation; as soon as the liquid became cloudy, a second tube was inoculated from it by means of a platinum needle; from this tube the fermentation was carried through two more tubes; a plate cultivation was made from the last tube 10 hours after inoculation. Again, the two kinds of colonies developed exactly similar in every respect to those obtained from the streak cultures.

It seems probable from these results, and also from a comparison of the fermentations made with the organisms from an actual drench (this Journal, 1893, 122), and from purified cultures with those from a single organism, which are described in the present paper, that the action in the drenches is a symbiotic one in which two or more organisms taken part.

The Fermentations.—During the time occupied by the isolation of pure cultures of the bacteria, two fermentations were conducted with the supposed pure cultures. These fermentations (or rather the second of them, for the first was unfortunately lost through the breakage of a bottle) may prove of considerable interest as throwing some light on the symbiotic action of the two organisms.

The first fermentation with pure cultures of the bacillus α (B. Furfuris) obtained from a *single* colony in glucose gelatine, was inoculated on September 16, 1894, the composition of the fermenting liquid being—

Glucose..........................	27 grms.
Peptone..........................	1·4 „
Salt solution....................	140 c.c.
Water............................	860 „
Pure calcium carbonate...........	10 grms.

This was contained in a narrow-necked litre flask fitted with a rubber stopper, and narrow delivery tube dipping under mercury, and sterilised with all the usual precautions. The fermentation began on the second day, reached its height from the 6th—8th day, and continued for 39 days, when gas ceased to come off. The examination of the gases will be described later on. When the fermentation was over, the liquid was brought to boiling temperature. It was then examined for the volatile acids in exactly the same manner as we described in our previous paper.

110 c.c. normal HCl was added and distillation commenced; the distillate was acid. The distillation was continued until the distillate ceased to be acid, forming fraction 1. Three more fractions were now distilled off, using respectively 10, 20, and 17 c.c. normal HCl. C.F. = 1·014.

The fractions were boiled with excess of $BaCO_3$, filtered, the $BaCO_3$ washed with hot water, the filtrate evaporated to dryness, and the barium salts dried at 130° C. till the weight was constant.* The salts were then decomposed with strong H_2SO_4, ignited, and the barium sulphate weighed. The following is a tabulated statement of the results:—

Fraction.	Weight of Salts.	Weight of $BaSO_4$	Per Cent. of $BaSO_4$
I.	1·2420	1·0915	87·88
II.	0·9915	0·9170	92·49
III.	1·2155	1·1980	98·56
IV.	0·6350	0·6230	98·11

Calculating fraction I. as a mixture of barium acetate and butyrate, and fractions II., III., and IV. as mixtures of barium acetate and formate,† we get:—

Fraction.	Weight of Salts.	Ba Butyrate.	Ba Acetate.	Ba Formate.
I.	1·2420	0·2630	0·9790	..
II.	0·9915	..	0·8933	0·0982
III.	1·2155	..	0·4400	0·7755
IV.	0·6350	..	0·2552	0·3798
Totals ..	4·0840	0·2630	2·5675	1·2535

Calculating the barium salts into their respective acids, we get:—

Fraction.	Formic Acid.	Acetic Acid.	Butyric Acid.
I.	..	0·4607	0·1488
II.	0·0398	0·4204	..
III.	0·3143	0·2071	..
IV.	0·1539	0·1201	..
Totals ...	0·5080	1·2083	0·1488

The total volatile acids produced amounting to 1·8651 grms.

The residual liquid containing the non-volatile acids was submitted to the test for lactic acid previously used (this Journal, 1893, 424), and it was found to be present.

The method employed for estimating lactic acid in our previous communication proving somewhat difficult, we endeavoured to improve it by extracting the concentrated solution of the non-volatile acids on prepared pumice stone with ether in a paper thimble contained in a Soxhlet fat-extraction apparatus. After repeated trials we found that this method did not give accurate results. The solution was therefore titrated with $\frac{1}{10}$th N sodium hydrate, using glazed litmus paper to determine the point of neutralisation. The acidity found corresponded to 2·438 grms. of lactic acid per 1,000 c.c. of the fermented liquid.

We have done several other fermentations with this organism and find the same acids produced and the same gases evolved, the results just given being fully confirmed. At the same time the amount of the acids produced and their proportions vary, that is to say, the quantity of acid from a given fermentation cannot be predicted with absolute accuracy, although the conditions under which we carried out the experiments were made as like as possible.

We give the total acids from four fermentations to show the amount of variation. I. is a symbiotic fermentation caused by organisms α and β; the remainder are fermentations by α alone.

Fermentation.	I.	II.	III.*	IV.
Total vol. acids, grms. per 1,000c.c.	2·4968	1·8651	0·9738	1·5636
Mean percentage $BaSO_4$ from Ba salts of vol. acids.	89·17	91·76	95·4	93·6
Lactic acid	8·9500	2·4380	1·4737	2·9700

*Comparison of Acids from Fermentation II. and III.**

——	Lactic.	Formic.	Acetic.	Butyric.
II.	2·4380	0·5080	1·2083	0·1488
III.	1·4737	0·3914	0·5593	0·0231

Fermentation III., 2,000 c.c.

Fraction.	Weight of Salts.	Weight of $BaSO_4$	Per Cent. $BaSO_4$
I.	0·7260	0·6535	90·01
II.	0·8150	0·7410	90·92
III.	2·1525	2·1342	99·15
IV.	0·3695	0·3627	98·16
V.	0·3530	0·3500	99·15

Calculation of Barium Salts as Barium Butyrate, Acetate, and Formate.

——	Ba Butyrate.	Ba Acetate.	Ba Formate.
I.	0·0597	0·0663	..
II.	0·0223	0·7927	..
III.	..	0·6662	1·4863
IV.	..	0·1469	0·2226
V.	..	0·1070	0·2440
Totals	0·0820	2·3791	1·9529

Equivalent to 0·0463 grm. butyric acid.
„ 1·1186 „ acetic „
„ 0·7828 „ formic „

or one-half of these quantities per litre of the fermented liquid.

NOTE.—Mr. Adrian J. Brown, of Burton-on-Trent, has been kind enough to examine a sample of the glucose used in the above fermentations, and found the rotary power to be equivalent to 95·6 per cent. pure dextrose. The zinc salt of the lactic acid produced had no rotary power.

The Gases.—In dealing with the gases evolved, we first compared those given off in the fermentation of glucose with that of bran under exactly similar conditions. The fermentation was conducted in open vessels as before described (this Journal, 1893, 123), and the gases were collected and examined in the same way.

* *Vide* Note on the Estimation of Butyric Acid, W. H. Willcox, J. Chem. Soc., Nov. 21, 1895.
† *Vide* J. Chem. Soc. (Trans.), 59, 94, App. II.

* 2,000 c.c. gave only about the same amount of acids as 1,000 c.c. fermentations. For percentages of acids, see table, p. 513.

Mean of three Analyses.

	Bran and Skins.	Glucose and Skins.
CO_2	25·2	24·5
O_2	2·1	1·5
H_2	46·7	49·8
N_2	26·0	24·2

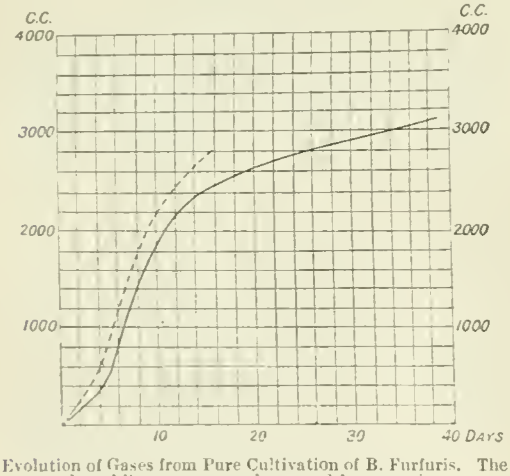
The composition of the gases is thus almost exactly similar, and, we think, fully proves our previous conclusions as to the change of the starch of the bran into glucoses by means of an unorganised ferment (cerealin).

In the closed fermentations we had previously collected only small quantities of gas over mercury, owing to the difficulty of continuously collecting large quantities which came off during the night.

In the fermentation of September 16, 1894, we collected the whole of the gas given off, taking samples every day over mercury, the gas coming off at night being collected over warm water. Of course this method does not give the total amount of gas evolved with absolute accuracy, but the exact composition of the gases was known from day to day, and the amount of CO_2 absorbed by the water could be calculated with moderate accuracy.

The fermentation was conducted in a narrow-necked litre flask fitted with a narrow delivery tube dipping under mercury, and sterilised with all the usual precautions. The temperature was maintained at 25°—30°, gas was evolved for 39 days, when it ceased to come off, the total amount collected being 3,435 c.c. One-half of this quantity, however, came off in seven days. About 300 c.c. of CO_2 was absorbed by the water during the whole period. The diagram (Fig. 4) shows the manner of evolution of the gases,

Fig. 4.



Evolution of Gases from Pure Cultivation of B. Furfuris. The dotted line represents the second fermentation.

the ordinates representing volume of gas and the abscissæ lapse of time after inoculation. The following table shows the composition of the gas at different stages of the fermentation. (The fermentation (II.) is the one of which the chemical analysis has been previously given, page 511):—

Composition of Gases evolved in Fermentation of 1,000 *c.c. Glucose with pure Ferment. September* 16, 1894.

	Day.						Mean.
	5.	9.	12.	19.	26–31.	35–39.	
CO_2	53·7	56·4	44·3	52·6	55·6	43·2	49·9
O_2	1·8	0·5	1·7	1·9	0·8	2·8	1·8
H_2	35·8	34·6	41·2	30·3	34·7	31·8	34·8
N_2	8·7	8·5	12·8	15·2	8·9	22·2	13·5

The total quantity of CO_2 actually collected = 1,563 c.c. = 3·090 grms.; the amount of CO_2 due to decomposition of the $CaCO_3$ by the acids produced was found to be 667 c.c. = (1·3189 grms.). The vol. of hydrogen collected was 1,086 c.c. = 0·973 grm.

In a second fermentation (III.) we endeavoured to ascertain the exact amount of CO_2 evolved, as in the previous fermentation this had not been done. It was therefore decided to absorb the CO_2 by means of potash.

The fermentation in this case was conducted in a narrow-necked flask of 2,000 c.c. capacity, connected by means of a narrow glass tube with two potash bulbs containing strong caustic potash, and furnished with a delivery tube dipping under water; the whole apparatus stood upon an iron plate, and was maintained at a temperature of 25°—30° in the same manner as the previous fermentation. The gases were evolved for 21 days—a considerable shorter period than the 1,000 c.c. fermentation; but resembling it in that one-half the gas was evolved in eight days. The diagram shows the curve as in the previous fermentation, which it resembles for the first 14 days, afterwards however stopping suddenly. When the fermentation was at an end the flask and contents were heated to boiling point, at the same time a current of air free from CO_2 was drawn through it, and the CO_2 given off being collected in potash bulbs as in the fermentation. Unfortunately the estimation of the CO_2 was rendered valueless owing to an accident.

The table shows the composition of the gases other than CO_2 evolved in this second fermentation.

Gases from Fermentation of 2,000 *c.c.* (*excluding* CO_2) *Fermentation III.*

	Days.						Mean.
	2—4.	4—5.	5—6.	11.	14—15.	16—17.	
O_2	3·4	2·2	1·5	0·42	0·9	2·4	1·48
H_2	81·3	83·3	82·4	79·0	71·7	72·2	77·72
N_2	15·3	14·5	16·1	20·5	27·4	25·4	20·78

The gas from days 18-21 was unfortunately mixed with air. On comparing the mean composition of gases other than CO_2 collected from both fermentations, we get the following result:—

	Fermentation, 1,000 c.c.	Fermentation, 2,000 c.c.
O_2	3·57	1·48
H_2	69·4	77·72
N_2	27·0	20·78

If now the O and part of the N in the proportion to form air be taken away, the composition of the gases from the two fermentations is found to be almost exactly similar:—

	Fermentation, 1,000 c.c.	Fermentation, 2,000 c.c.
H_2	84·4	83·9
N_2	15·6	16·1
	100·0	100·0

The gases from a third fermentation were almost exactly similar in composition, but the total volume was not measured.

A remarkable fact in this fermentation is the evolution of free N, which seems to be rare, except in the case of putrefactive organisms, as in the vast number of fermentative decompositions due to bacteria, almost the only gases found are carbonic anhydride, hydrogen, H_2S, and marsh gas.

Gayon (Schutzenberger, "Fermentation," 1876, p. 227), in 1875, in an investigation on the putrefaction of eggs, collected the gas given off from large ostrich eggs, and found

Organisms causing Bran Fermentation.
Pure Cultures.

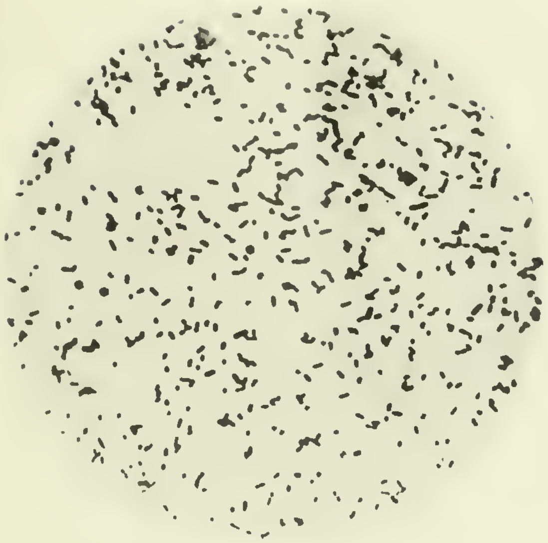


Fig. 2.
a

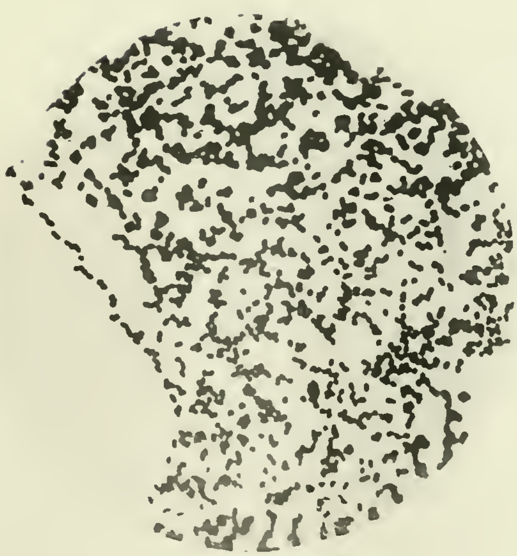


Fig. 3.
β

in it 29 per cent. of nitrogen; he adds, however, that its presence may be due to the accumulation of a certain quantity of air in the air-bubble before putrefaction.

Béchamp (Woodhead, "Bacteria and their Products," p. 125), found that yeast cells under suitable conditions, but sugar being withheld, produced pure nitrogen along with leucine tyrosin, a soluble albuminous substance coagulable by heat, an enzyme, a peculiar gummy substance, phosphates and acetic acid, alcohol and CO_2. These are almost the only instances where observers of repute have been convinced of the evolution of free N. by bacteria. We find that since the above work was carried out that Immendorf (Die Stickstoffkonservirung im Stalldünger, Jour. f. Landwirthsh. XLII. p. 69) has found certain bacteria in dung which form ammonium nitrite, and this body, as is known, splits up at a comparatively low temperature into nitrogen and water.

From the bacteriological as well as the chemical results, it is now evident that the fermentation as it takes place in practice is a symbiotic one in which two organisms play the most important part, and very probably cause the entire fermentation. This is shown by comparing the acids produced by the fermentation in the works with those produced by a mixture of the organisms α and β, the relative amounts being very close, while in all the fermentations with α alone a much less proportion of lactic acid is produced, as the following table shows:—

—	Fermentation in Works.	Fermentation α and β.	Fermentation α (II.).	Fermentation α (III.).
Formic acid......	2·8	0·8	11·8	16·0
Acetic „	22·5	16·4	27·9	22·7
Butyric „	1·2	4·5	3·5	0·9
Lactic „	73·5	78·3	56·7	60·2

The acetic acid, as far as we can ascertain, is produced directly from dextrose without the previous production of alcohol, since the presence of the latter is not shown by its tests at any stage of the fermentation. We have also ascertained that the organism is without action on dilute solutions of alcohol, in yeast water, no acid being produced.

We are indebted to Mr. H. S. Shrewsbury for the analysis of some of the gases and volatile acids, and also for the preparation of the diagrams. In conclusion we may state that the investigation of this fermentation in the tannery has been the means of pointing the way to a still more complicated process, viz., "bateing." It may even be possible in the future to place these processes on somewhat the same footing as the accurately understood fermentations in the brewing industry although the difficulties in the way are much greater.

Discussion.

Mr. James O'Sullivan asked if it had been observed whether there was any advantage in employing a bran "bate" in which the fermentation was carried on by the two bacteria mentioned in what might be termed a "practically pure" state. He was of opinion that in practice a bate was found to be more efficient when the fermentation was of a mixed character. Although alcohol had not been noticed, it was possible that the production of alcohol and its oxidation into acetic acid were simultaneous. They had an analogous case in the fermentation of maltose by saccharomyces cerevisiæ, in which, during the progress of the fermentation, there was no other sugar observed; yet, they knew that there was present in yeast a ferment capable of hydrolysing maltose into dextrose. Hence, in the case of the fermentation of maltose, the productions of dextrose and its fermentation must be simultaneous.

Dr. Clowes had no intention of criticising the authors' statement, or of questioning the authors io order to elicit further information. He wished, however, to state his conviction that the work carried out by the authors of the paper might be accepted as trustworthy. These gentlemen worked with the greatest care, and verified their first results by repetition; and there was no doubt that their results could not rest on a more sure experimental foundation. Mr. Wood was to be congratulated on the patient perseverance which he had shown in continuing his special line of research, and the Society would doubtless in future receive further instalments for publication as the research progressed. There was cause for congratulation to Mr. Wood and his co-worker, not only for their successful prosecution of their research, but also for the fact that Sir John Turney took a broad minded view respecting this most important line of investigation, and, as the responsible head of the firm with which Mr. Wood was associated, neither discouraged the prosecution of the work, nor interfered with its publication. The issue of results of this character from laboratories connected with large industrial concerns, should be considered as a voucher that operations were conducted on scientific principles, and should not only inspire confidence in the character of their products, but should also help to remove the doubt frequently expressed, as to the scientific basis underlying industrial pursuits in this country. In the present case Mr. Wood was undoubtedly one of leaders in the scientific improvements of the tanning industry, and his keen interest in the advance of the scientific side of the industry by means of research had placed him in this position, and had caused it to be generally recognised both in this country and abroad.

Dr. J. J. Sudborough asked whether the term pure culture was not used somewhat ambiguously by bacterioligsts as it served to denote two quite different forms of culture, viz., 1, a colony obtained from a single cell which must necessarily be an absolutely pure culture; 2, a so-called pure growth obtained by means of fractional cultivation, which, although in many cases probably consisting of a single species, was not to be absolutely relied upon. He further asked Mr. Wood whether they had conclusively proved the presence of formic, acetic, and butyric acids among the products of fermentation, and also whether these were the only volatile acids formed, as otherwise the numbers given were of little use. Dr. Sudborough drew attention to the recent investigations conducted by Dr. Crossley on the separation of volatile fatty acids by Wechsler's fractional neutralisation and distillation method, and asked whether the authors had experienced any difficulty in separating the three acids they mentioned. The numbers in the gas analysis tables also appeared curious, the percentage of oxygen varied without any apparent cause. The authors had not removed the air from their flasks at the beginning of the fermentation, and therefore one would expect the percentage of oxygen to gradually diminish as the air became diluted with the gases given off during the fermentation. According to the authors however (Table, p. 512) the per cent. of oxygen first diminished and then increased without any apparent cause unless leakage were suggested. But in any case the per centage of nitrogen was so great that the fact that the organism evolved nitrogen could be taken as established.

Mr. J. Golding said that as an agricultural chemist he should like to thank the authors of the paper for the work they had done. He knew of organisms that could assimilate free nitrogen, but the fact that there were other organisms capable of liberating it was new to him. Was the ether used for the lactic acid extractions dry?

Mr. F. Lott asked if the authors had tried the action of the organism on different salts? He thought the evolution of free nitrogen deserved attention; there were no doubt denitrifying organisms which might undo the work of those that aided the agriculturist.

Mr. Thomas Tyrer said that on receiving notice of the paper he read Mr. Wood's paper of 1891, and Wood and Willcox, 1893, and could only congratulate the authors on continuing such important work, pregnant with useful results to an important industry. It was pretty clear that the days of "rule of thumb" were numbered. Our industrial equality, not to say supremacy, depended upon scientific investigation, and Messrs. Wood and Willcox's work was of the kind required and should be encouraged in every possible way.

Note.—Ruge (Sitzungsber. d. Wien. Acad. d. Wiss, Vol. XLIV., 1862. 734) found that the gases of the large intestine contained 57·8 per cent. of N, and Gamgee remarks "in part a diffusate from the blood, but is certainly in part derived from the bacterial decomposition of proteids." (Gamgee, Phys. Chem., p. 467, Vol. II.)

In the forthcoming issue of the Journal would appear a report from the *Board of Trade Journal* on the German Leather Industry and Technical Schools in connection therewith, which he hoped would be carefully read.

Mr. J. T. Wood, on behalf of himself and Mr. W. H. Willcox, thanked Dr. Clowes and Mr. Tyrer for their appreciative remarks. In reply to Mr. O'Sullivan the research had shown that the best effect was produced by the action of at least two organisms working together. It was impossible to sterilize the skins as they had previously been passed through a dung bate, and, in his opinion, this was the source of the organisms in question.

They had previously pointed out that the action of the skins was due to the production of gases and organic acids. He did not know of any one organism which fulfilled the conditions so well as the two they had isolated. Possibly Mr. O'Sullivan was thinking of the dung bate, where the action was undoubtedly due to a mixture of organisms.

They had not had time to investigate the cause of failure in the lactic acid extraction, but the ether used was dry.

They had at present only tried the action of the organism *a* on dextrose and bran infusions.

In reply to Dr. Sudborough he pointed out that only in the case of yeast could a culture be made from a single selected cell. In plate cultivations made from sufficiently diluted mixtures containing only one or two species, he thought one might safely presume that the bacteria from a single colony of such a plate had sprung from a single cell. The macroscopic appearance of the colonies often helped one to decide.

He referred him to their previous paper for the tests used to show what acids were present; they never attempted to prepare the pure acids. With regard to the gases, the volume of air in the flasks and connections amounted in one case to 50—60 c.c., in another to 100 c.c., whereas the volume of gas collected was 3,000 c.c. The gases from the first two days were rejected as containing air. The amount of N was greater on the 39th day than on the 8th day. They could not explain the variation in the amount of oxygen; they simply gave their results and made no attempt to theorize.

Dr. Clowes wished, as this was the last general meeting of the Section at which Mr. Carulla would preside as Chairman, to acknowledge, on behalf of the members, their indebtedness to Mr. Carulla for his devotion to the interests and development of the Section, as well as of the general Society. By his constant presence at all their meetings, his courtesy and kindness to all, and his development of the movement for extending the membership of the Section in the Midland towns within reach of Nottingham, Mr. Carulla had rendered the term of his office memorable.

The Chairman said that as this was the last meeting of his term of office he would like to remark on the success that had attended the experiment commenced two years ago, of fixing the dates for meeting at the beginning of each Session, and trusting for papers to the good offices of the members as the months came round, irrespective of any promises when fixing the dates. It was true that they had in Dr. Clowes a reserve, or tower of strength, but as a matter of fact, although he had read several papers in the ordinary way, they had only been compelled to call on him in an emergency on one occasion. He (the Chairman) therefore heartily thanked all the readers of papers, and would only add, in conclusion, that much of the success of the past two years was due to the courtesy and diligence of their Hon. Sec., Mr. J. T. Wood. It had been a pleasure to work with him from the very commencement when they were both new to their respective offices. He was bound to express the conviction that, high as was the standing of the Society of Chemical Industry at the present time, it would be carried still higher by officers so courteous and devoted to duty as was their Hon. Sec. He heartily thanked Mr. Wood on behalf of the whole Section.

NITRIFYING ORGANISMS.

BY JOHN GOLDING.

(Lecturer in Agricultural Chemistry, University College, Nottingham.)

Mr. John Golding exhibited a series of lantern slides, photographs, water cultures and microscope slides illustrative of the assimilation of nitrogen by leguminous plants.

Mr. Golding having referred to Dr. Voelcker's paper before the London Section, went on to say that the lantern slides which he (Mr. Golding), was exhibiting were taken from plants grown at the University College, Nottingham, in pots of pure quartz sand, which pots had received only pure chemicals as food.

These slides comprised all the commoner farm crops photographed at the flowering stage. Abundant growth was shown where a complete manure containing nitrogen, phosphorus and potassium, had been added, while in the case of the cereals, rye grass, and other non-leguminous plants starvation to a more or less marked degree was demonstrated when any one of these constituents was omitted. With the leguminous plants, however, it was shown that the pot which received no combined nitrogen throve well, providing there were plenty of nodules produced on the roots.

Mr. Golding's pots of sainfoin and lucerne had failed to produce nodules though grown under precisely the same conditions as the clover, peas, and beans which were abundantly nodulated. They were, however, afterwards fertilised by being taken for exhibition purposes to Leicester.

Mr. Golding suggested that where new crops were grown in any neighbourhood the "nitragin" fertilising material should be tried.

Photographs were shown of two bean plants grown in sterile sand, in the absence of combined nitrogen, all other constituents of plant food being added. One plant was fertilised and showed abundant growth, while the other which was not fertilised had no nodules formed on the roots and was very stunted and unhealthy. The water cultures exhibited showed nodules on the roots of gorse, laburnum, clover, alsike, peas, beans, and vetches. The difference in the appearance of the nodules was very apparent. The microscope slides and micro-photographs shown, illustrated the difference in appearance of the organisms when taken direct from the nodules which they inhabit. Mr. Golding pointed out that so far as his experiments had gone, this difference vanished when the organisms were cultivated on artificial media, though they still apparently retained the power of fertilising only those plants very nearly allied to the kind from which they were obtained.

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The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Vice-Chairman:* T. J. Parker. *Committee:* H. Clementson, W. D. Horne, G. A. Prochazka, Clifford Richardson, and G. W. Thompson.

Meeting held on Friday, May 21st, 1897.

PROF. CHAS. F. CHANDLER IN THE CHAIR.

THE PRESENT STATUS OF THE HYOSCINE-SCOPOLAMINE QUESTION.

BY LOUIS MERCK, PH.D.

Hyoscine has been an object of active controversies, more so, probably, than any other one of the notable alkaloids. These controversies, after apparently becoming dormant for a time, have again and again sprung up and engaged scientists of note on opposite sides.

It was from the so-called amorphous hyoscyamine, the mixture of bases obtained from hyoscyamine seeds, that Ladenburg first isolated a substance to which he gave the name of hyoscine. The same discoverer assigned to the newly-found base the formula $C_{17}H_{23}NO_3$, thus claiming it as an isomer of atropine and hyoscyamine. Hesse, on the other hand, contended that the new base was possessed of the composition $C_{17}H_{21}NO_4$. He also declared it to be identical with the substance which E. Schmidt had isolated from scopolia atropoides and which had been named scopolamine. Subsequently, E. Schmidt found the hyoscine hydrobromate of the markets to consist almost exclusively of the hydrobromate of scopolamine. He does not, however, consider the *non-existence* of a hyoscine $C_{17}H_{23}NO_3$ to be thereby demonstrated. He argues in favour of the possibility that a base of such composition may indeed occur in the mixtures of alkaloids obtained from the solanaceæ; that Ladenburg had actually got hold of that base, which gave him the data for his formula and description of what he called "hyoscine"; and that the same base has since then happened to be isolated again.

Be that as it may, the "hyoscine" put upon the market by E. Merck has been and is assuredly identical with scopolamine. It is only for the purpose of obviating misunderstandings in commercial and medical circles, where a change of name is apt to create great confusion, that our house has retained in its trade-list the designation of "hyoscine" for the base from hyoscyamus, while applying that of "scopolamine" to the base from scopolia atropoides. Since the opening of this controversy the Merck laboratories have, in working considerable quantities of the solanaceous drugs for alkaloids, steadily been directed toward the object of identifying a base $C_{17}H_{23}NO_3$, which is to possess the properties of hyoscine. So far, however, we have not succeeded in isolating such a base.

In the course of these labours we were indeed fortunate enough to isolate a base $C_{17}H_{23}NO_3$, thus isomeric with hyoscyamine and atropine, from duboisia myoporoides. This base we have named "pseudo-hyoscyamine." Its properties, however, differ essentially from those known for hyoscine. For instance, hyoscine is an oily liquid, while pseudo-hyoscyamine melts at about 132° to 134°. Thus, the possibility of any confusion between these two appears wholly excluded. The base isolated from Duboisia myoporoides by J. Gadamer (albeit in quantity insufficient for exact determination) is equally unlikely to represent Ladenburg's "hyoscine," inasmuch as Gadamer, from his analytic results, concludes that it contains but 15 atoms of carbon. Our own investigations at the works covered these solanaceous drugs: Belladonna, duboisia, datura, stramonium, hyoscyamus, scopolia.

On examining even very large quantities of the bases residuary from the manufacture of atropine, we have so far encountered no substance which would even approximately be capable of identification with the "hyoscine $C_{17}H_{23}NO_3$." Furthermore, repeated efforts were made by us to isolate all the alkaloids from duboisia. All that was obtained on such occasions was: hyoscyamine, hyoscine (or scopolamine) $C_{17}H_{21}NO_4$, considerable quantities of amorphous bases, and pseudo-hyoscyamine. Never was an alkaloid met with in this work answering to the formula given by Ladenburg for hyoscine, and at the same time possessing its characteristic properties regarding salts and double salts—properties which would have rendered such an alkaloid incapable of being overlooked. The same negative result was had from investigation of the alkaloids of stramonium seeds, which, likewise, were worked in quantities of thousands of kilogrammes. The Merck laboratories are also continually working for alkaloids, large quantities of hyoscyamus seeds, and scopolia roots. Also in these lines of manufacture we constantly kept our attention directed toward isolating the alkaloids occurring among the residuary bases. Nevertheless, the search for the particular base here in question has proved equally fruitless on these two drugs.

In this connection I may be pardoned the liberty of alluding to still another interesting fact developed at our laboratories. The statement is frequently met with in literature, that hyoscine (that is, scopolamine) has been isolated from the residual bases obtained in the manufacture of atropine. Therefore, it might readily be inferred that hyoscine is a side-base to hyoscyamine or atropine in the belladonna. We have studiously kept this issue in view during the working of many hundreds of thousands of kilos. of belladonna roots. And still we have never been able to discover hyoscine (meaning scopolamine) among the residual bases resulting from these extensive operations. This experience of ours thus directly contradicts the inference before mentioned. The experiences of others, as quoted before, may be presumed to have been due to defective sorting of the belladonna roots, among which may have remained roots of other solanaceæ. Or, the residues remaining from the manufacture of various solanaceous alkaloids, and utilised for obtaining side-bases, may not have been kept absolutely apart.

It may be contended that, in the Merck researches, the hyoscine or scopolamine that might have been present could have been overlooked in consequence of its own minimal quantity. This contention, however, is met by the fact that the mixtures of residual bases were, at the end, also subjected to splitting; whereupon the appearance of scopoline among the products of this operation must have indicated that hyoscine had been present, if such were indeed the case. From a single batch, for instance, of residual bases thus treated, 100 kilos. of tropine were obtained, whereas the presence of a higher-boiling base was not demonstrable.

To return, after this little digression, to my main topic, I would once more summarise our laboratories' experiences regarding the latter, to wit: With due consideration of the various solanaceous drugs, and with the use of very considerable quantities of them, the results so far have failed to show the possibility of obtaining a base possessing the properties of hyoscine and answering to Ladenburg's formula of $C_{17}N_{23}O_3$. Thus, this whole question has, by this time dwindled down almost exclusively to a controversy between O. Hesse and E. Schmidt as to the propriety of designating the surely-established alkaloid $C_{17}H_{21}NO_4$, either as "scopolamine" or as "hyoscine."

The polemics between the two investigators named have, however, recently extended into still another chapter of the solanaceæ-alkaloid research, which bears close relations to to the one I have here discussed. O. Hesse published an observation made by him in the scopolamine hydrobromate of commerce, to this effect:—He frequently found this salt to contain also notable quantities of another base, differing from scopolamine by the absence of optical rotatory power. This would, of course, have the result that such a mixed salt would show a smaller arc of rotation than that due to the pure scopolamine hydrobromate.

He succeeded in isolating this inactive base, and gave it the name of "atroscine." Hesse argues against any supposition of identity as between his "atroscine" and the so-called "inactive scopolamine," found by E. Schmidt. His reasons herefor are two:—Firstly, he did not succeed in obtaining, by the treatment of hyoscine with alkalis, an inactive base of equal composition. Secondly, He claimed on the other hand, that the decrease in optical activity resulting in hyoscine upon treatment with alkalis is due altogether to a splitting of the hyoscine into a "split-base" which he calls "oscine," and tropic or atropic acids. O. Hesse's "oscine" is stated as being identical with E. Schmidt's split-base "scopoline."

E. Schmidt, again, gives the genesis of his "inactive scopolamine" as being likewise brought about through the action of alkalis on scopolamine; with the additional statement that it can also be produced by using silver oxide instead of the alkalis. He states that he never encountered any "atroscine" in the process of isolating alkaloids from commercial scopolia roots or hyoscyamus seeds. Schmidt further explains that accordingly as the liquors from scopolia root are treated with less or more intensively-acting alkalis one may obtain at will either a normal scopolamine (rotating about 24 to 25°), or a feebly-rotating one. He succeeded even in directly obtaining, from one and the same scopolia root, a normally (that is, strongly) rotating and a feebly-rotating scopolamine. The former was obtained by alkalinising the liquors with sodium bicarbonate or ammonia; the other by employing strong bases, such as soda-lye, concentrated solution of potassa, &c.

It is thus still an open question, whether or not Hesse's "atroscine" should be regarded as identical with Schmidt's "inactive scopolamine." Upon considering the contradictory verdicts of medical authorities who have tested both these substances as to their action on the eye, one might almost incline toward the second alternative; that is, non-identity.

O. Hesse, in his treatise, quotes medical councillor Dr. Königshöfer's conclusions from physiological tests made by him with atroscine, to the effect that its action differs in certain points from that of scopolamine. The passage quoted reads as follows: "We thus find that this remedy" (that is, atroscine) "acts identically with atropine and scopolamine, in so far as its mydriatic effect is concerned; while in the matter of paralysing the accommodation it considerably surpasses both these substances in promptness of action as well as in duration of effect; the ratio of difference ranging from double to quadruple."

E. Schmidt, on the other hand, reports that Prof. Dr. Uhthoff, Director of the Eye Clinic at the University of Marburg, in conjunction with Dr. Axenfeld, Lecturer, arrived at the following important conclusion:—In comparative tests with a strongly rotating scopolamine hydrobromate (25·43°) and a very feebly rotating salt (6·62°), ". . . . . . . it was found that no difference whatever could be shown between the effects of these two salts."

To these contradictory reports published by Schmidt and by Hesse, I can add the following facts gathered from observations made by us at the Darmstadt Laboratory. Ever since this property of hyoscine, of strongly rotating the plane of polarisation, has been known, special regard has been given to this point at our laboratories in working the side-bases from hyoscyamus seeds for hyoscine. The product of each separate batch was invariably examined for the determination of its optical properties. In the course of these observations it was found that we always obtained a hyoscine of normal rotatory power with but very slight variations, that is, ranging between about 24 and 25 degrees. We have been unable so far to discover a feebly rotating preparation obtained from hyoscyamus seeds.

In the manufacture of scopolamine from scopolia roots, we made the optical rotatory power a subject of special research only quite recently; and in consequence I can give you the results only on an amount of scopolamine obtained from about 10,000 kilos. of the roots. The scopolamine hydrobromate from this quantity, on being crystallised, was shown to be absolutely pure, but to possess a lævo-rotatory power of only 13·47°.

In connection herewith I should state that, in working the scopolia roots and in working up the side-bases for scopolamine, exactly the same stages were adhered to, as in working the hyoscyamus seeds and in working up their side-bases for hyoscine. Especially in the manner of using alkali in both series, the most punctilious care and attention were exercised in order to make the conditions in both exactly equal. Hence, I cannot well refrain from the conclusion that the alkali can hardly have caused the difference in rotatory power in these cases of ours; for, had it done so, a more feebly rotating product must have resulted in the isolation of the hyoscine as well as in the other case. This experience seems to me to argue largely in favour of O. Hesse's view, that two chemically equal basic substances, which so far can be distinguished from one another only by their optical activities, are contained in the ordinary scopolamine hydrobromate; and that, quite possibly, they exist performed already in the scopolia root.

The few observations so far available cannot, of course, suffice to settle the pending controversy. But it affords me gratification to be in a position to promise the early publication of further results from considerably larger batches of scopolia roots, in which likewise the various results will be most carefully noted; which, I hope, may contribute toward the further elucidation of this question.

THE MANUFACTURE AND APPLICATIONS OF LACTIC ACID.

BY ALAN A. CLAFLIN.

This paper consists of extract from notes on observations taken during seven years' experience in the manufacture of lactic acid on a large scale on lines laid down by my predecessor, Mr. Chas. E. Avery. While some of my facts are not new, considerable work having been done very recently on this subject; and while none of my investigations have been carried farther than the daily exigencies of manufacture required, yet I trust that from the exceptional opportunities that I have had, they may be found of interest.

The lactic acid industry dates back about 20 years, when Mr. Avery began his investigation, which resulted in the process which he covered from 1881 to 1885 by American and foreign patents. The production of lactic acid on a large scale by fermentation is interesting because it employs micro-organisms to split up the glucose molecule into two molecules of ethylidene lactic acid. The micro-organism that does this work is well-known as the bacillus acidi lactici. This bacillus has been described variously. Not from any inacuracy of the observer, but because it is of great variety itself, a certain variety developing most rapidly in a certain medium. The bacillus which does the work in a highly nitrogenous saccharine solution is a large species. Its form is a double truncated cone, averaging in length 2 to $2\frac{1}{2}$ mm., and about 1 mm. in diameter. It has the tendency of all lactic bacteria to link itself together in pairs or short chains. The most abundant lactic bacteria in sour milk are only $\frac{8}{10}$ mm. long, but a little thicker in proportion than those found in my saccharine solutions.

The manufacture of lactic acid has three divisions:—The preparation of the saccharine solution, the fermentation, the conversion of the fermented liquor into commercial lactic acid. The source from which the saccharine solution is obtained is not important. The location of the factory determines the raw material that can furnish a glucose liquor most cheaply. The proportional composition of the saccharine solution is very important. For complete decomposition the saccharine solution should not vary between the limit 1·05 to 1·075 sp. gr.; for technical reasons the higher density is preferable. A solution of that density will contain from $7\frac{1}{2}$ to 11 per cent. of saccharine matter. It is not essential to have all the saccharine matter in the form of grape sugar. In fact, it seems to be advantageous to have 10 to 15 per cent. of it as cane-sugar, which becoming inverted in the process, furnishes fresh material, as it were, for the ferment. To do its work thoroughly, the lactic bacillus needs be well nourished by nitrogenous matter. The most convenient form for this nitrogenous material is as it comes from vegetable life, as extracted, for instance, from bran by the action of boiling water and dilute acid. At different times I have used material obtained from animal and mineral sources. In the laboratory it is difficult to detect material differences. On the large scale, I prefer a vegetable source; there seems to be a complete utilisation of the material. The amount of nitrogenous material should be at least 8 per cent. of the saccharine constituents. I assume by nitrogenous matter a body of substantially the composition of albumin containing 15 to 20 per cent. of nitrogen in complex form. Perhaps it would be more accurate to say the total nitrogen should be nearly 2 per cent. of the saccharine material, and preferably combined

with carbon. If a mineral food is to be employed, ammonia salts should be in larger proportion than nitrates. A low percentage of phosphates will suffice, and the presence of much potassium does not seem to be essential.

The requisite saccharine solution made up and boiled for at least an hour, to make sure of sterilisation, is conveyed into the fermentation tank. There it is rapidly cooled to 55° C. or lower, not going below 45°, and impregnated. Impregnation takes place at a higher temperature than observers have generally stated, a peculiarity perhaps arising from the large scale at which the work is carried on. I may state here, in a general way, that the modified conditions caused by working the bacteria on a large scale, give an opportunity for variation from laboratory results. A large body of fermentable liquor is less susceptible to foreign spores, more energetic in its development, and productive of more obvious results than a solution such as is usually found in the laboratory. Consequently I feel that the chemist, who is to investigate and utilise, to the fullest extent nature's great oxidising agent, the mycoderma aceti, and nature's great reducing agent, the bacillus butyricі, must be as familiar with them in a 5,000 gallon vat as in the pure culture state in a test tube.

In continuous manufacture the ferment solutions are impregnated from a preceding ferment liquor, in which a lively fermentation is in progress. Decided advantage is obtained by using a generous amount of such liquor. 20 per cent. is none too much. For an original impregnation, lactic bacteria must be obtained from an outside source. I prefer to use for that source, milk that has been allowed to stand at a temperature of 45° until slightly sour. If the milk is allowed to sour too long, before using as an impregnating fluid, a tendency is shown for butyric bacilli to develop. This same objection is true if rotten cheese is used. Should the saccharine solution be allowed to ferment spontaneously, everything may happen or nothing. A good lactic fermentation may develop, a meagre alcoholic, or a meagre viscous, a little butyric, or a little of each, and very much butyric, the last of which is most probable. The best original impregnation I ever had was obtained in the following manner :—A pure culture of the lactic bacillus was obtained from the bacteria in a ferment tank, and preserved. Previously sterilised milk was impregnated from this culture, and kept under pure culture conditions for a day, and then used with noticeable advantage over the spontaneously soured milk.

The impregnation accomplished in the saccharine solution, which should be neutral or faintly acid, not alkaline, the main process in the manufacture of lactic acid is begun. The success or failure in the manufacture depends wholly in the management of the fermentation. For economical production, over 90 per cent. of the glucose must be converted into lactic acid. Any unconverted glucose works a double injury, not only being a loss of material, but also making the resultant lactic acid liable to subsequent decompositions. In our factory to-day we have practically no residue of undecomposed glucose, and the yield of lactic acid is over 98 per cent. As I have said before, the impregnation takes place at 45° or above, and from that point the temperature is allowed to decrease somewhat as the fermentation solution grows older. If it is desirable, as it usually is, to have the fermentation proceed briskly, the temperature must be higher than when, as is occasionally the case, it is desired to have a slow fermentation. It should be borne in mind, however, that a lactic fermentation of itself gives out considerable heat; the more so if quick running. As the fermentation progresses, the solution must be neutralised with milk of lime, or chalk in suspension. The limits of acidity in which lactic bacteria are healthy are rigidly confined between 0·02 and 0·5 of 1 per cent. Keeping the acidity of the fermentation between these limits is the main preventative against butyric fermentation, as the regulating of the temperature is the preventative against alcoholic fermentation. If the fermenting solution is over neutralised, the butyric ferment will at once begin to act, and once active is very difficult to control. It is the greatest enemy to the lactic fermentation, destroying the lactic acid that has been made, and devouring new quantities as fast as they can be produced. In 24 hours one-third of the lactic acid may be destroyed by the butyric bacillus. Anti-ferments cannot be used with much safety in lactic fermentation. The lactic bacillus is more susceptible than other ferments, and is killed while others are only stagnated. Mustard-seed oil is about the only exception that I know. This has apparently a deadening effect on butyric fermentation, with no serious effect on the lactic fermentation. The lactic fermentation is best completed in from three to six days, although its life may be prolonged up to a fortnight. Any prolongation of life is attended with the danger of butyric invasion. When the fermentation is ended, the liquor must be heated sharply to kill all bacteria and spores and prevent subsequent fermentation.

The fermented liquor, which is now a solution of calcium lactate, with a multitude of dead lactic bacteria floating in it, is filtered and evaporated. If a pure lactic acid is desired, the calcium lactate is allowed to crystallise and purified by repeated crystallisation. For a commercial lactic acid, decomposition by sulphuric acid of the dense uncrystallised solution of calcium lactate gives an acid of sufficient purity. The free acid obtained from this decomposition is further concentrated to such strength as the market demands. In evaporating free lactic acid there is danger of forming lactic anhydride. It is possible to have conditions such that the longer evaporation takes place the less percentage of lactic acid is obtained. In concentrated solutions, very little lactic acid is lost by volatilisation. In dilute solutions much acid may be mechanically carried away with steam, especially if a blast is used. A 50 per cent. solution is about as concentrated as it is economical to manufacture. This appears on the market as a syrupy liquid, with more or less brown colour, and a specific gravity of 1·20, and contains about 7 per cent. lactic anhydride.

The applications of lactic acid to-day, while limited, are increasing. The majority of all that is produced is used by the woollen dyer as an assistant in mordanting with bichromate of potassium. For this purpose I believe it has advantages which the technical world is beginning to appreciate. Without discussing at length the comparative merits of tartaric, oxalic, and lactic acids, I will just mention the advantages which are claimed for lactic acid, namely, greater reducing power, greater solubility of itself and its salts, and less corrosive action. The first claimed advantage the greater reducing power of lactic acid towards chromium salts and chromic acid, is generally admitted in comparison with oxalic acid, and also in the case of tartaric acid, although the fact that tartaric acid reduces chromium salts more quickly, has led to some discussion. Admitted that lactic acid has the greater reducing power, the value of this property in the dye-bath is not yet absolute, as with many colouring matters I do not find that the best results are obtained with the chromium reduced to its lowest terms. Yet, if lactic acid does the most economical reducing, it is but a question of correct proportioning of the recipe, to obtain the desired shade at the lowest cost. The second and third advantages—greater solubility and less corrosive action—are unquestioned. In corroboration of my estimate of the value of lactic acid in the woollen industry, the following statistics of lactic acid are submitted. In 1894 the dyehouses of the United States and Canada used about 100,000 lb. lactic acid, while none was used in England or Europe; in 1895 there was a domestic consumption of 1,000,000 lb., and a foreign consumption of 500,000 lb.; in 1896 the domestic consumption was 1,200,000 lb. and the foreign 1,000,000 lb.

Lactic acid is used to a very limited extent in the calico printing industry. Some discharge effects can be obtained advantageously with it. In most instances, however, the hygroscopic nature of the acid is injurious in its effect on colours which have to be steamed.

Next to the woollen industry the most important application for lactic acid is in the preparation of hides for tanning. The efficiency of the bran drench for removing the lime from the skin, and making it porous and in good condition for receiving the tannage, is due to the lactic acid produced by the fermentation of the bran. A dilute solution of lactic acid will do this work as well, and is much easier

to control in its action. The slightly higher cost is more than compensated for by the prevention of waste. To the researches of Mr. Wood (J. T. Wood, this Journal, 1885, 27 *et seq.*) and Mr. Andreasch (F. Andreasch, Der Gerber 21, 506; 22, 513) this application is attributable. In America over 300,000 lb. of lactic acid have been consumed, mainly by a few tanneries, during the last six months.

In the household lactic acid is used medicinally, and is a substitute for other acids and fruit juices in making acid beverages. The large possible application of lactic acid for domestic use—the replacing of cream of tartar by the acid lactate of calcium—has not yet been put in practical operation.

With regard to the analysis of lactic acid, I feel I can add little to what Allen has collected in the latest volume of his Organic Analysis, (A. H. Allen, Commercial Organic Analysis, Vol. III., Part III., 411 *et seq.*). On account of the solubility of all its salts, the direct determination of lactic acid is a long and delicate process. Generally the indirect method—determining total acidity and percentage of foreign acids—gives most expeditious results. There is not much sophistication of lactic acid. Sometimes acetic and mineral acids are found in it, and now and then samples of so-called lactic acid containing no lactic acid at all. Usually, however, commercial lactic acid is pure, except from products incident to manufacture and not deleterious in effect. The strength varies from 20 to 50 per cent., according to the price and for what purpose the acid is intended to be used.

Discussion.

Dr. L. Reuter asked whether ether or alcohol was used to separate the lactic acid from the mixture of calcium lactate and sulphuric acid.

Mr. Claflin said that the bulk of the sulphuric acid was neutralised by the calcium of the calcium lactate, calcium sulphate being practically insoluble in lactic acid. No ether or alcohol was used. The solution of lactic acid was simply evaporated. In reply to a question from Dr. G. A. Prochazka, for percentages higher than 30 per cent. it was advantageous to evaporate *in vacuo*; for less percentages air would do perfectly well. Copper vessels were employed for evaporating.

In reply to a question whether any difference in the fermentation from the amount of electricity in the air was observed, on the principle of thunder storms making milk sour, the author stated he had never noticed any difference, because the ferment solutions were well protected from atmospheric influences and kept at a uniform temperature. Lactic acid made by evaporation had more or less colour according to circumstances. It contained no sulphuric acid or sulphate of lime. Baryta was used to remove any traces of sulphuric acid, although sulphate of lime was very nearly insoluble in concentrated solutions of lactic acid. The temperature employed was about 115° C.

Mr. R. C. Woodcock asked in what form the nitrogenous matter, intended to act as food for the bacteria, was added to the solution—whether as ammonia salt or as albumenoids. And what was the final condition of this nitrogen in the solution after the fermentation germ had done its work.

Mr. Claflin said the nitrogen seemed to be taken up by the ferments themselves; for when, after fermentation, the solution was filtered and the dead bacteria collected and analysed, there was as much nitrogen in them as in the original solution.

The Chairman said that some interesting investigations had recently been made in Boston with regard to the bacteria in milk, and it was found that by the use of special precautions milk free from bacteria could be drawn from the udder of the cow, showing that the milk in the cow was free from bacteria, but in the process of milking became impregnated. The cow was covered with dust, which was full of germs, so that in the ordinary course the milk in the pail became impregnated as soon as it was drawn from the cow, and in many cases more bacteria had been found in milk as delivered than there were in an equal quantity of sewage, the milk favouring the rapid development of the bacteria.

THE DETECTION OF ARSENIC AND ANTIMONY, EITHER ALONE OR IN MIXTURE.

BY P. H. CONRADSON.

In 1889 there appeared in the "Druggists' Circular and Chemical Gazette" an article setting forth the sensitiveness of the well-known Hager's silver nitrate test for arsenic (evolution of arseniuretted hydrogen) with zinc and dilute sulphuric acid, letting the evolved gas act upon filter-paper moistened with nitrate of silver solution. If arsenic is present the silver paper blackens. Although the nitrate of silver paper is all that can be desired as regards delicacy for arsenic, still the test in the presence of sulphur and antimony is not certain, neither does it fully decide whether antimony might be absent or present. On this ground I set about to devise a simple test for the detection of antimony, whether alone or in mixture with arsenic. After a good deal of study on the subject, I found the following indicator, or test paper (which I will call "nitro-iodine paper") to work very satisfactorily indeed.

The material to be tested for arsenic or antimony, or both—such as colours, paper-hangings, fabrics, or other organic or inorganic matter—is treated, according to Hager's method, with concentrated solution of soda nitrate, dried, and burnt upon a porcelain saucer, residue boiled with potash solution in excess, filtered, and acidified with dilute sulphuric acid, treated with permanganate of potash, to destroy any organic matter that still may be present; or the material to be tested may be treated in any other suitable or well-known manner to insure the solution of the arsenic and antimony compound.

After the material to be tested has been dissolved in sulphuric acid, the following procedure and precautions have been found to work satisfactorily:—

Four test papers are used, freshly prepared and still moist. Nos. 1, 2, and 3 are prepared by placing a drop or two of lead acetate, silver nitrate, and mercury chloride solutions respectively on the middle of a piece of filter paper, large enough to cover the test-tube. No. 4 is prepared by putting two drops of nitric acid (sp. gr. 1·20) on the middle of a piece of paper, allowing it to spread, and then placing two drops of potassium iodide solution (1 in 10) on the middle of the nitric acid spot. All these test papers should be prepared just before using, and in order, as described later on.

The apparatus used is an ordinary test-tube, about 8 ins. long and 1½ ins. in diam., with some cotton, granulated zinc (free from sulphur, arsenic, and antimony), and dilute sulphuric acid (also free from arsenic and antimony). In place of the granulated zinc, pure magnesium ribbon can be used with great satisfaction.

The test is made as follows:—Put some of the pure zinc in the test-tube, add some dilute sulphuric acid, insert a loose cotton plug, and lay test paper No. 1 (lead paper) on the top of the test-tube. If the paper does not become dark coloured after 10 to 15 minutes the zinc is free from sulphur, or if the paper should darken somewhat after, say, 5 minutes, then take a fresh paper from time to time until the lead paper is not coloured. I have found that zinc containing a little sulphur may give quite a strong reaction at first, but after 5 to 10 minutes it hardly shows any sign of sulphur. Of course, should the generated hydrogen after 15 to 20 minutes still show a reaction for sulphur on the lead paper, the zinc or acid, or both, are unfit. As soon as no reaction on paper No. 1 is shown, the silver paper No. 2 is inserted; should no coloration take place after 15 minutes, the zinc and the acid may be considered all right for most cases.

Of course if the acid and zinc are known to be free from sulphur and arsenic, the above preliminary tests can be omitted; however, it is always advisable to make these two preliminary tests, as I have found from experience, using granulated zinc, that some lumps are free from sulphur and arsenic, and others from the same lot contain these impurities.

Now some of the suspected material in solution is added, but not too much at a time. The gas should never be generated too fast, or the papers will not give so distinct shades or colours. Then insert test paper No. 4. If no reac-

tion appears, the material is free from sulphur. Then insert No. 2 silver paper. If there is no coloration, even after some time (15 to 20 minutes), the material is apparently free from arsenic and antimony. Should a coloration appear on the test paper, then either arsenic or antimony, or both, may be present. In that case remove the silver paper and insert paper No. 3—the mercury chloride paper. If arsenic alone be present, a pure lemon-yellow spot appears, which gradually deepens to a pale yellowish-brown colour. This reaction has to be watched carefully, as the yellow colour disappears rather fast if too violent generation of the gas is allowed, or much arsenic should be present. Antimoniuretted hydrogen produces on this paper a brownish-grey spot, or colour with no yellow in it and quite distinct from the arsenial coloration. If no yellow colour should appear on this paper at first, but a grey or brown colour without any yellow tint, especially round the border of the moistened paper, arsenic may be considered absent, but antimony is probably present.

Of course it should be borne in mind that the test with each paper should at least continue 15 minutes, with a rather slow rate of evolution of gas. If it has taken 10 or 15 minutes to show a coloration on the silver paper No. 2, in case there should be no coloration of any kind after 15 minutes on the mercury chloride paper No. 3, it will be advisable to add to the test-tube a small quantity of the solution to be examined. It is, therefore, advisable to start with quite a quantity of granulated zinc.

After applying the mercury chloride paper, if no coloration appears or a clear lemon-yellow coloration, antimony is probably absent. To positively verify the absence of antimony, apply test paper No. 4—the nitro-iodine paper, prepared as above described. If antimony is present, first a clear bright yellow colour is produced on the paper, which sometimes darkens to deep yellow, with appearance of orange crum coloured spots on the edges: this reaction is very distinct and characteristic for antimony. Arsenic does not interfere with this colour produced by antimony on paper No. 4, neither does pure hydrogen. The bluish colour, or tint produced when potassium iodide comes in contact with nitric acid on the paper, disappears before the yellow colour is produced by the antimony.

If magnesium ribbons are used in place of granulated zinc, it is very easy to regulate the amount of gas generated, simply by dipping the ribbons to a greater or less extent into the acid solution.

Recapitulation.

Colours Produced on the Different Test Papers.

No. 1. Lead paper.—Sulphur produces brown to black colours.

No. 2. Silver paper.—Arsenic or antimony, brown to black tints.

No. 3. Mercury paper.—Arsenic, yellow to yellowish-brown.

No. 3. Mercury paper.—Arsenic and antimony, varied tints, yellowish to brown.

No. 4. Nitro-iodine paper.—Antimony, bright yellow to orange crum yellow.

No. 4. Nitro-iodine paper.—Arsenic, no colour.

No. 4. Nitro-iodine paper.—Hydrogen, no colour.

Discussion.

The Chairman said that when he had had occasion to make tests for the detection of arsenic and antimony he had always preferred to pass the gases given off by zinc and sulphuric acid into a solution of nitrate of silver. The arsenic went into solution, precipitating metallic silver, and the antimony formed antimonide of silver, which went to the bottom as a precipitate. This made a complete separation, giving a solution containing arsenic and a precipitate containing antimony, which, upon filtration, gave the antimony on the filter and the arsenic in the filtrate. One had only to cautiously neutralise the filtrate by dilute ammonia to get a yellow precipitate of arsenite of silver; and, after washing the black precipitate of silver and antimonide of silver, and boiling it up with a little tartaric acid and a few drops of hydrochloric acid, a solution of antimony was obtained, which immediately gave a beautiful orange precipitate with sulphuretted hydrogen. Thus, one saw the products in the form of yellow arsenite of silver and orange sulphide of antimony. He liked to see the precipitates, because one then got a tolerable idea of relative proportions.

NOTE ON TURPENTINE OIL AND ITS SUBSTITUTES.

BY F. H. CONRADSON.

A turpentine oil was received, called "wood turpentine." It was clear, perfectly water-white, with an odour towards terebene. Its specific gravity was 0·845, flashing point below 80° F., burning point 94° F., and on evaporation left nothing—at least, nothing resinous. From the low specific gravity and low fire-test, it was apparently not pure oil of turpentine, but a mixture of either rosin spirit or benzine, or both, with ordinary oil of turpentine.

Distillation, according to Allen, was tried, and the distillate repeatedly treated with sulphuric acid according to Allen's direction; but I could not separate the fractional distillates in such a way as to distinctly say whether all the impurity or adulterant was rosin spirit or benzine (kerosene).

On treatment with sulphuric acid (H_2SO_4) the spurious turpentine became heated only a few degrees after vigorous agitation; on the other hand, pure turpentine, and also so-called fernoline turpentine, heated very considerably, and had to be cooled down with water. This difference in rise of temperature confirmed the belief that the oil in question was a mixture. Allen's test with castor oil was tried, both with pure oil and mixed oil (turpentine and benzine), but no separation took place.

Nothing definite could be made of the permanganate test. I happened to shake some rosin spirit I was distilling with a strong solution of SO_2 in water, and found that the rosin spirit was immediately coloured a beautiful yellow, the aqueous layer beneath being clear. I tried the same with pure turpentine oil, and this oil did not colour at all, or only slightly. The wood turpentine was next tried, and gave the same yellow colour as the rosin spirit. Benzine, gasoline, and 160° oil gave no coloration. With fernoline turpentine considerable heat was evolved, and on shaking a white emulsion was formed, which, on standing, separated into a brownish under layer and a yellowish-white upper layer. This sample of fernoline turpentine had been in the laboratory about eight months in a partly-filled bottle, often open. When the oil first came it left, on evaporation, less than 0·75 per cent. of residue, but now leaves a considerable amount of sticky residue, showing that it has undergone partial oxidation.

This yellow colour produced by SO_2 on rosin spirit, and not on pure turpentine, may be recorded. Further, some rosin was dissolved in the pure oil and treated with SO_2; about the same coloration occurred as in the rosin spirit.

The spurious turpentine and the rosin spirit in SO_2 water treatment, after standing several hours, commenced to change colour on the surface, namely, brown towards blue; and on standing over night, the yellow colour was gone, and the liquid had become a reddish to a bluish-brown. The pure turpentine did not perceptibly change its first appearance. The turpentine to which rosin had been added did not change colour, but in the main kept its yellow colour.

A sample of pure turpentine oil, water-white, clear, and leaving on evaporation only a small residue (Standard Oil Co.'s make), has a specific gravity of 0·870; other sample, one of 0·867. Flash in open cup, 100° F.; burn, 110° F.

A mixture of turpentine oil of 0·870 sp. gr., volume 90 per cent., and 150 oil of 0·794 sp. gr., volume 10 per cent. gave 0·861 sp. gr.

A mixture of turpentine oil of 0·870 sp. gr., volume 90 per cent., and benzine of 0·718 sp. gr., volume 10 per cent., gave 0·8544 sp. gr.

The mixture, with 10 per cent. of benzine, flashed at the ordinary temperature (70° F.), so if 10 per cent. or more of naphtha or benzine is present, the lower specific gravity as well as low flashing point is sufficient indication of

adulteration (if no rosin spirit should be present). The flashing point of the mixture with 150° oil would, of course, be as high, or higher, than for pure oil of turpentine.

There are several tests given from time to time for the detection of petroleum oil in oil of turpentine, and the following one, in addition, may be of some use because of its simplicity, as well as surety, for a qualitative test at least. Pure oil of turpentine, say 50 c.c., is evaporated down to 1 or 2 c.c. on a water- or steam-bath, the residue (usually rather thick) carefully poured into a perfectly dry test-tube, and from 5 to 10 c.c. pure acetic acid (glacial) 99·5 per cent. used in the test, and the mixture shaken. If the oil be pure the liquid remains perfectly clear, no separation taking place. If the oil contain as small an amount of kerosene as 10 per cent., the mixture, on shaking, becomes cloudy or milky, and on standing for an hour or more, the petroleum separates on the top. Glacial acetic acid (99·5 per cent.) dissolves a mixture of turpentine oil and petroleum oils very readily up to 30 or 40 per cent. of adulterant, hence the necessity to evaporate the turpentine oil down to the prescribed volume (see this Journal, 1890, 1005).

Discussion.

Mr. G. W. Thompson said there was a recent communication in one of the Journals referring to the presence of oxidation substances in turpentine, having the nature and reactions of aldehyde, which could be removed by treating the turpentine with an alkaline sulphate or bi-sulphate. Something similar to that reaction might take place in the author's treatment of the rosin spirit, or oil containing rosin spirit, with sulphurous acid. He himself had wished to make use of the test referred to in the communication, by treating turpentine with an alkaline bi-sulphite, but had been unsuccessful. If anyone present could offer any suggestion on that point he would be very much obliged, because in the use of turpentine for varnishes there were very peculiar reactions between turpentine and manganese salts. For instance, if oleate of manganese, as pure as they could make it, were added to ordinary turpentine the solution became heated and darkened in colour. There seemed to be some oxidation of oleate by the turpentine. They knew turpentine had the power of absorbing ozone and oxygen, and in this case it seemed to give it up to the manganese. He wanted to find out if it were an aldehyde substance that produced this action.

Mr. Woodcock said that if the oxidation of turpentine were carried on at about 120° F., after the turpentine had absorbed certain quantities of oxygen, there were large quantities of camphoric peroxide formed. That was a very peculiar product, inasmuch as under certain conditions it decomposed, giving off peroxide of hydrogen, and leaving a residue consisting chiefly of soluble camphor.

PRACTICAL HINTS IN LIMESTONE ANALYSIS.

BY K. J. SUNDSTROM.

In manufacturing bicarbonate of soda the writer had to use limestone, varying in composition with every cart load, and it was therefore an imperative necessity to analyse it quickly and correctly. A gas analysis could be made speedily, but gave no idea of the proportions between lime and magnesia, while the limestone should not contain at most more than 5 per cent. $MgCO_3$, not over 1·5 per cent. SiO_2, and only traces of Al_2O_3. We had to have a complete analysis finished in three hours, and the amount of $MgCO_3$ should not differ more than 0·25 per cent. at utmost from the right one.

After many different trials and experiments made by my assistant, Mr. H. E. Harrison, we are satisfied that the following methods, if executed as described, will give a correct and satisfactory result. The difference between this method and that of Fresenius, which requires a time of 24 hours, will be found less than $\frac{1}{10}$ per cent.

Selection of Sample.—Take pieces about 1 cb. in. in size from different parts of the bed, pile, or shipment to be tested. Pulverise the whole sample to pass through a 900-mesh wire sieve. Mix well, spread out in a layer, and with the point of a spatula take about 10 grms. in small portions from different parts of the layer. Reduce this small sample to an impalpable powder, first in a porcelain and then in an agate mortar.

(A.) Weigh out two portions of 1 grm. each of the finely-powdered sample, transfer to small-sized basins, add to each about 100 c.c. distilled water. To one run in 25 c.c. normal HCl, cover with a watch glass, and allow to stand until all action ceases. Heat to boiling, cool, and neutralise with normal NaHO, using a drop of methyl-orange as indicator. c.c. HCl − c.c. NaHO = HCl required to saturate the carbonates of lime and magnesia.

(B.) To the other portion of 1 grm. add cautiously 5 c.c. concentrated HCl, keeping the basin covered with a watch glass to prevent loss by spurting. After all effervescence has ceased, evaporate to complete dryness over a low flame. When dry, cool and take up with a little hot water and a few drops of concentrated HCl. Heat to boiling and filter through an ashless filter, being careful to wash all insoluble materials into the filter. Wash with boiling water until free from all trace of chlorides.

(C.) Dry the filter and contents and ignite in a platinum crucible to bright redness, cool under desiccator, and weigh for SiO_2.

(D.) Neutralise the filtrate and washings from (B) with NH_4HO in slight excess. Heat to boiling, filter and wash the precipitate, if any, until free from chlorides. Dry at 110° C. and ignite; cool and weigh for Al_2O_3 and Fe_2O_3.

(E.) Heat the filtrate and washings from (D) to boiling and add a concentrated solution of ammonium-oxalate, "also heated to boiling." Allow to stand until settled clear, which is done, if rightly treated, in two or three minutes; decant the clear solution into a filter and dissolve the precipitate in HCl and re-precipitate with NH_4HO. Allow to settle, decant as before, and then wash the whole precipitate into the filter, and wash with hot water until free from chlorides and oxalates. Dry at 110° C. and ignite in a platinum crucible, at first cautiously, and then over a blast-lamp, until completely converted to CaO. Cool under a desiccator, weigh, and calculate per cent. $CaCO_3$ from weight of CaO.

To check results titrate the CaO with the normal HCl.

Divide percentage of $CaCO_3$ by 5 (= c.c. normal HCl required for $CaCO_3$) subtract the quotient from c.c. normal HCl required for (A) and multiply remainder with 4·2 for per cent. of $MgCO_3$.

For practical purposes we can recommend this method, and we have successfully used it for the last two years, and in many instances been able to finish a complete analysis in two hours.

Example:—1 grm. stone = 19·85 c.c. normal HCl.

The weight of the ignited CaO 0·538 grm. multiplied with 1·785 = 96·033 per cent. $CaCO_3$, the check with normal HCl = 19·2 c.c. and 19·2 × 5 = 96·0, which corresponds very nicely.

19·85 − 19·2 = 0·65 c.c. normal HCl for $MgCO_3$.

0·65 × 4·2 = 2·73 per cent. $MgCO_3$.

SiO_2 weighed = 1·06 per cent. and Al_2O_3 + Fe_2O_3 not present.

Thus the result was the following:—

		Per Cent.
$CaCO_3$	=	96·00
$MgCO_3$	=	2·73
SiO_2	=	1·06
$Al_2O_3 + Fe_2O_3$	=	0·00
Difference	....	0·21
		100·00

Direct determination of the $MgCO_3$ as $Mg_2P_2O_7$ gave 2·79 per cent.

The Chairman then announced that the Session was now closed, and that when it reopened in October he hoped to be able to invite the Section to hold their meeting in the new Havemeyer Laboratory of Columbia College.

Obituary.

PETER HART.

VICE-CHAIRMAN OF THE MANCHESTER SECTION OF THE SOCIETY OF CHEMICAL INDUSTRY.

Peter Hart was born at the village of Orford, near Warrington, on June 6th, 1834. His father was employed in Muspratt's Alkali Works, at Newton-le-Willows.

He left school when ten years old, and went as a clerk to a lawyer at Tarporley, Cheshire.

When Dr. Jas. Young, F.R.S., then manager of Muspratt's Works, took the management of Tennants' Works at Ardwick Bridge, Manchester, he induced Peter Hart's father to come and assist him. Peter Hart soon left Tarporley to join his father in Manchester, but returned to Warrington to enter the office of Mr. W. Beamont, a well-known lawyer and antiquarian of that town, and first Mayor of Warrington. He stayed here three years, acquiring a general education under Mr. Beamont's superintendence. In January 1849 he returned to Manchester and entered the service of Messrs. Tennant, commencing in the office. A few months later an event occurred which placed him in the position he desired—that of assistant to James Young. He now attended evening classes in chemistry at the Old Mechanics' Institution in Cooper Street, under Dr. Allen, and thus wisely supplemented the experience he was gaining under Young. It was whilst Young was working out his process for distilling paraffin oils from crude petroleum and from cannel shales, that Peter Hart thus acted as his assistant. About the year 1851 Young manufactured, at Ardwick Bridge, some tons of solid caustic soda from the ordinary caustic liquors, for use in his oil-refining process. Probably this is the earliest record of the manufacture of solid caustic in England. It is a date previous to that of Gossage's patent, *viz.*, May 18, 1853. Young finally left Ardwick Bridge in order to carry out his process of paraffin oil production on a large scale, and the elder Hart then assumed the managership, with his son as chemist of the works.

About this time Peter Hart improved the then existing method employed by Young for the estimation of tin in *Cassiterite.* Knowing the extreme activity of potassium cyanide as a reducing agent, he made use of this power in carrying out the improved method. He added a known weight of the *Cassiterite* to fused potassium cyanide, removed the cyanate and excess of cyanide, dissolved the reduced tin in hydrochloric acid, and titrated in the usual manner. Among numerous analytical methods published in the *Chemical Gazette* and devised by Hart, one is of special interest. It is the one he worked out for the "Estimation of Sesquioxide of Chromium in Chrome Ore"—a method now well known everywhere. It is based on the solubility of sesquioxide of chromium in fused borax, with subsequent oxidation with potassium nitrate and alkaline carbonate. This method is published by Fresenius in his classical work on Quantitative Analysis (the 4th edition, pages 387—388), with a note of reference as to original source, *viz.*, the *Chemical Gazette* of 1855.

At the request of Dr. Sheridan Muspratt in 1860, when he published his noted "Dictionary of Applied Chemistry," Peter Hart wrote the article on the "Manufacture of Sulphuric Acid." In 1866 his father met with an accident in the laboratory, which ended in his death, and the son then assumed full managerial control of the works.

Hart read, in the years following, quite a series of papers before the Literary and Philosophical Society of Manchester.

In 1885 Messrs. Tennants and Co. decided to remove their works from Ardwick Bridge—almost the centre of Manchester—to Clayton, and the duty of designing new plant and arranging the new factory devolved upon Hart, and was carried out under his superintendence.

Gifted with indomitable perseverance, shrewd judgment, and a quaint humour, Peter Hart was a typical *Manchester man* in the best sense of the word. He died on Sunday evening, May 30th, after a short illness, in his sixty-third year.—W. S.

Dr. C. REMIGIUS FRESENIUS.

GEH. HOFRATH UND PROFESSOR, DIREKTOR DES CHEMISCHEN LABORATORIUMS ZU WIESBADEN.

We note with regret the decease, on the 11th inst., of that great veteran of analytical chemistry, C. Remigius Fresenius, whose laboratory in Wiesbaden, whose text-books on qualitative and quantitative analysis, and whose organ, the *Zeitschrift für Analytische Chemie*, have long ago attained world-wide fame.

Fresenius was born on December 28, 1818, at Frankfort-on-the-Maine. His chemical training he obtained in Giessen, in the laboratory of Liebig, and at the close of his student career he remained with his great teacher as assistant, along with Witt and Hofmann, at that time also Liebig's assistants.

Fresenius now prepared himself for, and eventually assumed, the rank of a "Privatdocent" of the University of Giessen. This position he, however, shortly relinquished, in order to found a laboratory of his own in Wiesbaden. To the development and perfection of this institution Fresenius has dedicated a life-long active interest, and though in the main the courses of chemical study there have been on the applications of the science to industry, yet many young chemists have not considered their curricula complete without including Fresenius' analytical course at Wiesbaden.

In the earlier years of his life Fresenius had become engaged in industrial pursuits. He was connected with the *Verein fur Chemische Industrie* in Frankfort, and the *Verein Chemischer Fabriken* in Mannheim. In both these companies Fresenius was a member for many years of the respective directorates, and once or twice he held the position of chairman. In the words of his biographer, Dr. Otto N. Witt, in *Die Chemische Industrie*, June 15, 1897, reference may be made, in closing, to the esteem with which he is regarded both in the Vaterland and beyond it: "Sein Andenken wird bei Allen, die ihn gekannt haben, fortdauern als das eines bedeutenden Gelehrten und eines wahrhaft guten Menschen."—W. S.

Journal and Patent* Literature.

* Any of these specifications may be obtained by post by remitting 8*d.*—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

I.—PLANT, APPARATUS, AND MACHINERY.

Asbestos for Chemical Purposes, The Manufacture of Acid-Proof. F. Schrader. Chem. Zeit. 1897, **21**, 285.

Although the name asbestos is generally understood to apply to a fibrous substance largely composed of magnesium silicate, yet the name has been commercially applied to so many articles possessing varied chemical and physical properties, that it is not surprising that the above-mentioned properties are not clearly understood. Many articles termed asbestos contain no magnesia or only traces.

Chemical manufacturers have long desired and felt the need of a substance suitable for packing or millboard, and capable of withstanding alike high temperature and resisting strong acids and alkalis. The author has therefore investigated about 50 different forms of asbestos, which were on the market, with this object, and has arrived at the following conclusions :—1st. That to withstand alike high temperature and strong acids, or caustic alkali, asbestos should be of the character of hornblende, in which the base should be to silicic acid as 1 : 1, and of the formula $RSiO_3$, R representing magnesia.

Asbestos (serpentine asbestos) in which the proportion is 3 : 2, is capable of resisting high temperature, but not acids—even tartaric and acetic acids removing the bases and leaving almost pure silicic acid without apparently destroying the fibrous condition.

Boiling for four hours with dilute hydrochloric acid (5 per cent.) also effects the same end.

This latter form of asbestos (serpentine asbestos), on account of its fine and soft fibre, is particularly suitable for weaving and many technical applications other than chemical; whilst the former, owing to its acid-resisting character, is capable of employment in chemical works.

—O. H.

PATENT.

Material suitable for Belting, Carding, Cloths, Soles of Boots, and the like Uses; Impts. in the Manufacture of. F. W. Oliver, London. Eng. Pat. 10,104, May 12, 1896.

See under V., *page* 532.

II.—FUEL, GAS, AND LIGHT.

Mond Gas-Producer Plant and its Applications. H. A. Humphrey. Proc. Inst. Civil Eng. 1897, **129**, 1—29.

See this Journal, 1889, 505—510; also (for Patents), 1884, 236; 1886, 235; and 1894, 938.

The experiments described in this paper were made on a producer plant similar in its general arrangement to that previously described in this Journal, 1889, 505 *et seq.* Improvements had, however, been introduced in making the producer circular, and by means of a system of concentric tubes, economising to a greater extent the heat present in the different portions of the apparatus.

The present tests were made in 1895, under the ordinary working conditions as regards the producers, whilst the gas from 62, 82, and 94 tons of fuel per day respectively, was driven through one set of towers. The best results were obtained from the first test, that is, when the absorbing plant had the least work to do; but the figures of the second trial have been selected as more nearly corresponding with usual conditions. The fuel consisted of Nottingham slacks fed into the producers in charges of 8 to 10 cwt. at a time; the burnt ashes were removed from three of the six lute-holes alternately, the lutes untouched during one shift being drawn at the next.

The density of the sulphate liquor was kept at 35° Tw. (34 per cent. of sulphate of ammonia by weight), so that for every ton of solid sulphate subsequently made, it was necessary to evaporate $2\frac{1}{2}$ tons of water. By actual measurement of all the condensed steam from the sulphate plant, it was found that 5·6 tons of steam were condensed in performing this work, together with the necessary pumping.

The results of the trial showed that for every ton of fuel fed into the producers, about $2\frac{1}{2}$ tons of steam and 3 tons of air were blown through the grate, the mixture being at a temperature of 250° C. Of this steam, 1 ton is furnished by the system of regeneration and $1\frac{1}{2}$ tons is added as exhaust steam. More than $\frac{1}{2}$ ton of steam is decomposed in passing through the hot fuel, and nearly $4\frac{1}{2}$ tons of gas are formed from 1 ton of coal, equal to about 160,000 cb. ft. at ordinary atmospheric temperature. This gas has a calorific value of 81 per cent., calculated on the calorific value of the original 1 ton of fuel, and in a Babcock-Wilcox boiler, gives an evaporation of somewhat more than 7 tons of water. When used in a gas engine, it is sufficient to give 2,166 I.H.P. hours, being at the rate of 1·03 lb. of slack per I.H.P. hour, the thermal efficiency of the engine being 23·8 per cent. For each ton of slack gasified, the plant requires a supply of exhaust and direct steam equal to 0·242 ton of slack; this is allowed for in all calculations. The efficiency of the tubular generator is 81·9 per cent., against one of 72·07 when using circulating water. Diagrams are given showing the changes in pressure and temperature throughout the absorbing plant, the highest pressure being 310 mm. of water.

By burning cold Mond gas with the proper amount of cold air, the temperature of combustion, as actually observed, is 1,150° C.; whilst the theoretical temperature, calculated from the calorific value and specific heats, is 1,606° C. In this calculation the gas is taken as saturated at 15° C., and the air as half-saturated with aqueous vapour at the same temperature. When regeneration is employed, the difference between a poor and a rich gas begins to disappear, and with Mond gas, used in its wet condition, it is easy to reach temperatures beyond the range of any form of pyrometer available. With a Callander pyrometer, a temperature of 1,525° C. has been observed in a flue through which burnt gases from a Mond gas-fired steel furnace were passing.

In treating of the applications of producer gas, the author states that where gas has replaced slack-firing, 1·1 to 1·2 ton of coal in the producers is equivalent to 1 ton of good slack carefully burnt by the old method, and that very frequently the working results approach much more nearly to one of equality in weights of slack. Among the advantages claimed for the use of gas are, saving of labour and prolonged life of plant, certain cast-iron pans at the works of Messrs. Brunner, Mond, and Co. lasting with gas-firing four times as long as with coal-firing.

To show what might be accomplished in the supply of cheap power, the author takes as a hypothetical example a factory requiring a continuous power of 10,000 I.H.P. With a Mond producer and recovery plant to supply the necessary gas, and assuming the whole power to be utilised to drive dynamos yielding 7,000 E.H.P. at the terminals, allowing 20,000*l.* as the cost of the producing plant, and 120,000*l.* for buildings, gas engines, dynamos, &c. (exclusive of land) —the working expenses, day and night, for a year are estimated at 35,100*l.*, which includes interest on capital at 8 per cent. On this basis the total cost of one kilowatt-hour is 0·184*d.*, or of one E.H.P.-hour 0·137*d.* Should the mechanical power be used without transformation, this cost would be reduced to 0·0865*d.* per I.H.P.-hour.

The paper closes with three appendices, which contain the data from which the figures given in the text are abstracted, a few of which follow :—

Appendix I.—Table V.

Cost of working per Ton of Sulphate of Ammonia made.

	£	*s.*	*d.*
Total cost of all fuel (28·56 tons at 6*s.* 2*d.*)	8	16	1
Wages at producers (23 tons at 6·4*d.*)	0	12	3
Manufacturing wages, administration	0	1	9
„ „ labour	0	19	8
Repair wages and materials, including renewals	0	18	3
Gas for lighting purposes	0	3	11
Lubricants	0	1	8
Sulphuric acid, 0·95 ton at 145° Tw.	1	4	4
Total for above	12	17	11
Selling price of sulphate, naked at works	7	4	6
Final works cost of 23 tons of fuel gasified	5	13	5
Or the cost of 1,000 cb. ft. of gas (at 15° C.) is	0	0	0·351
The gas for 1 I.H.P.-hour will cost	0	0	0·02685

Appendix I.—Table VI.

Analysis and Calorific Value of Various Gases.

(Products cooled to 18° C.)

—		Mond Producer Gas from Bituminous Fuel.	Siemens' Producer Gas.	Dowson Producer Gas from Anthracite.	Lencauchez Producer Gas from Anthracite.	Solvay Coke-Oven Gas.	Coal Gas (Illuminating).	Pittsburgh Natural Gas.
Hydrogen	Vol. per Cent.	24·8	8·6	18·73	20·0	56·9	48·0	22·0
Marsh gas	„	2·3	2·4	0·31	..	22·6	39·5	67·0
C_nH_{2n} gases	„	Nil	Nil	0·31	4·0(?)	3·0	3·8	6·0
Carbonic oxide	„	13·2	24·4	25·07	21·0	8·7	7·5	0·6
Nitrogen	„	46·8	59·4	49·98	49·5	5·8	0·5	3·0
Carbonic acid	„	12·9	5·2	6·57	5·0	3·0	Nil	0·6
Total volume	„	100·0	100·0	100·00	100·0	100·0	100·0	100·0
Total combustible gases	„	40·3	35·4	44·42	45·0	91·2	98·8	95·6
Theoretical.								
Air required for combustion	„	112·4	101·4	113·2	154·0	410·0	581·0	806·0
Calorific value per cb. ft. in lb. ° C. units	„	85·9	74·7	88·9	115·3	284·0	381·0	495·8
Calorific value per litre in grm. ° C. units	„	1,374	1,195	1,432	1,845	4,544	6,096	7,932

Appendix I.—Table VIII.

Efficiencies of Gas Producers.

Description of Producer.	Calorific Power of Gas.	Figure of Merit.*	Cold Gas Efficiency.	Authority.
Mond circular producer	1,367	8,627	0·810	Author
Ordinary rectangular brick producer, original form	894	6,210	0·562	C. F. Jenkin
Same, after modification by Mr. C. F. Jenkin	1,311	7,750	0·712	„
Odelsjerna producer at Avesta, 1888, Prof. Akermann	1,549	8,110	0·754	„
Examples given by Mr. von Jüptner (1)	1,154	7,060	0·565	„
Examples given by Mr. von Jüptner (2)	1,044	7,060	0·468	„
Dowson producer, using Garnant anthracite	1,321	7,540	..	„
„ „ „ anthracite	1,272	6,590	0·689	„
Wilson producer, using slack	1,261	7,730	..	„
„ „ „ Durham coal	1,238	7,140	..	„
„ „ another example	1,314	7,420	0·702	„

* The "figure of merit" is the heat of combustion of the gas per kilo. of carbon contained in it. Kilo. ° C. units.

From various tables in Appendix III. the following values are selected.

Using Mond gas to generate the current for the electric light at the caustic soda plant of the Winnington works, the total cost (including renewal of carbons and lamps, and without deduction for the ammonia) was 9*l*. 3*s*. 9*d*. for light of 17,040 candle power over 320 hours. The cost of coal-gas to give the same light, and allowing $3\frac{1}{3}$ cb. ft. (at 2*s*. per 1,000) per 16 candle-power hour, would be 119*l*.

Average power at switchboard	100·0 volts.
Average current at switchboard	184·0 ampères.
Number of Board of Trade units at switchboard	5,888
Cost per unit (1,000 Watt-hours)	0·394*d*.
Cost of fuel and labour for Mond gas (without recovery plant) per 1,000 cb. ft.	0·507*d*.
Corresponding cost of Dowson gas per 1,000 cb. ft.	1·8*d*.

—J. T. C.

Coal-Dust Fuel. C. Schneider. Mitt. aus der Praxis des Dampfk. und Dampfm.-Betr. 1896, 255; and Proc. Inst. Civil Eng. 1897, **127**, [1], 38—39.

Account of experiments on the Schwartzkoff and Friedeberg systems of burning coal-dust fuel. The experiments were made with a Lancashire boiler designed for a pressure of 7 atmospheres, and of the following dimensions:—Length, 7,500 mm.; diameter of shell, 2,000 mm.; diameter of flue, 750 mm. Each flue had six Galloway tubes; the heating surface was 68·22 sq. m., of which 13·26 sq. m. was protected by fireclay from the direct action of the flame.

The fuel used was Westphalian coal, containing 83 per cent. of carbon, 5 per cent. of hydrogen, and having a heating value of 7,837 thermal units; and Oberschleswig coal, containing 77 per cent. of carbon, 5 per cent. of hydrogen, and having a heating value of 7,168 thermal units. The coal was ground so as to pass through a sieve of 900 meshes per sq. cm.

With the Schwartzkoff apparatus, 1 kilo. of the Oberschleswig coal-dust evaporated 8·9 kilos. of water (calculated from water at 0° C. to steam at 100° C.), the heat utilised being 79 per cent. of the total. When the combustion was forced, the evaporation was 8·3 kilos. of water per kilo. of coal, and the heat utilised 74 per cent. With the Westphalian coal-dust, 8·9 kilos. of water were evaporated per kilo. of coal, the heat utilisation being 72 per cent. The Friedeberg apparatus did not act quite so well. The air used varied from 1·14 to 1·41 times the theoretical volume required. The temperature of the gases and the percentage of carbonic acid varied very little, and the combustion was nearly smokeless.—A. S.

Coal-Gas, The Enrichment of. T. Holgate. Proc. Inst. Civil Eng. **127**, Part I., 1897, 1—37.

The illuminating power of gas from bituminous coal may be raised to 20 candles by four methods, *viz.*:—(1) by admixture with gas from cannel, boghead, and other shales; (2) by addition of the vapour of light hydrocarbons; (3) by addition of oil-gas; and (4) by admixture with carburetted water-gas. Carbon dioxide and nitrogen are useless constituents of coal-gas, and their exclusion improves its illuminating power; the more so the less efficient the burner. 16-candle coal-gas is diminished by 0·5 or 0·25 candle in an Argand burner, and by 0·8 or 0·43 candle in a flat-flame burner, for 1 per cent. of carbon dioxide or nitrogen respectively, mixed with it. Allowing for diminution of volume, the cost of enrichment of coal-gas by removal of carbon dioxide by lime purification is 0·54*d*. and 0·292*d*. per candle per 1,000 cb. ft. for the Argand and flat-flame

burners respectively. The nitrogen liberated in the distillation of the coal cannot be excluded from the gas, but a quantity not exceeding 2 per cent. is left therein if atmospheric nitrogen be excluded by careful working. Aqueous vapour is injurious to the illuminating power, but in practice it cannot be excluded from the gas.

The cost of enrichment by the admixture of two gases is calculated by the following rule:—Let a, b, and c denote illuminating power of enriching, unenriched, and enriched gas; and p, q, and r the cost per 1,000 cb. ft. of enriching, unenriched, and enriched gas; then $\frac{r-q}{c-b} = \frac{p-q}{a-b}$ = cost of enrichment per 1,000 cb. ft. per candle-power.

A ton of coal yielding 11,900 cb. ft. of 15-candle gas is assumed to cost 8*s.* 9*d.*, and a ton of cannel yielding 11,466 cb. ft. of 28·54-candle gas, 15*s.*, delivered at the works. The assumed cost per gallon of carburine, having a specific gravity of 0·680, and distilling completely between 90° and 230° F., is $7\frac{1}{2}$*d.*; that of petroleum spirit, sp. gr. 0·700, distilling between 95° and 310° F., is $5\frac{1}{2}$*d.*; and that of 90 per cent. benzol, sp. gr. 0·881, is 1*s.* The Maxim-Clark carburettor is that usually employed for enriching gas by the vapour of these light hydrocarbons. A sample of spirit prepared from the hydrocarbon residue of the compressed oil-gas works appeared to be superior to carburine and benzol as a carburetting agent, but the increase in illuminating power was not exactly determined. Oil-gas for enriching purposes is prepared by the Young and Bell fractional gasification method or by the Caldwell and Tatham oxy-oil method. The first method gives 22,000 cb. ft. of gas per ton of Scotch shale oil, sp. gr. 0·875; similar but rather heavier oil (sp. gr. 0·878) yields by the second process 23,205 cb. ft. of gas, to which 4,080 cb. ft. of oxygen at 4*s.* 6*d.* per 1,000 cb. ft. are added in the receiving main. The oil in both cases is assumed to cost 3*l.* 7*s.* 6*d.* per ton delivered, and the illuminating power of the gas is estimated on the basis of its enrichment value (see table). The Lowe type of carburetted water-gas plant makes 1,000 cb. ft. of 24-candle gas from 3·45 gals. of Russian kerosene and 45 lb. of gas-coke, and the same quantity of 30-candle gas from 5 gals. of kerosene and 40 lb. of coke. Gas-coke is taken at 6*s.* 8*d.* per ton, and kerosene at $2\frac{1}{4}$*d.* per gallon, delivered. The following table is compiled on the basis of the foregoing prices for raw materials, and of current rates of wages, and covers all capital and maintenance charges, whilst allowance is made for the receipts from by-products wherever such are obtained. The holder price of 15-candle coal-gas is assumed to be 9*d.* per 1,000 cb. ft.

TABLE SHOWING COST OF ENRICHMENT PER CANDLE.

Enriching Agent.	Cost per 1,000 cb. ft.	Illuminating Power by Direct Testing.		Illuminating Power by Enrichment Value.		Cost of Enrichment per 1,000 cb. ft. per Candle.	
		Candles.	Cost per Candle.	Candles.	Cost per Candle.	Direct Testing.	Enrichment Value.
	d.		*d.*		*d.*	*d.*	*d.*
Cannel gas	16·75	28·54	0·586	..	..	0·572	..
Carburine vapour	..	..	..	..	..	..	1·218
0·700 spirit	..	..	..	..	..	..	1·072
Benzol	..	..	..	..	..	..	0·600
Young's oil-gas	40·00	..	..	90	0·444	..	0·413
Oxy-oil-gas	48·00	..	..	126	0·381	..	0·351
Carburetted water-gas	13·92	24·0	0·580	*	..	0·546	..
" "	17·60	35·0	0·503	*	..	0·430	..

* It has been observed that the enriching value of carburetted water-gas is from 6 to 20 per cent. higher than the direct illuminating power. Similar behaviour is probably true of cannel gas.

—J. A. B.

Acetylene, A Bunsen Burner for. A. E. Munby. Proc. Chem. Soc. 1897, [179], 103.

WE employ a Bunsen burner of special dimensions, the tube being 5 mm. in internal diameter. A slightly wider tube may be used, provided the mouth be curved inwards, so that the actual exit does not exceed the diameter mentioned; if larger, the flame tends to strike down. The gas jet is very small, being only capable of delivering about 1 cb. ft. of acetylene per hour under 6 ins. water pressure, such a rate of consumption giving an ordinary working flame. The air holes and collar are arranged as in an ordinary Bunsen, the exact size of the former not being of much importance, provided they be large enough to admit the air required. The burner is protected with a cap, when not in use, as its efficiency depends upon the jet maintaining its dimensions. A generator capable of giving gas under 7 ins. water pressure with the full number of burners in use, is required. Under this pressure a large and perfectly blue flame is obtained, which may be turned down to what may be termed a quarter Bunsen flame, equivalent to burning the gas under 3 to 4 ins. water pressure. This is the smallest pressure with which the burner will give a non-luminous flame. When turned lower, the zone of partial combustion appears, since the draught is then insufficient.

The heating effect of the flame is of course very great, enabling one to dispense with the blow-pipe for some operations, such as small fusions. From a few experiments on heating equal quantities of water under like conditions with coal-gas and acetylene, it would seem that in practice, for equal volumes burnt, the latter has nearly twice the heating power of the former.

Calcium Carbide, New Economical Preparation of. H. Marx. Monit. Scient. 1897, **11**, [4], 276—281.

THIS paper consists of a critical discussion of Heibling's work on the electrolytic preparation of iron alloys (this Journal, 1896, 726). In thermo-electric metallurgy, it is necessary that the whole substance under treatment should become fluid, and also that the slag produced should be a conductor. When lime is employed as a flux, a slag of fusible calcium carbide is formed, which, besides being a valuable product, is an excellent conductor. The results of some experiments carried out at Puteaux, show in a marked manner the value of lime as a flux, the horse-power diminishing according to the amount of lime used, whilst the yield of acetylene per kilo. of calcium carbide, increases.

—J. L. B.

Acetylene, A New Method of Storing. G. Claude and A. Hess. Comptes Rend. 1897, **124**, 626—628.

ACETONE at 15° and under ordinary pressure, dissolves 25 volumes of acetylene, and under 12 atmospheres, 300 volumes. Thus the solubility increases nearly in proportion to the pressure. The solubility in acetone is diminished by half on passing from 15° to 50°, and therefore the pressure in a reservoir doubles for a rise of about 30°. With liquid acetylene a pressure of 24 atmospheres increases to 70 for a rise of 18°. Dissolved acetylene may therefore be stored safely in weaker and lighter vessels than liquid acetylene, and since its coefficient of dilatation is very much lower, the vessels may be more nearly filled. A platinum wire, maintained at bright redness by electric current, was held for an indefinite period with impunity in a solution under 3 atmospheres pressure of acetylene in acetone.—J. A. B.

Acetylene Gas for the Generation of Power, The Applicability of. H. Schrey. Ann. für Gewerbe und Bauw. 1896, Nov. 1, 164; Proc. Inst. Civil Eng. 1897, **127**, [1], 51—52.

This is the record of some experiments conducted by Ravel in America. It was found that 1·35 parts of acetylene mixed with 1 part of air was explosive, and the explosive force of the mixture rose rapidly as the dilution with air increased, the maximum being attained when 12 volumes of air were mixed with 1 volume of acetylene. Beyond this limit, the explosive force decreases, until at 20 of air to 1 of acetylene, it is entirely extinct. The special characteristics of acetylene are, great rapidity of the transmission of flame, low ignition temperature (about 480 C.), high combustion temperature, and an extraordinary energy evolved in the explosion. 6·35 cb. ft. of acetylene gas sufficed to generate 1 H.P. per hour. In a small 2-H.P. motor, acetylene gas gave 2·1 times the work of the same volume of illuminating gas. It was found that the cooling of the cylinder by lubrication has a much more important effect upon the working than is the case with coal-gas.
—A. S.

Incandescent Lighting, A Report on the Use of Spirit for. Dr. Hayduck. Zeits. für Spiritusind. 1897, Supplement II., 22—23.

The illuminating power of incandescent spirit-lamps depends primarily on the mantle and secondarily on the structure of the lamp. The mantle must correspond in shape with the flame. The lamp must be easily managed, readily lighted and extinguished, burn with a steady flame free from smoke and smell, and must not be dangerous. Of several lamps from different firms, the "Phœbus" complied, it is said, with these conditions, and gave the best result. For an hourly consumption of 85 c.c. of 86 per cent. (volume) spirit, it gave an illuminating power of 31 candles. At the current price of spirit it would yield a light of 10 Hefner units strength, at a cost of 0·65 pfennig (in Germany), whereas the same light costs 0·84 pfennig per hour by an ordinary petroleum lamp. Several lamps are described in detail.—J. A. B.

Ethylene, Action of Nickel on. P. Sabatier and J. B. Senderens. Comptes Rend. 1897, **124**, 616—618.

A layer of nickel reduced by hydrogen, if heated to 300° and upwards, completely decomposes pure dry ethylene passed over it. The products are only carbon, hydrogen, and methane, in varying proportions. The amount of hydrogen increases with the temperature. Freshly reduced iron, copper, and cobalt, and spongy platinum and palladium, were inactive, and nickel filings less active than the reduced metal. The nickel and ethylene appear to form an unstable compound, which decomposes into carbon, methane, and nickel; and hydrogen is formed by a secondary reaction. The explanation offered by Moissan and Moureu (Comptes Rend. **122**, 1240; this Journal, 1896, 530) of the decomposition of acetylene by reduced metals in the cold—*viz.*, that it was due to the heat of absorption of the gas by the porous metal provoking decomposition of the endothermic gas—cannot apply in the present case.
—J. A. B.

Carbonic Oxide, A Reaction of. A. Mermet. Comptes Rend. 1897, **124**, 621.

See under XXIII., *page* 561.

Acetylene, Action upon Cupric Salts. H. G. Söderbaum. Ber. **30**, [7], 814.

See under XXIV., *page* 569.

Hexane and Trimethylene, Decomposition of, by Heat. F. Haber and H. Oechelhäuser. J. für Gasbeleuchtung, 1896, **39**, 799, 813, and 830.

See under III., *page* 528.

PATENTS.

Vapour or Gas from Liquid Hydrocarbons, An Improved Apparatus for Producing. A. I. van Vriesland, Amsterdam. Eng. Pat. 5991, March 6, 1897.

From the bottom of an oil reservoir a pipe rises a short distance, serving as an air-supply shaft. From the reservoir cover rises an exit pipe having a narrowed nozzle. A tubular sheath, "formed of the most absorbent material possible," is drawn over the air shaft, and projects concentrically above it to a certain extent, the projecting part being supported by stiffening wires. The reservoir is filled with the hydrocarbon, or with a substance saturated therewith, so that the sheath draws up the hydrocarbon and evaporates it within the pipe that carries the nozzle. Air is drawn up the supply shaft, becomes saturated with the hydrocarbon vapour, and passes out at the nozzle as a combustible mixture.—H. B.

Combustible Gases, Improved Process and Plant for Generating. B. H. Thwaite, Westminster. Eng. Pat. 8384, April 21, 1896.

Improvements in processes and plant described in previous patents by the same inventor, *viz.*, Eng. Pat. 13,385, 1894 (this Journal, 1895, 794); and Eng. Pat. 18,445, 1894 (this Journal, 1895, 856). When producing gas in a single-vessel generator from hydrocarbon fuel, the two tubes for gas and air, each having openings into the generator at the top and the bottom, have each a valve so connected together by gearing that both are reversed by turning one hand wheel. Thus, the fuel may be preliminarily and occasionally intermittently blown up to a state of incandescence before the air is introduced. When using a duplex gas generator working in a continuous cycle, the upper part thereof is encased in a water jacket for the purpose of generating steam.—R. S.

Carburetted Water-Gas, Impts. in Apparatus for the Manufacture of. The Economical Gas Apparatus Construction Co., Ltd., London, and L. L. Merrifield, Franklin, U.S.A. Eng. Pat. 11,530, May 27, 1896.

One superheater and fixing chamber is connected with two generators in such a way that the gases and vapours from the top of either generator can be led to the top of the other generator, and, after passing down through the fuel therein, are conducted to the superheater, where they are carburetted in the usual way. The order of the action of the generators can be reversed by means of suitable valves. Steam may also be passed downwards through both generators simultaneously, or upwards, in each case passing directly to the carburetting chamber.—H. B.

Water-Gas, An Improved Manufacture of. J. E. Goldschmid, Frankfurt a/Main, and C. Dellwik, Stockholm. Eng. Pat. 11,632, May 28, 1896.

By means of a pipe passing beneath the grate, and a series of twyers at various heights in the generator, air is introduced into the coke or the like at different points. The coke is rapidly raised to incandescence, producing chiefly carbon dioxide. The steam is then passed alternately from above and from below, preferably two admissions from the bottom to one from the top. A locking arrangement is applied to the valves in such a way that the outlet valve for the combustion gases cannot be closed as long as the water-gas valve remains closed, and *vice versa*.—H. B.

Oil or Vapour Burners or Atomisers for Heating Purposes, Impts. in. H. H. Lake, London. From T. R. Browne, Altoona, U.S.A. Eng. Pat. 12,338, June 5, 1896.

The burner is adapted for use outside a furnace, to which it is connected by an adjustable arm. It consists of an oil-spraying nozzle, having a central air channel and an annular outer air channel, and oil passages situated between these channels. Owing to the sides of the annular air passage converging towards the mouth-piece of the nozzle, the latter throws a jet of air and oil, which first converges towards and then diverges from a point in front of the nozzle. Situated about 4 or 5 ins. in front of the nozzle is

the receiving nozzle of the furnace, having its inner walls proportioned and flared to conform with the size and shape of the mixed oil and air jet, and having its outer part abruptly flared outwards. Instead of compressed air, steam may be used in atomising the oil.—H. B.

Gas, The Manufacture or Preparation of Improved Odorising Substances [Isocyanides] for Artificially Imparting Odour to. H. Strache, Vienna. Eng. Pat. 14,941, July 6, 1896.

To impart a strongly characteristic odour to odourless poisonous or explosive gases, they are led over material (shavings, pumice, &c.) saturated with one or more "carbylamines" (isocyanides), or over the isocyanides alone. In producing the latter, an amine (say 30 grms. of aniline) is dissolved in alcohol (600 grms.), then decomposed with alkali (100 grms. of sodium hydrate to 70 grms. of water), and chloroform (45 grms.) is cautiously added, with stirring. The mixture is frequently shaken during one day, and then allowed to settle, when the dilute product, containing about 3 per cent. of isocyanide, may be employed directly for odorising gases. To obtain a concentrated product, pour the calculated quantity of aniline over fine granular soda lime, heat to about 100° C., and add cautiously the calculated amount of chloroform, drop by drop, with continual stirring. After cooling, a part of the carbamine can be poured off from the solid chlorides of sodium and calcium; what remains can be obtained in strong solution by leaching with the smallest practicable quantity of alcohol.—H. B.

Hydrogen Gas, The Utilisation of, for the Obtainment of Light, Heat, and Power. L. V. Pratis and P. Marengo, Turin. Eng. Pat. 16,277A, July 22, 1896.

The burner for the hydrogen is of much the usual type, and the mantle is suspended over it in the usual way. When using hydrogen for driving gas engines, the inlet port for the air should have a sectional area of about 18 times that for the hydrogen. The three claims are: for the obtaining of light by burning hydrogen in contact with hoods of platinum gauze or of earths, &c.; the utilisation of hydrogen for obtaining heat or power; and the utilisation of hydrogen by compressing it and applying it to the illumination of trains, &c.—H. B.

Illumination, partly by Flame and Partly by Incandescence; Impts. in the Method of and Apparatus for Effecting. J. B. de Léry, New York. Eng. Pat. 7920, March 27, 1897.

The claim is for the "combination, with a gas burner adapted to throw a series of flames into the form of a crown, of a circular or disc-like web or netting of incandescing material arranged above the crown of flame in a horizontal plane." "This is placed at such a level as to be struck by the tips of the flames." The combined illumination by flame and by incandescence is also claimed.—H. B.

[Mantles.] Incandescence Bodies for Illuminating Purposes, Impts. in the Manufacture of. W. Nicholls, London. Eng. Pat. 9244, May 1, 1896.

In manufacturing mantles in the usual way, the fluid is to be prepared from the following:—Zirconium nitrate, 250 parts; calcium chlorate, 100 parts; ammonium nitrate, 160 parts; uranium nitrate, 5 parts.—H. B.

Mantles or Incandescing Bodies for Incandescent Gas Lamps, Impts. in the Manufacture of. J. F. Duke, West Dulwich. Eng. Pat. 13,659, June 20, 1896.

To render the mantles more durable, to increase their lighting power, and to obviate the greenish tinge usually acquired by the light after some time, the mantles, after receiving their usual coating of collodion, are dipped in a solution of barium salt (preferably cold saturated $BaCl_2$ solution). They are then dried and highly heated over a Bunsen burner until the barium salt is converted into oxide, a slight fusion taking place, which causes the barium oxide to be permanently retained.—H. B.

[Mantles.] Incandescence Bodies for Incandescent Gas Lighting, Impts. relating to. C. Killing, Düsseldorf. Eng. Pat. 14,365, June 29, 1896.

The provisional specification describes the use of a large number of substances added in minute quantity to pure thoria mantles, but the complete specification describes only the manufacture of mantles in the usual way from fluid composed of 4 grms. of thorium nitrate, 10 c.c. of water, and 8 drops of iridium chloride solution containing 0·0023 grms. Ir per c.c. The claim is for "An incandescence mantle containing, in addition to thoria, a small quantity of iridium, substantially as described."—H. B.

Peat for Fuel, and to Apparatus therefor; Impts. relating to the Preparation of. C. D. Jenkins, Boston, U.S.A. Eng. Pat. 8402, April 21, 1896.

The peat is first passed through a horizontal drying chamber provided with a screw conveyor, with a heating jacket, and with pipes connected with an exhaust for drawing off air and other gases from the peat. Dry or superheated steam or heated compressed air is forced through this chamber. After leaving the dryer, the peat is passed into a special press or pug mill, two forms of which are shown, in which it is forced by a taper screw through a gradually decreasing passage while subjected to a partial vacuum. It is finally cut up into pieces of a convenient size and shape. —R. S.

Artificial Fuels [Briquettes], Improved. R. Bradshaw, Ash Grove, Liverpool. Eng. Pat. 9599, May 6, 1896.

Blocks or briquettes are formed either solid (that is, of one mixture throughout), or compound (that is, having a core or a number of cores or layers of a more readily inflammable material than the bulk of the block). The solid briquettes are formed of a base of bituminous or anthracite coal-dust, or slack, or coke-dust, or a combination of the same, with ingredients for binding, and are shaped with flat surfaces or with corrugations. The main bulk of the compound briquettes is formed of coal or coke dust or anthracite, farina, alga, and potato starch; while the cores or more inflammable layers are formed of petroleum, astatki, resin, gas-tar, saltpetre, or their equivalents.—R. S.

Artificial Fuel, An Improved, and Process for Producing the same. J. Kohlndorfer, Landshut, Bavaria. Eng. Pat. 5669, March 3, 1897.

Petroleum is converted into a fuel of consistent form, by heating it with soda lye (about 10 per cent.) and a fatty substance (about 10 per cent.), or resin; either coal-dust, sawdust, &c. may also be added.—R. S.

Illuminating Gas, Impts. in Treating Peat, Coal, and other Carbonaceous Substances for the Obtainment of. J. Meikle, Maryhill, N.B. Eng. Pat. 11,951, June 2, 1896.

A spray of hydrocarbon or carbonaceous oil is injected by means of superheated steam into gas made from peat, coal, and other carbonaceous substances, either at the time when being formed or immediately afterwards.—R. S.

Coke Ovens, Impts. in Horizontal. O. Imray, Southampton Buildings, London. From F. Brunck, Dortmund, Prussia. Eng. Pat. 12,535, June 8, 1896.

The air supply for such ovens is highly heated before passing into the oven, by its passage through a grating-like partition wall, on each side of which is a flue through which the hot gases from the oven pass.—R. S.

Acetylene Gas, Method and Means or Apparatus for Facilitating the Combustion of. C. Coppeaux, Brussels. Eng. Pat. 9630, May 6, 1896.

The object of this invention is to overcome difficulties in the use of acetylene for lighting purposes arising from the relatively large volume of air required for its perfect combustion. Air is supplied under pressure to the burners from a conduit.—J. A. B.

Gas [Acetylene] Generators, Impt. in. W. Jordan, Chancery Lane. From The Morley Acetylene Gas Co., Wheeling, and Wall St., New York, U.S.A. Eng. Pat. 10,126, May 12, 1896.

In one form the generator consists of an enclosed vertical, cylindrical vessel, placed within another cylindrical vessel, open at the top and containing water, a central tube open at both ends passing upwards through the bottom of the generator, so that water can pass from the outer reservoir to a number of branch outlets arranged at different levels in the central tube, and flow upon the carbide. Another tube passes through the said pipe and serves as a delivery pipe, being open at the top and leading to a gas-receiver, either a collapsible bag or a small gasometer. In either case, as the gas-receiver rises, the pressure on the gas is increased by means of weights, tending to drive the water away from the carbide. Suitable valves are provided for regulating the passage of the gas.—R. S.

Generating Gas [Acetylene, &c.], Improved Apparatus for Automatically. G. Webb, jun., Shepherd's Bush, and J. W. Kelly, Finsbury Pavement. Eng. Pat. 10,725, May 18, 1896.

Relates to automatically generating such gases as acetylene, by the action of water upon a solid compound.—R. S.

Acetylene Gas, Improved Method of and Apparatus for Generating. M. C. A. Fourchotte, Paris. Eng. Pat. 12,017, June 2, 1896.

Attached to the bell of a gasholder is a pipe or chamber, the lower end of which is sealed in the water in the tank, and slides over the mouth of a smaller tube which rises through the bottom of the tank nearly to the level of the water. This tube communicates externally with a generator containing calcium carbide. There is confined within the pipe attached to the bell a certain volume of air or gas, which becomes compressed by the sinking of the bell and forces water from the tank through the tube to the generator. The gas generated in the latter passes through a hydraulic seal into the gasholder, the bell of which it lifts, and thus relieves the pressure of the air in the chamber and cuts off the flow of water to the generator. When the bell again falls, a further supply of water is forced into the generator. —J. A. B.

Acetylene Gas, An Improved Apparatus for Generating. F. H. Haviland, A. Holloway, J. B. Collier, and W. H. Murch, Bournemouth. Eng. Pat. 15,122, July 8, 1896.

A vessel is divided horizontally into two compartments, of which the upper serves as a cistern and the lower as a gasholder. There is a space between the compartments. The water preferably flows from the cistern through a syphon-shaped pipe to a "pressure cylinder," and thence to the base of the gasholder. The pressure cylinder remains filled so long as there is water in the cistern. One or more generators are supplied with water from the gasholder. The water enters the generator at its base, and either passes up a pipe from the mouth of which it drips on to the calcium carbide, or is carried up by a wick, filling the annular space between the wall of the generator and a perforated carbide receptacle within it. The gas passes from the generator, through a cooling coil within the cistern, to a purifier and drying chamber, and thence to the point of consumption or to the gasholder.—J. A. B.

Acetylene Gas, Rendering it inactive when brought into Contact with Metals with which it forms Explosive Compounds, A Process for. G. Isaacs, Charlottenburg. Eng. Pat. 15,139, July 8, 1896.

To prevent acetylene, which, it is found, always contains ammonia, forming explosive compounds with copper, silver, mercury, and other metals, any sulphuretted hydrogen present is first eliminated by means of powdered iron ore, after which the gas is treated with substances which will absorb the water and ammonia present.—R. S.

Acetylene Gas, Impts. in Means or Apparatus for Use in the Production, Storing, and Application of. T. Holliday, Huddersfield. Eng. Pat. 885, Jan. 12, 1897.

The apparatus consists of one or more generators placed on a level with or below a displacement gasholder. The generator contains a reticulate cage to retain the lime residue which falls from the calcium carbide in a perforated cage above it. (Eng. Pat. 5813, 1896; this Journal, 1897, 319.)—J. A. B.

Acetylene Gas, Impts. in or relating to the Production of [from Carbides]. V. Daix, Paris. Eng. Pat. 911, Jan. 13, 1897.

The carbide is placed in vessels arranged within the generator so that the water overflows from the upper vessel into the one next below it, and only a small portion of fresh carbide is at any time in contact with water. The carbide receptacle is surrounded by a water chamber in which the gas evolved is washed and cooled. The gas enters a bell gasholder, to the bell of which is attached a siphon device which supplies water to the generator from the gasholder tank until the bell has risen above a certain level.—J. A. B.

Acetylene and certain other Gases, An Improved Generator for. E. H. Fernand des Essards, Paris. Eng. Pat. 1153, Jan. 15, 1897.

The generator consists of two superposed hermetically-closed vessels, communicating by a device which permits water to flow from the upper to the lower vessel under constant pressure.—J. A. B.

Acetylene Gas Lamps and Generators. W. H. Dennis, Minneapolis, U.S.A. Eng. Pat. 2976, Feb. 4, 1897.

The object is to provide a generator which may be embodied in a table or wall lamp. Two tanks are attached to a suitable upright, which may serve to convey the gas to the burner or burners. One tank contains water, which may pass through a duct near its base to the foot of the other tank, wherein is a carbide holder. The water may pass up the annular space or "water-leg" between the walls of the tank and carbide holder and overflow into a distributing cone on the top of the latter. The gas generated, passes upwards through a basket containing purifying material, and then through a pipe to a condensing coil in the water-tank. In the event of too rapid production, the pressure of the gas depresses the water in the water-leg, and prevents it flowing into the carbide holder.—J. A. B.

Acetylene and other Gases, An Improved Combined Automatic Generator, Condenser, and Holder for. H. R. Bean, Plaistow, and H. Ringwood, Poplar. Eng. Pat. 5756, March 1, 1897.

Within a double-walled tank moves a bell forming a gasholder, to the crown of which is attached a chamber containing a basket filled with carbide; the lower portion of the chamber is contracted and surrounded by an air vessel which regulates the pressure of gas. The top of the tank bulges to form an annular space for the reception of water displaced by the pressure of the gas generated.—J. A. B.

Acetylene Gas, Impts. in the Manufacture of [Use of Steam], and in Apparatus therefor. G. Pereire, Paris, E. Sorel, Paris, and B. Cruvellier, Marseilles. Eng. Pat. 6997, Mar. 17, 1897. (Under International Convention.)

Steam is used in place of liquid water for the decomposition of calcium carbide, and the action may be instantaneously stopped by closing a steam-cock.—J. A. B.

Acetylene Gas, Impts. in Apparatus for Generating. E. G. Brewer, London and Leeds. From The Gesellschaft für Acetylen-Gaslicht Basel, Bale, Switzerland. Eng. Pat. 7744, March 25, 1897.

A generator with removable bottom is immersed in a water-tank. A basket containing calcium carbide is supported on the bottom of the generator. A conical cap projects over the top of the basket, and prevents water reaching the carbide from the top. To the top of the

generator is attached a cross tube, one arm of which leads to a gasholder, and another to a safety-valve. The vertical arm is attached to a tube supporting a water vessel and furnished with a five-way cock, by means of which air or gas from the generator may pass to the water vessel while water passes in the opposite direction, or by means of which gas may pass out through a side arm. The top of the water vessel is provided with a tube having a cock with double passage ways, one of which communicates with a funnel and the other with the open air. Thus the air can escape from the vessel while it is filled with water through the funnel. By manipulation of the cocks, water passes slowly from the water vessel and reaches the carbide from beneath, while gas replaces the water taken from the vessel, and allows its flow to continue.—J. A. B.

Acetylene Gas, Impts. in Apparatus for Automatically Generating. H. H. Lake, London. From F. Ferracciù, Savona, Italy. Eng. Pat. 7782, March 25, 1897.

The apparatus consists of a gasholder supported on a tripod, and a generator which is clamped to the bottom of the gasholder and connected with the tripod. The generator contains four small movable baskets each with a central tube suitably perforated so that water descending from a funnel on the top basket first moistens the carbide in the lowest basket. The space within the gasholder bell is nearly filled by an impermeable drum attached to its crown and serving as a float. The acetylene evolved, causes the float to rise and when its bottom has reached the level of the water in an external basin, the supply of water to the generator is automatically cut off.—J. A. B.

Electrical Furnaces [Carbides, &c. under Vacuum or Pressure], Impts. in or relating to. A. J. Boult, London. From O. Patin, Paris. Eng. Pat. 19,290, Sept. 1, 1896.

Electrical furnaces are described "adapted for the manufacture of calcium carbide and treating in vacuum [or under pressure] bodies which are liable to become oxidised by the action of air." "The important feature of the invention is the moving of the material as it is being treated by the electrodes." The four claims are for mechanical parts, and their combination and arrangement constituting a complete electrical furnace.—J. C. R.

Electric Furnaces [Arc moved by Magnets], Impts. in and connected with. F. J. Patten, New York, U.S.A. Eng. Pat. 4138, Feb. 16, 1897.

This invention, illustrated by drawings, "consists in a system of electric furnace construction in which an electric arc, being established between electrodes, is made to move by magnetic (electro-magnets shown) influence from one point to another so as to arrive at and affect material that it would not reach if it remained practically steady at one point or in a fixed position." Claim 1 is :—"The method of operating an electric furnace which consists in passing the material to be operated upon between electrodes, subjecting the arc to the influence of a magnetic field, the lines of force of which are substantially transverse to the direction of the arc, and reversing or alternating either the current in the arc or the magnetic field, whereby the arc is reciprocated transversely to the path of the material."
—J. C. R.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Hexane and Trimethylene, Decomposition of, by Heat. F. Haber and H. Oechelhäuser. J. für Gasbeleuchtung, 1896, **39**, 799—805, 813—818, and 830—834.

This work is a continuation of that of Haber and Samoylowicz, to which reference is given in a New Book notice, this Journal, 1896, 302, and Journ. f. Gasbel. 1896, 377 *et seq.*

The heating was effected in an electric resistance furnace. The resistance consisted, for temperatures below 1,700° C., of a thin tube of platinum iridium 400 mm. long, 55 mm. wide, and 0·05 mm. thick, with copper tabs soldered to the ends for connection with the electric current. The middle 240 mm. of the tube was closely wrapped round with asbestos cloth. In this way the temperature of the covered part could be maintained at anything up to about 1,700° C. without the uncovered ends becoming unduly hot. The gas to be heated was passed through another thin-walled tube, just fitting comfortably inside the platinum tube. For moderate temperatures, this inner tube was made of hard glass, for high temperatures of special porcelain (Royal Berlin, No. 7). This porcelain and the metal both began to soften about 1,700° C., and above this temperature a gas-coke tube was substituted for the platinum, the inner tube being dispensed with. In the latter case the tube was, of course, somewhat porous, but by keeping a slight excess of pressure at the inlet, enough of the decomposition products could be driven through for collection at the outlet. All precautions were taken for maintaining temperatures constant and for obtaining true measurements of temperature.

In the analysis, Bunte's burette was generally used. Special tests with pure ethylene showed that (contrary to the statement of Winkler, whose ethylene probably contained ether vapour) ethylene is quantitatively absorbed by bromine, and this method was therefore used for determination of ethylene and its homologues. Hydrogen was estimated by fractional combustion.

Hexane.—Decomposition did not commence until the temperature reached about 700° C. At 700°—900° the results were about the same as in the previous experiments except that the acetylene percentage was very slightly higher. At temperatures between 900°—1,000° C. the hexane decomposed yielded on the average—

	Per Cent.
Methane	27·7
Olefines (mostly ethylene)	22·1
Acetylene	1·1
Hydrogen	2·4
Benzene	6·8—10
Carbon	3·3
Tar	29·2

The production of methane is thus much larger than at lower temperatures, that of the olefines less. Very small quantities only of the higher members of the paraffin and olefine groups were formed. Acetylene, benzene, carbon, and hydrogen appear to be secondary products, the main decomposition of the hexane seeming to be into methane, acetylene, and a highly complex tar. This latter carbonizes on nitration and is probably therefore of condensed olefine structure of low molecular weight, rather than of aromatic structure.

It appears that above about 900° C. the simple decomposition of the hexane is accompanied by destruction of the compounds formed.

Trimethylethylene.— Trimethylethylene yielded the following decomposition products.:—

—	At 750°—790°	At 930°—940°
	Per Cent.	Per Cent.
Methane	22·5	27·7
Olefines (mostly ethylene)	26·8	8·5
Acetylene	0·09	0·3
Hydrogen	0·3—1·3	1·7
Other gaseous products	..	4·5
Carbon	Trace	5·1
Benzene	..	8·0—13·0 } 47·1
Tar (containing a little benzene)	45·2	33·7—39·1 } 47·1

At 750°—790° the quantity of methane represented about one methane group from each molecule of trimethylethylene decomposed. The decomposition resembled that of hexane, in the olefines being mainly represented by ethylene, in the small quantities of benzene, acetylene, and hydrogen, and carbon formed, and in the quantity of methane produced. The tar, on the other hand, consisted mainly of olefines of higher molecular weight than the trimethylethylene. The almost entire absence of higher paraffins shows that at low temperatures the addition of hydrogen does not take place readily.

At the higher temperature (930°—940°) trimethylethylene showed similarity to hexane in the higher production of methane (more than 1 molecule to the molecule of initial substance) and of benzene.

At 1,050°—1,060° C. the gaseous products contain 50 per cent. of hydrogen, 39 per cent. of methane, and 5 per cent. of ethylene, but very heavy tars were formed and so much carbon separated that the tube very soon became stopped. The tar contained naphthalene and benzene. Only a trace (0·005 per cent.) of acetylene was found.

A few experiments were also made with benzene and acetylene.

Benzene.—Below 900 almost no decomposition occurred. Between 900° and 1,000° decomposition took place with the formation of higher aromatic condensation products. These products are not the same as those found in the tars from hexane and from trimethylethylene, so that the condensation products from the aliphatic compounds are not secondary products from benzene, but probably condensation products from benzene and acetylene.

Acetylene.—When acetylene was passed into the tube at 620° C. the temperature immediately rose to, and remained at, about 638°—645° C. The products were mainly liquid, ethylene forming only 3·2 per cent. and other gaseous products 4·2 per cent. of the initial gas. Of the liquid, about 40 per cent. was benzene and the remainder of higher boiling point. No naphthalene was observed. When acetylene was introduced into the tube at 770° the temperature rose to 790°, and then much carbon, and a very thick and heavy tar, containing both benzene and naphthalene, was formed. The gaseous products consisted of hydrogen with traces of methane and olefines.

The author could not observe the luminous decomposition of acetylene described by Lewes when his acetylene was quite pure, but noticed it directly a trace of air was present.
—L. T. T.

Shale Oil, The Production of, in France. F. Miron. Oesterr. Zeits. f. Berg u. Hüttenw. 1897, **45**, 80.

Shale, in the Permian formation, is quarried and distilled for the production of oil in the districts of Autun (Saône et Loire) and Buxières-la-Grue (Allier). The schist is broken up and carried on endless bands to the retorts, which are constructed of cast-iron. Distillation is carried on for 24 hours. From the 12th to the 20th hour the oil is distilled off, before and after this chiefly steam coming over. The vapours pass through two condensers, ammoniacal liquor being condensed in the first, and oil in the second. The average yield from 1 cb. m. of shale is 40—70 litres of crude oil, 50—65 litres of ammoniacal liquor, and 25—35 cb. m. of uncondensed gases. The crude oil is a dark fluorescent liquid with an alliaceous odour, and a sp. gr. of 0·87—0·91.—R. B. B.

[*Anthracene testing.*] *Alizarin, Analytical Methods employed in the Manufacture of.* A. G. Perkin. J. Soc. Dyers and Colourists, 1897, 88.

See under XXIII., *page* 563.

Benzene from Petroleum Spirit, A Means of Distinguishing. A. Gawalowski. Chem. Zeit. Rep. 1897, **21**, [35], 100.

See under XXIII., *page* 562.

PATENT.

Destructive Distillation of Carbonaceous Substances, Impt. in Vertical Retorts for the. A. C. T. Tarbrax. Cobbinshaw, near Edinburgh. Eng. Pat. 9944, May 11, 1896.

Over the lower mouth or end of each retort is fixed a bottom piece or table, and "a blade" extends across the middle portion of this, and is so arranged that it can be rocked by means of a rod and other gearing outside, on an axis approximately at the surface level of the bottom piece.
—R. S.

IV.—COLOURING MATTERS AND DYES.

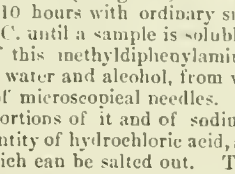
Nitroso-Methyldiphenylamine. Ch. Cloëz. Comptes Rend. **124**, (1897), 898—901.

It is a well-known fact that dimethylaniline is readily converted into a nitroso-derivative, but methyldiphenylamine does not yield a dinitroso-derivative, although it contains two phenyl groups. Nitroso-methyldiphenylamine can be obtained in good yield by dissolving 40 grms. of methyldiphenylamine (b. pt. 284°—287° C.) in 200 grms. of hydrochloric acid (sp. gr. 1·165 at 17° C.), cooling to −10° C., and gradually adding 15·2 grms. of sodium nitrite in 70 c.c. of water, maintaining the temperature below −5° C. Crystals of the nitroso-methyldiphenylamine hydrochloride then separate out, and, after solution in water, give, with sodium carbonate, a precipitate of the base. The base is stable, and can be kept indefinitely. It crystallises from methylic alcohol in greenish plates melting at 44° C., and can be employed for the production of dyestuffs. Thus by condensing it with dimethyl-*m*-amidophenol in an alcoholic or acetic acid solution it yields a dyestuff very similar to Capri blue, dyeing tannin-mordanted cotton. Moreover, on sulphonation it gives a wool dyestuff. By condensing 2 parts by weight of nitroso-methyldiphenylamine with 3 parts by weight of gallic acid, a product is obtained soluble in sodium carbonate, dyeing wool and silk violet blue, and giving similar shades on mordanted cotton, but somewhat redder than those obtained with gallocyanine. Methyldiphenylamine (185 grms.) can also be sulphonated by heating for 10 hours with ordinary sulphuric acid (100 grms.) to 160° C. until a sample is soluble in alkalis. The sodium salt of this methyldiphenylamine sulphonic acid is very soluble in water and alcohol, from which it crystallises in aggregates of microscopical needles. By treating equimolecular proportions of it and of sodium nitrite with the theoretical quantity of hydrochloric acid, a nitroso derivative is obtained, which can be salted out. This product serves for the preparation of sulphonated dyestuffs. Thus with gallic acid it gives a very bluish-violet which dyes evenly on wool, and approaches certain indulines in shade. With dimethyl-*m*-amidophenol a blue is obtained identical with that formed by sulphonating the direct blue already described.—T. A. L.

Amido-orcinol and Methylamido-orcinol, Derivatives of. F. Henrich. Ber. **30**, 1104—1110.

Amido-orcinol and methylamido-orcinol hydrochlorides are produced by reduction with stannous chloride and hydrochloric acid of the corresponding nitroso-compounds.

Oxidation of Methylamido-orcinol.—An alkaline solution of this compound is readily oxidised in air, or by the action of hydrogen peroxide, to a substance of the formula $C_{15}H_{14}O_4N_2$. It appears that the methylamido-orcinol is first oxidised to the quinone-imide $C_6H_2.CH_3(NH)(OCH_3)O$, and this product reacts with another molecule of the original substance to form a compound having the constitution—



(or the position of the methyl and methoxy groups may possibly be reversed). Methyl alcohol is also a product of the reaction. The oxidation product forms fine dark-red needles which melt at 253° C., and possess feeble basic properties.

Oxidation of Amido-orcinol.—The reaction in this case is more complicated and depends upon the relative proportions of amido-orcinol and alkali present. If to an aqueous solution of 1 mol. of amido-orcinol hydrochloride is added a solution of 2 mols. of caustic soda, oxidation takes place in the air in two or three days; ammonia is developed and the solution shows a deep violet colour with a reddish tone; on the addition of sulphuric acid pale red plates are precipitated consisting of a mixture of two colouring matters.

If, however, the proportion is three or more molecules of caustic soda to one of amido-orcinol hydrochloride, the solution after oxidation is red with a yellowish tone, and on the addition of sulphuric acid, a mixture of three colouring matters is precipitated, of which one can be separated from the other two by extraction with benzene. The author is at present engaged in the investigation of these colouring matters.—R. B. B.

Anthranilic Acid, Derivatives of. H. Rupe. Ber. **30**, 1097—1100.

Nitro-anthranilic Acid cannot be obtained by direct nitration, an acetyl group must first be introduced and nitro-acetylanthranilic acid is then prepared by careful treatment at a low temperature with nitric and sulphuric acids. In deacetylising this product, unless great care be employed, the carboxyl group is split off and paranitraniline formed. To obtain the result desired, the nitro-acetyl derivative is boiled with concentrated hydrochloric acid, when, on addition of water, a substance separates out which on crystallisation from boiling water forms yellow needles of nitro-anthranilic acid (nitro-amido benzoic acid, 5:2) with a melting point of 263° C., and identical with the compound prepared by Hübner from nitrosalicylic acid, ether, and ammonia. This compound possesses very feeble basic properties but it may be readily diazotised and the diazo compound conjugated with phenols or amines, when colouring matters are obtained dyeing similar shades to those yielded by analogous dyestuffs from paranitraniline, but possessing the property of dyeing with mordants by virtue of the carboxyl group which they contain. A specially useful colouring matter is obtained by coupling with amido-naphthol disulphonic acid H, the dyestuff yielding on cotton, with iron mordant a dull olive, with aluminium a pale mauve, and with chromium a full mauve.

Effect of Phenylhydrazine on m-nitro-o-chlorbenzoic Acid Ethyl Ether.—1 mol. of this ether (formed by passing hydrochloric acid gas into an alcoholic solution of the acid) is warmed for some hours on the water-bath with 3—4 mols. of phenyl-hydrazine. The dark red mass is crystallised from boiling alcohol, and yields, on cooling, fine needles of nitro-phenyl-indazolone. On concentration of the mother-liquor, long golden yellow needles of nitrophenylhydrazido-benzoic acid ethyl ether are formed—

$$NO_2.C_6H_3\left\langle\begin{array}{l}CO.OC_2H_5\\NH.NH.C_6H_5\end{array}\right.$$

and this on warming with alkalis is converted into nitro-phenyl-indazolone—

$$NO_2.C_6H_3\left\langle\begin{array}{c}CO\\NH\end{array}\right\rangle NC_6H_5$$

This compound dissolves readily in alkalis, and from a concentrated solution in caustic soda the sodium salt crystallises in red plates with a green reflex which possess distinct dyeing properties. Unmordanted wool is dyed a yellowish-green, and wool mordanted with chromium a yellowish brown.—R. B. B.

Nitramines and Nitrosamines [Liebermann's Reaction, &c.], Characterisation of Aromatic. J. Pinnow. Ber. **30**, 833.

See under XXIII., *page* 562.

Alizarin [Anthracene Testing, &c.], Analytical Methods Employed in the Manufacture of. A. G. Perkin. J. Soc. Dyers and Colourists, 1897, 81.

See under XXIII., *page* 563.

PATENTS.

New Colouring Matters [Yellows, Browns], Manufacture or Production of. Levinstein, Ltd., and I. Levinstein, Manchester. Eng. Pat. 7596, April 10, 1896.

Azo-dyestuffs obtained from dihydroxy and dihydroxy-sulphonic acids of the naphthalene series are converted into their nitroso compounds yielding nitroso-azo dyestuffs which dye chrome mordanted fibres yellow to brown shades, fast to light and milling. They also dye animal fibres from an acid bath. The diazo compound from 14 kilos. of *p*-nitraniline is combined in a weak acetic acid solution with 26·2 kilos. of the sodium salt of dihydroxynaphthalene sulphonic acid R. When the combination is complete a solution of 7·5 kilos. of sodium nitrite is added and the whole is run into 30 kilos. of hydrochloric acid (20° B.) diluted with water and cooled. After stirring for about 12 hours the new dyestuff is salted out. An analogous dyestuff is obtained by converting dihydroxynaphthalene sulphonic acid R into its nitroso compound, and after adding sodium carbonate, running in a solution of diazobenzene. In place of this latter, the diazo compound from either α- or β-naphthylamine or from *p*-amidonaphthol sulphonic acid may be employed, the resulting dyestuffs giving reddish-brown, brown, and deep-brown shades on wool from an acid bath, or deep brown to black-brown shades respectively on chrome mordanted fibres.—T. A. L.

Production of Dyes [from Cotton-Seed Oil], Impts. in. L. P. Marchlewski, Manchester, E. S. Wilson, Strood, and E. Stewart, London. Eng. Pat. 9477, May 5, 1896.

Dilute alkaline solutions extract from crude cotton-seed oil, a colouring matter for which the name gossypol is proposed. This may be isolated by precipitation of the fatty acids present in the solutions by appropriate metallic salts, followed by treatment of the residual liquors with an acid like hydrochloric acid, to decompose the alkali compounds left, the colouring matter being then precipitated in the form of flocks. It may be purified by repeated crystallisation from acetic acid and alcohol, and forms yellow crystals, easily soluble in alcohol, ether, boiling acetic acid, and aqueous alkali solutions, but insoluble in water.

Condensed with nitroso-dimethyl- and nitroso-diethyl-aniline in alcohol or acetic acid, gossypol yields dyestuffs which dye from an acetic acid or alcoholic solution, cotton and wool mordanted with iron, chrome, or tin, yellow-brown or brown. The dyestuffs are insoluble in water. Soluble derivatives, suitable for dyeing mordanted wool, are produced on sulphonating them. With α-nitroso-β-naphthol and β-nitroso-α-naphthol in concentrated sulphuric acid at a temperature of 55°—75° C., gossypol reacts, giving compounds which dye mordanted, and to some extent, unmordanted wool, brown.—E. B.

Basic Colouring Matters [Blues], Impts. in the Manufacture of. Read Holliday and Sons, Ltd., and H. Dean, Huddersfield. Eng. Pat. 11,309, May 23, 1896.

The patentees propose to substitute diamido-diphenyl-methane for aniline in the rosaniline blue process, and state that the dyestuffs produced, although similar in shade, possess different properties, their hydrochlorides being more soluble in water and have a greater affinity for cotton fibres. A mixture of 600 lb. of diamido-diphenylmethane, 100 lb. of rosaniline base, and 7 lb. of benzoic acid is heated to 160° C. for several hours, until a sample shows a pure greenish-blue colour. The melt is then purified by dissolving in dilute hydrochloric acid and precipitating the dyestuff with salt. The excess of base may be recovered from the mother-liquor by the addition of alkali. Another method given is to heat 500 lb. of diamido-diphenylmethane, 100 lb. of aniline salt or hydrochloride of the methane base, with either 100 lb. of amido-azobenzene or with 100 lb. of benzene-azo-α-naphthylamine to 160° C. The reaction is finished in the first case when a sample dissolves in dilute hydrochloric acid with a bluish-grey colour, and in the second, when it dissolves to a green solution in concentrated sulphuric acid. The purification of the dyestuff is carried out as in the first example.—T. A. L.

Colouring Matters [Cotton Greys, Browns, Blacks], Impts. in the Production of. Read Holliday and Sons, Ltd., Jos. Turner and Jas. Turner, Huddersfield. Eng. Pat. 11,370, May 26, 1896.

According to the specification, dyestuffs as indicated in the title are formed by heating with sulphur and sodium sulphide, benzene or naphthalene sulphonates or their

homologues, sulphonated nitroso-hydroxybenzene or naphthalene derivatives or their homologues and diamidonaphthalenes and their sulphonic acids, either alone or mixed with any of the following substances; phenols, naphthols, their nitro or nitroso, amido, amido hydroxy or dihydroxy derivatives, amido, amido hydroxy or dihydroxy derivatives of nitro or nitroso phenols or naphthols or sulphonic acids of any of the foregoing. A mixture of 3 kilos. of sulphur, 20 kilos. of sodium sulphide, and 4 kilos. of benzene disulphonic acid is heated in an iron vessel at 220°—320° C. for about 4 hours, until no increase in the strength of the colour is observed. The melt forms a black mass soluble in water, and dyes cotton drab shades from a hot or cold bath. For the 4 kilos. of benzene disulphonic acid may be substituted, the same weight of any other sulphonic acid above named, or 2 kilos. of a sulphonic acid and 2 kilos. of a phenol, naphthol, &c. The dyestuffs obtained may be purified by dissolving and precipitating in the usual manner. They are employed for dyeing purposes preferably in presence of sodium sulphide.—T. A. L.

Polyazo [*Cotton Brown*] *Dyestuffs.* S. Pitt, Sutton. From L. Cassella and Co., Frankfort-on-Maine, Germany. Eng. Pat. 12,142, June 3, 1896.

In tetrazo-azo compounds of the formula—

$$\mathrm{II} - N = N - P - N = N - C_{10}H_5(SO_3H) - N = N - \mathrm{I}$$

it is found the diazo group I acts much more strongly than the diazo group II, a property of which advantage is taken in the manufacture of certain trisazo compounds derived from paradiamines, as benzidine, *o*-tolidine, dianisidine, *p*-diamido-diphenylamine, and 1.3′- and 1.2′-naphthylamine sulphonic acids, and other dyestuff components.

It has been ascertained that after the diazo group I is saturated, the diazo group II can be combined with amines, which, in consequence of their susceptibility to the action of nitrous acid, would be affected by re-diazotisation, were they first combined, along with one of the naphthylamine sulphonic acids above mentioned, with a tetrazotised paradiamine.

Among the trisazo compounds obtainable as a result of this discovery, those which are produced by the combination of diazo group I with phenol and that of diazo group II with Chrysoïdines and Vesuvines are of special technical value. As an example: benzidine (18·4 parts) is tetrazotised and combined with 1·3′-naphthylamine sulphonic acid (22·3 parts). The intermediate compound obtained, is diazotised, the solution made slightly alkaline with sodium carbonate, and phenol (9·4 parts) added. After 5—10 minutes, the solution is rendered slightly acid, and Bismarck Brown (40 parts) is added. The dyestuff forms immediately. It dyes unmordanted cotton a brown colour very fast to light.

The azo compounds derived from *m*-phenylenediamine and the diazo compounds of sulphanilic acid, naphthionic acid, amido-salicylic acid, γ-amidonaphthol sulphonic acid, and 1.1′.3.3′-amidonaphthol disulphonic acid, employed in the above reaction in place of Bismarck Brown, give dyestuffs which dye unmordanted cotton respectively yellow-brown, cinnamon-brown, red-brown, and dark brown.—E. B.

Violet [*Azo*] *Dyestuffs suitable for Dyeing Wool, Production of.* S. Pitt, Sutton. From L. Cassella and Co., Frankfort-on-Maine, Germany. Eng. Pat. 12,556, June 8, 1896.

Azo-colouring matters, fast to acids and alkalis and unusually so to light, are produced by the action of diazotised 1.1′-amidonaphthol sulphonic acids on secondary α naphthylamines. Thus, a colouring matter dyeing wool violet from an acid bath, is obtained by diazotising 1.1′.3.3′-amidonaphthol disulphonic acid (32 parts) at 10°—15° C., and adding, at about 60° C., a solution of ethyl-α-naphthylamine hydrochloride (prepared from 18 parts of the base). Analogous compounds are obtained from the 2.4- and 3′.4-disulphonic acids of 1.1′-amidonaphthol and from methyl-α-napthylamine.—E. B.

Cotton Colouring Matters [*Yellow-Orange*], *The Manufacture and Production of New.* A. G. Green and A. R. Wahl, Manchester. Eng. Pat. 12,922, June 11, 1896.

By the action of caustic soda lye on *p*-nitrotoluene sulphonic acid, Walter, in 1883, showed that condensation products were formed, which dyed unmordanted cotton, orange-yellow shades fast to light (Bull. Soc. Ind. Mulhouse, 1887, 99). These products are known commercially as Sun Yellow, Curcumine S, and Direct Yellow G. According to Eng. Pat. 2664 of 1888 (this Journal, 1889, 41), by performing this condensation in presence of oxidisable substances, the dyestuffs known as Mikado Orange and Mikado Brown are produced. It was subsequently shown that these are reduction products of the first condensation products and can be obtained from them according to Eng. Pat. 23,672 of 1892 (this Journal 1893, 1023). A similar dyestuff is obtained (Eng. Pat. 16,708 of 1893; this Journal, 1894, 801) by oxidising diamidostilbene disulphonic acid with alkaline hypochlorites. A great similarity to all these substances is shown by the dyestuffs known as Oxyphenine, Chloramine Yellow, and the like, which are obtained by the action of alkaline oxidising agents on dehydrothiotoluidine sulphonic acid and primuline. The patentees have discovered an intermediate series of dyestuffs by condensing *p*-nitrotoluene sulphonic acid with a dehydrothio compound by means of weak aqueous caustic soda. The products formed dye unmordanted cotton, orange-yellow shades fast to light, chlorine, alkalis, and acids. When these new products are subjected to the action of reducing agents they are converted into new dyestuffs giving bright orange shades on unmordanted cotton and possessing the same valuable properties as the foregoing. The following quantities are employed in the first method:—A solution containing 28 lb. of sodium *p*-nitrotoluene sulphonate (85 per cent.) and 17 lb. of sodium dehydrothio-*p*-toluidine sulphonate in 10 gals. of water, is heated with 0·5 gal. of caustic soda lye (24 per cent. NaOH) for 3—4 hours at 90°—100° C. The product separates as a bright orange precipitate and is filtered and dried. It dyes unmordanted cotton a bright orange-yellow fast to light, chlorine, alkalis, and acids. It is less soluble than curcumine and can be readily salted out. Dilute hydrochloric acid completely precipitates it from its aqueous solution, whilst curcumine under similar conditions gives no precipitate. It is further distinguished from curcumine in being readily soluble in a strong solution of sodium bisulphite. In order to convert it into its reduction product, 50 lb. of the dyestuff obtained according to the previous example, are dissolved in 80 gals. of water together with 2 gals. of caustic soda lye. A solution of 10 lb. of glucose in 10 gals. of water is then added and the mixture is boiled until a sample dissolves in concentrated sulphuric acid, with a blue colour. After salting out, the product is filter-pressed and dried. It forms an orange-brown powder and dyes unmordanted cotton reddish-orange shades fast to light, alkalis, and acids. Its aqueous solution gives a reddish-brown precipitate with dilute acids, whilst Mikado Orange under similar conditions gives a brownish solution but no precipitate. The shade can be varied by employing a different quantity or different quantities of the reducing agent.—T. A. L.

Azo-Colouring Matters, Impts. in and connected with. I. Levinstein and Levinstein, Ltd., Manchester. Eng. Pat. 14,144, June 26, 1896.

According to Eng. Pat. 22,641x of 1891 (this Journal, 1893, 258), the 1.1′-naphthylamine sulphonic acid cannot be used as a "middle component" in place of α-naphthylamine. Similarly, in Eng. Pat. 2789 of 1892 (this Journal, 1893, 143), it is stated that of the naphthylamine sulphonic acids which Clève obtained from naphthalene β-sulphonic acid, only the β and δ acids were suitable. The patentees confirm these statements so far as they relate to the tetrazo derivatives of benzidine and its homologues, but find that the same restrictions do not apply when *p*-phenylene diamine is employed. Tetrazo dyestuffs may be obtained from *p*-phenylene diamine either by means of *p*-nitraniline or *p*-amido-acetanilide, the azo compound obtained being

reduced or hydrolysed respectively. The diazo compound from 13·8 kilos. of *p*-nitraniline is run into a neutral solution containing 24·5 kilos. of the sodium salt of 1.1'-naphthylamine sulphonic acid. The nitro-azo dyestuff is filtered off and the press cake dissolved in 300 litres of hot water together with soda or other suitable alkali, and reduced by adding sodium sulphide. The colour changes from bluish-violet to orange-red, and the reduced azo dyestuff, which may be salted out, becomes soluble in dilute hydrochloric acid. It is diazotised with 14·4 kilos. of sodium nitrite and 10 kilos. of hydrochloric acid (20° B.), forming a sparingly soluble brown powder, which is added to an alkaline solution of 48 kilos. of γ-amidonaphthol sulphonic acid. After stirring for 12 hours, the mixture is heated to 60° C., salted out, filter-pressed, and dried. The dyestuff gives deep indigo-blue shades on unmordanted cotton, dyes wool deep black from an acetic acid bath, and may be further diazotised and combined on the fibre.
—T. A. L.

Colouring Matters [Browns; Blacks] from Dinitronaphthalenes, The Manufacture and Production of. B. Willcox, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 20,250, Sept. 12, 1896.

An extension of Eng. Pat. 7766 of 1896 (this Journal, 1897, 436) applying the process there described to 1.4'-dinitronaphthalene. About 200 kilos. of this substance, 200 kilos. of grape sugar, 800 litres of water, 520 kilos. of caustic soda lye (24 per cent. NaOH), and 550 kilos. of sodium bisulphite solution (40 per cent. $NaHSO_3$) are heated on a boiling water-bath until the dinitronaphthalene is dissolved. After adding 10,000 litres of hot water the solution is filtered and acidified with 700 kilos. of hydrochloric acid (32 per cent. HCl), boiled and allowed to cool, when the dyestuff separates as a blackish-brown powder, which dyes wool brown from an acid bath. By employing mixtures of 1.4'- and 1.1'-dinitronaphthalene, shades varying from violet-black to black may be produced, and the treatment with bisulphite and the reducing agent may be either simultaneous as in the example given or consecutive.
—T. A. L.

New Colouring Matters [Hydrazone Compounds] of the Triphenyl Methane Series, and the Method of Producing the same. J. Ville, Paris. Eng. Pat. 27,775, Dec. 5, 1896.

Rosolic acid (10 kilos.), hydrazine sulphate (13 kilos.), and caustic soda (4 kilos.) are heated together at about 100° C. for 24 hours, giving successively a red, then a blue, and, finally, a violet colouring matter. Methyl- and ethyl-hydrazine may be employed in place of hydrazine. The colouring matters formed are of the same class as those described in Eng. Pat. 16,039 of 1896 (this Journal, 1897, 437), and are similarly applied in dyeing.—E. B.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

Silk Weighting in Switzerland, Regulations concerning. Färber Zeit. 8, [10], 161—162.

The silk weavers of Zürich have entered into an agreement with the Swiss silk dyers which forbids the weavers to import or to use, and the dyers to dye, any silk, whether of home or foreign origin, that has been weighted to such an extent as to seriously impair the strength of the fibre, on pain of heavy fines. The agreement is for one year, dating from April 1, and its conditions are to be rigorously enforced.—I. S.

Silk Weighting in Germany. Leipziger Färber- u. Zeugdr. Zeit. 46, [6], 243. (See also preceding abstract.)

Following the example of the Swiss silk weavers and dyers, with respect to the limits of the weighting of silk, the silk dyers of Crefeld have entered into an agreement which stipulates that the maximum degree of weighting for taffetas in the future shall be 30—40 per cent., and for other kinds of silks 50—60 per cent. The majority of German dyers and manufacturers are said to have promised their adherence to this regulation.—I. S.

Silk, The Finishing of. H. Silbermann. Färber Zeit. 8, [9], 136—137.

In many cases the appearance or "feel" which silk material is to have, is determined by the treatment of the yarn. Such goods are merely cleaned or scoured after leaving the loom, to fit them for the market. A piece that requires to be dressed receives for a soft finish a dressing with a composition consisting of starch or dextrin, gelatin, glycerin, and emulsified oil. The relative proportion and consistence of the preparation varies according to the quality of the cloth. For a stiffer finish the dressing employed consists of starch, soluble glass, and gelatin. For a very stiff finish the goods are first padded with a silicate of soda solution and tannin, and after a heavy squeeze, receive a dressing composed of dextrin, silicate of soda, gelatin, and emulsified oil. The dressing should be coloured the same shade as the silk to which it is to be applied.
—I. S.

Wool, Scrooping and Lustreing. E. Thiele. Färber Zeit. 8, [8], 120—121. (See also this Journal, 1896, 589—590; 1897, 41.)

To obtain a maximum of scroop and lustre with a minimum of discoloration the author recommends the following processes, the quantities given being in each case for 10 kilos. of knitting yarn:—

(1.) *For Light Shades.*—1·5 kilo. of bleaching powder in 500 litres of water, to which are added 1·5 to 2 litres of concentrated hydrochloric acid. The yarns are worked in this bath for 30 to 45 minutes at 60°—70° C.

(2.) *For Dark Shades.*—3 kilos. of bleaching powder and 1·5 litre of concentrated hydrochloric acid in 500 litres of liquor. The goods are worked for 45 minutes at 50° C.

(3.) *For Light or Dark Shades.*—To 400 litres of a solution of bleaching powder of ½° B., 3 litres of concentrated hydrochloric acid are added, and in this the goods are worked for 30 minutes at 50° C. For light shades the yarn is bleached with an aqueous solution of sulphurous acid.

The goods are "soaped," either before or after dyeing, for 10 minutes at 50° C., with 2 grms. of soap and 12 c.c. of sulphuric acid per litre.—I. S.

Silk Weighting, Estimation of. Leipziger Farber- u. Zeugdrucker Zeit. 46, [6], 259.

See under XXIII., *page* 564.

PATENTS.

Material suitable for Belting, Carding Cloths, Soles of Boots, and like Uses; Impts. in the Manufacture of. F. W. Oliver, London. Eng. Pat. 10,104, May 12, 1896.

The material described, consists of two or more layers of cotton, jute, or other fabric saturated with and cemented together by liquid celluloid. When an asbestos fabric is employed, the material may be used as a packing for joints.
—R. B. B.

Waterproof Fabrics, Impts. in the Manufacture of. F. W. Oliver, London. Eng. Pat. 10,105, May 12, 1896.

In the case of certain fabrics, *e.g.*, stockinettes, which are too elastic and porous to admit of a thin film of celluloid being directly formed upon them, a transfer paper is first coated with celluloid solution, the film being then moistened with solvent and applied to the surface of the fabric, after which the paper is washed away.—R. B. B.

Material suitable for Floor Coverings and other Industrial Purposes, New or Improved Process for the Production of. H. Karle, Seckenheim, Germany. Eng. Pat. 2934, Feb. 3, 1897.

Cellulose, which must be free from acid, is worked up with an equal bulk of linen or cotton fibre into a thin pulp. The pulp is poured upon a fine sieve, a fleece of raw cotton or cotton wool is spread over it, and on this another layer of pulp. The slab, thus formed, is pressed between rollers, dried by heat, rolled upon a drum between steel wire webbing, and saturated with a jelly made from hair and calcium sulphohydrate (CaH_2S_2), then dried and again pressed. When dry, the material is fulled or milled with

soap powder, treated with alkaline lye, dried, further treated with casein and borax, or with glycerin and lead oxide, and finally passed between calendering rollers.—R. B. B.

Artificial Silk Thread, Impts. in the Manufacture of, and Apparatus therefor. R. W. Strehlenert, Stockholm. Eng. Pat. 3832, Feb. 12, 1897.

A defect in the apparatus hitherto used for the manufacture of artificial silk is that, whenever a thread breaks between the mouthpiece and the bobbin on which the fibre is wound, it is necessary to stop the working of the apparatus. According to the present improvement several fibres are pressed out from a series of mouthpieces to a common point under water, where they are twisted together before being wound on the bobbin. The mouthpieces are attached to one or more cylinders, which receive a rotary motion in a horizontal plane, and motion in a similar direction is imparted to the water into which the threads are passed. It is claimed that by this device a broken thread is immediately caught up by an adjacent strand, and that uninterrupted working of the apparatus is thus secured.—R. B. B.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Dyeing Process, A Contribution to the Study of Reactions in. M. A. Reychler. Bull. Soc. Chim. 1897, **17**, 449—155.

When wool which has been well scoured and washed, is boiled in water, the liquid is found to be neutral to litmus and phenolphthalein, but gives a brown coloration with Nessler's reagent, and on distillation yields an alkaline distillate containing ammonia; there appears, therefore, to be present in the liquid, either an organic ammonium salt or an amido compound.

In the author's experiments, the same sample of wool was repeatedly boiled in water, and the various resulting liquors examined in several respects. The water originally possessed an electrical conductivity of 3—4 millionnèmes; *i.e.*, 1 c.c. offered a resistance between $\frac{10^6}{3}$ and $\frac{10^6}{4}$ Siemens units. After boiling 72 grms. of wool in a litre of water, the conductivity had increased to 398×10^{-6}; the residue from evaporation of this liquid contained lime, magnesia, potash, soda, and traces of silica, alumina, and ferric oxide; whilst one-half of the liquid yielded after distillation 0·045 grm. of ammonium chloroplatinate. After boiling five times in fresh water, the conductivity of the final bath was 45×10^{-6}, and the amount of ash was greatly reduced. Treatment with water at 75° C. twice repeated, reduced the conductivity of the liquid to 19×10^{-6}. Half the sample of wool was then treated with $\frac{1}{2000}$ normal hydrochloric acid at 75°, and the other half dyed in a solution of magenta of corresponding strength. In the first case, the conductivity of the bath was 58×10^{-6}, in the second case 61×10^{-6}—a very slight difference. Probably in dyeing, the Rosaniline hydrochloride is at once dissociated, the base combining with certain groups in the keratin molecule, and the acid behaving as if in the free state.

Widely divergent results are obtained by the treatment of wool with dilute hydrochloric acid, and by the addition of this acid to the water in which wool has been digested; for, whereas in the former case the conductivity of the acid liquid is reduced from 160 to 60 units, in the latter case it is not sensibly diminished, the bases present in the liquor apparently not sufficing to neutralise the acid. The conductivity of 60 units shown by the liquid in which wool has been treated corresponds to that of solutions of certain metallic chlorides of corresponding strength ($\frac{1}{2000}$ normal), the acid in this case being apparently entirely neutralised by the bases extracted from the wool.

When the amount of hydrochloric acid in these liquors is estimated by means of silver nitrate, it invariably appears greater than should theoretically be the case, and the author attributes this to precipitation by silver nitrate of some extractive principle of the wool.—R. B. B.

Dyeing of Cotton Yarn, Recent Progress in. Oesterr. Wollen- u. Leinen-Ind. 1897, 113.

Two interesting processes of recent introduction are those by which "scroop" and a silk-like lustre may be imparted to cotton yarn. To obtain "scroop," the yarn, after dyeing, is worked successively in baths containing calcium acetate, soap, and an organic acid (acetic or tartaric), and finally dried without washing. For the production of a silky lustre, the cotton is mercerised in a stretched condition, according to the process of Thomas and Prevost (this Journal, 1896, 804).

A very important discovery is that of the production of a blue on cotton, which is said to be fast to bleaching, *i.e.*, to the action of chlorine. This is effected by means of Alizarin Blue F (Meister, Lucius, and Brüning), a highly purified product, applied to cotton mordanted with Turkey-red oil and chromium oxide; after dyeing, the yarn is steamed, washed, and soaped.

The use of Paranitraniline Red is rapidly extending, especially since it has been established that good results may be achieved without the use of ice. To effect this, sodium acetate must not be added to the diazo solution until the latter is required for use, and with this precaution the solution may be preserved for three days at 22° C. without decomposition.

A recent advance in the price of Aniline salt causes the competitors of Aniline Black among the direct colours to be regarded with special interest. Fast Black B (Badische Anilin u. Soda Fabrik) is dyed cold in as concentrated a liquor as possible, with the addition of an amount of sodium sulphide equal to the weight of the dyestuff. The yarn is entirely immersed in the bath for an hour, then washed several times with water, worked at 50° C. in a bath of alum and acetic acid, and shaded, if desired, with a little Methylene Blue, after which it is treated with copper sulphate to increase the fastness to light, and finally soaped. The colour is very fast to washing and to acids, but rubs off slightly; it appears very suitable for warp-dyeing. Other direct blacks are Diaminogene B (Cassella), Dianil black (M. L. and B.), Benzo-chrome black (Bayer), and Diamine jet black Cr (Cassella), the two latter being treated with potassium bichromate after dyeing. All these blacks are still somewhat dearer than Aniline Black, but this is partly counterbalanced by their greater ease of application, and the fact that they in no way impair the useful properties of the fibre.

The blue dyed with Diaminogene Blue and Diamine Azo Blue (Cassella) is proving a useful substitute for indigo, but its progress is hindered by the fact that many conservative consumers insist upon regarding the "rubbing-off" test as the only indication of the fastness and desirability of a blue colour—a relic of the days when indigo was practically without a competitor.

As regards machinery, there is little progress to report during the past year. The dyeing of cops makes but slow headway, and appears to be profitable only when the dyers are also manufacturers, and large quantities of standard shades are required.—R. B. B.

Imitation Gold Thread, A Cause of Oxidation of [and Tendering]. L. Vignon. Bull. Soc. Chim. 1897, **17**, 441—443.

For the production of imitation gold thread, a fine metallic ribbon, consisting of a core of copper coated with brass, is wound around a cotton thread which has been dyed yellow. These threads frequently become black or tarnished, and this may arise either from atmospheric causes or from acids or acid salts being left in the cotton fibre after dyeing. Tarnishing has, however, occurred in cases where it could not possibly be attributed to either of the above causes, and the author has traced the defect to the presence of common salt in the dyed cotton. He considers that the copper-zinc couple formed by the metallic ribbon may set up electrolytic decomposition of the salt, and that the liberated chlorine or hydrochloric acid either acts directly upon the metal, or the chlorine in conjunction with atmospheric moisture brings about oxidation. He therefore recommends that in dyeing thread intended for this purpose, salt should invariably be replaced by sodium sulphate.—R. B. B.

p-Nitraniline Red, Discharging finished. H. Schmid. Färber Zeit. 8, [10], 149—152.

Hitherto white or coloured effects on a *p*-nitraniline red ground were produced by means of resists; that is, substances which decomposed the diazo compound (*e.g.*, stannous salts, alkaline sulphites, &c.), with or without the addition of some dyestuff, were printed on the naphthol-prepared fabric before passing the same through the developing bath. This process, however, has many defects. The coloured designs are not fast, and cannot be fixed by subsequent steaming; and, for the sake of the white, the oil, which is essential for obtaining a bluish red, has to be omitted from the naphthol solution. The red is in consequence of a yellowish hue, which is turned yellower still by the action of the tin salt on the diazo solution of the developing bath. On account of these and other defects the want of a process that would make it possible to discharge the developed red, made itself felt. But the agencies and methods in common use for discharging azo colours were found to be inadequate for this purpose. The destruction of *p*-nitraniline red by reduction and splitting up of the compound into *p*-phenylenediamine and amidonaphthol according to the equation—

$$C_6H_4(NO_2)N:NC_{10}H_6.OH + 10H =$$
$$2H_2O + C_6H_4(NH_2)_2 + C_{10}H_6(NH_2)OH$$

presupposes an energetic source of hydrogen, not available by hitherto known methods.

But if a substance having a solvent action on the colouring matter be added to the reducing agent, the action of the hydrogen is considerably augmented. Such a substance is glycerin, in which the *p*-nitraniline red dissolves off the fibre on heating, but still more readily during steaming. Thus, by adding glycerin (or a glyceride) to a stannous salt of an organic acid, a perfectly white discharge on *p*-nitraniline red can be obtained. For this purpose the quantity of glycerin need not be large, as its action is continuous, dissolving fresh portions of red so soon as the part in solution has been reduced.

The process is as follows:—The discharge composition, consisting of an organic stannous salt, glycerin, and a thickener, is printed on the ready-dyed cloth in the usual manner, dried, and passed through a steam ager (5—7 minutes) at the maximum steam pressure. The ager must be free from oxygen; hence in presence of aniline black as part of the design, the goods are allowed to hang over night in an oxidation chamber at 36°—40° C. before steaming.

After steaming, the goods are immediately passed through a tepid acid bath (40 grms. of strong hydrochloric acid per litre) to remove the *p*-phenylenediamine and amidonaphthol formed during the process, and are then well washed to remove all traces of tin.

Where there is a white discharge only, the goods may with advantage be passed through an acidified solution of bleaching powder, whereby the white is still further improved, whilst the red not only does not suffer, but is brightened by chlorination.

The process is not applicable for discharging *α*-naphthylamine clarets, but can be used for the other analogous azo colours produced on the fibre.—I. S.

Aniline Black, The Influence of Lime Stains on. R. J. Flintoff. J. Soc. Dyers and Colourists, 1897, 107—108.

The author has observed, in some cases, stains on aniline-dyed cloth, owing to the imperfect development of the black. On examining the cloth, he found it to contain lime, which acted as a resist. Subsequent experiments have shown that small traces of lime are sufficient to prevent the complete development of the black.—I. S.

Benzochrome Black for Cotton Warps. Heinrich. Färber Zeit. 8, [8], 118—119.

The author recommends this dyestuff as a substitute that may be used for aniline black for the dyeing of cotton warps which are to be woven together with undyed wool.

The colour of such warps must be fast enough to stand the subsequent process to which the cloth has to be submitted during the dyeing of the woollen parts; and benzochrome black is said to answer every requirement. It is dyed at a boiling temperature with 5 per cent. of anhydrous sodium sulphate and 2 per cent. of soda ash. The goods are subsequently treated in a separate bath, at a temperature of 80°—90° C., with 2—3 per cent. of potassium bichromate, 2 per cent. of copper sulphate, and 1 per cent. of acetic or ½ per cent. of sulphuric acid.—I. S.

Chromium Mordant [on Cotton], An Improved. E. Knecht. J. Soc. Dyers and Colourists, 1897, 108—109.

Sodium or potassium bichromates, as such, are useless for the mordanting of cotton. Gatty, by passing cotton impregnated with potassium bichromate through an atmosphere of sulphur dioxide, obtained a precipitation of chromium hydrate on the fibre. Scheurer and Blondel (this Journal, 1892, 33—34) first suggested the use of sodium thiosulphate as a reducing agent along with potassium bichromate in the presence of excess of ammonia. The pieces are padded in this mixture, dried, and steamed under pressure for one to two hours. Subsequently Prud'homme, and simultaneously the author, made the observation, that in presence of ammonia a chromate and a sulphite have no action on each other; as soon, however, as the ammonia is volatilised, reduction takes place, and chromium hydrate is precipitated. Relying on this fact, H. Koechlin, at the author's request, has worked out the practical process, which is as follows:—Potassium bichromate, 50 grms.; sodium bisulphite of 56° Tw., 100 grms.; ammonia, 100 grms., in one litre. Pad in this solution, dry, steam for two minutes in a rapid ager, and wash. W. H. Gardner obtained much better results than with the above by increasing the bisulphite to 260 grms., the other proportions remaining the same. Excellent shades, it is said, are obtained in dyeing with alizarin colours on this mordant; but in most cases some lime salt should be added to the dye-bath.—I. S.

Oxycellulose, A Curious Formation of. E. Knecht. J. Soc. Dyers and Colourists, 1897, 109.

If calico be first mordanted with chromium, and then treated warm, along with ordinary calico, with a 3 per cent. solution of hydrogen peroxide in presence of ammonia or caustic soda, the chromium hydrate rapidly dissolves, forming the yellow chromate; the mordanted material at the same time becomes more and more tender, until disintegration sets in, whilst the other unmordanted piece is not materially affected. If both pieces be steeped in methylene blue, the tendered one dyes a full shade, whilst the other is only tinted. These facts point to the formation of oxycellulose, but the author was unable to find an explanation why this substance should be formed in the one case and not in the other.—I. S.

Lactic Acid as an Assistant in Wool Chroming. P. Fuchs. Färber Zeit. 8, [9], 133—135. (See also this Journal, 1897, 140.)

The author confirms the statement of C. Dreher (Färber Zeit. 7, 233) and of C. Archer (this Journal, 1897, 140), that satisfactory results are obtained with lactic acid only in the presence of sulphuric acid. The proportions recommended are 1·5 per cent. of potassium bichromate, 3 per cent. of lactic acid, and 1 per cent. of sulphuric acid of 66° B. In view, however, of the fact that by this method the chromium oxide goes too quickly on to the fibre, thereby involving the risk of unevenness, the author recommends the use of "lactoline," which is a mixture of 1 mol. of potassium lactate with 1 mol. of free lactic acid ("bilactate"); the method of application remaining the same as before.—I. S.

Gums for Thickening. C. Gassmann. Färber Zeit. 8, [9], 135.

See under XVI., *page* 548.

Waste Water from Print Works, The Purification of. R. J. Flintoff. J. Soc. Dyers and Colourists, 1897, 99.

See under XVIII. B., *page* 551.

PATENTS.

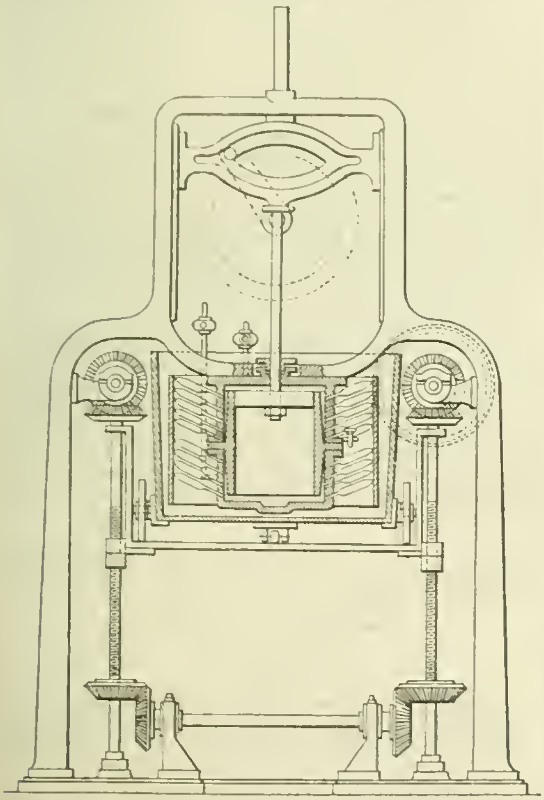
Bleaching Certain Vegetable Fibres, more especially those of the Urtica Family; Impts. in. A. F. B. Gomess, London. Eng. Pat. 10,936, May 21, 1896.

The fibres are immersed, first in a solution of a hypochlorite of an alkali or alkali earth, at 3°—8° B., for 1—10 minutes, and then in nitric acid at ½°—3° Tw. for 1—5 minutes, or in these solutions in the reverse order. After the treatment, the fibres are well washed in water in the usual manner.

—E. B.

Dyeing, Bleaching, Washing, and Rinsing Wound Yarns [*Cop Dyeing Apparatus*], *Impts. in Apparatus for.* G. Linkmeyer, "Herford," Germany. Eng. Pat. 8125, March 30, 1897.

Cops on hollow perforated spindles, are affixed to an air-tight cylinder, which is provided around its entire circumference with oblique perforations for their reception. The spindles are secured in position by a casing, which slides round them from below, causing them to be displaced



slightly upwards In this way they hold the casing without further means of attachment being required. The cylinder and cops along with the casing are enclosed in a tank, which is filled with water, &c. to a height above the highest position reached by the piston.

When the piston is moved, the action is similar to that of a double-acting pump; thus, on the downward stroke of the piston, the liquid in the bath passes through the perforations (above the piston) from the tank into the cylinder, whilst underneath the piston the reverse action takes place.

The motion of the piston is arrested at each end of the cylinder for an interval of time, the duration of which is determined by the length of curved slots in a crank crosshead.—E. B.

VII.—ACIDS, ALKALIS, AND SALTS.

Citric Acid, Preparation of, by Fermentation. Zeits. f. Spiritusind. 1897, **20**, 149.

In the German Patent, No. 72,957, of April 5, 1893 (see this Journal, 1894, 57, 80, 275, 772), the preparation of citric acid by fermentation of carbohydrates is claimed, and species of *Citromyces* are especially mentioned as bringing about this fermentation. The patent is now supplemented by a new claim (Ger. Pat. 91,891) based upon the discovery that the same result may be obtained by means of *Mucor piriformis.* The latter fungus is found on putrefying fruit, especially on pears and apples; its spore carriers only grow in a moist atmosphere, and form long white filaments, terminated by brownish-black heads. It can readily be obtained in pure culture by sowing the spores in a suitable medium, such as sugar solution, beer wort, steamed rice, starch paste, &c., the ordinary room temperature being favourable for its growth. The solution becomes acid from the formation of citric acid.—A. K. M.

Freezing Points of Dilute Sulphuric Acids. W. Hillmayr. Monatsh. f. Chem. **18**, 27—30.

Pfaundler and Schnegg, in 1875, and later, Pictet (this Journal, 1895, 35), have investigated this subject. The startling result of Pictet—that acids containing 6·77 per cent., and below, of H_2SO_4 freeze above 0° C.—induced the author to repeat his determinations. Six mixtures of the concentrated acid and water were made, containing, as nearly as possible, 1, 2, 3, 4, 5, and 6 per cent. of H_2SO_4, and their exact composition ascertained by analysis. The freezing points of these acids were then determined with great care, and the results compared with those of Pfaundler and Schnegg by means of the interpolation curve which those authors deduced from their results. The following table shows the close agreement of the present results with those older ones, and proves the error of Pictet's work with acids of similar strengths:—

Per Cent. H_2SO_4.	Freezing Point observed. ° C.	Freezing Point interp. (P. and S.)	Difference.
1·95	− 0°·78	− 0·74	+ 0·04
3·00	− 1·16	− 1·16	0·00
4·01	− 1·65	− 1·60	+ 0·05
5·13	− 2·05	− 2·12	− 0·07
6·02	− 2·55	− 2·58	− 0·03
8·02	− 3·61	− 3·69	− 0·08

If the molecular weight be calculated from the depression of the freezing point according to Raoult's method, the six acids give values very close to 49, and if the freezing points be then re-calculated from a molecular weight of 49, the following figures are shown:—

Per Cent. H_2SO_4.	Amount of H_2SO_4 to 100 H_2O.	Molecular Weight calculated from Depression of Freezing Point.	Depression of Freezing Point		Difference.
			Observed.	Calculated from Molecular Weight 49.	
1·95	1·99	48·4	0·78	0·770	+ 0·010
3·00	3·09	50·6	1·16	1·198	− 0·038
4·01	4·18	48·1	1·65	1·611	+ 0·039
5·13	5·41	50·1	2·05	2·097	− 0·047
6·02	6·41	47·1	2·55	2·483	+ 0·067
8·02	8·72	45·9	3·61	3·383	+ 0·227

The agreement is very close, though it is difficult to explain the half molecular weight.

Pfaundler and Schnegg tried to calculate that hydrate, the assumption of the presence of which in their solutions would make the freezing-point curve a straight line. They found that this would be, for strengths up to 12¼ per cent. H_2SO_4, 10·309 H_2O. If from the author's results the

molecular weight be calculated on the assumption that the acids are mixtures of water with the hydrate H_2SO_4, $10H_2O$ the results agree nearly with the half molecular weight 139; but on now re-calculating as before the depression of the freezing point from this molecular weight, the agreement is seen to be reasonably close only in the case of the very dilute acids:—

Amount of H_2SO_4, $10H_2O$ to 100 H_2O.	Depression of Freezing Point		Difference.
	Observed.	Calculated.	
5·860	0·78	0·801	−0·021
9·293	1·16	1·270	0·000
12·844	1·65	1·756	−0·106
17·040	2·05	2·329	−0·279
20·610	2·55	2·817	−0·267
29·455	3·61	4·026	−0·416

So that the assumption of the existence of this hydrate in solution is not borne out by the application of Raoult's method.—J. T. D.

Double Chromates. J. Zehenter. Monatsh. f. Chem. **18**, 48—55.

A boiling solution of potassium bichromate, neutralised with sodium carbonate, and then evaporated, first over a flame and then *in vacuo* over sulphuric acid, gave plates or columns, yellow in colour, having a specific gravity of 2·719 at 15° C., soluble in water to the extent of 64·2 parts in 100 at 14° C., losing no weight at 150°—160°, and giving, on analysis, figures expressed by the formula—

$$3K_2CrO_4 + Na_2CrO_4.$$

The same salt was obtained by Hauer by spontaneous evaporation of mixed solutions of potassium and sodium chromates, and also by Rose, who melted together potassium bichromate and sodium carbonate. Further evaporation of the mother-liquor from the above salt gave a crop of fragile crystals, sp. gr. 2·575, of which 100 parts of water at 14° dissolve 66·4 parts, and which proved to be the same salt with half a molecule of water of crystallisation. On longer standing a third crop was obtained, of yellow needles, very soluble, of the formula $Na_2CrO_4 + 4H_2O$. The yield was respectively about 40 per cent., 30 per cent., and 20 per cent. of the weight of the potassium bichromate used. A comparison of these results with Johnson's method of preparing sodium chromate, by the evaporation at the freezing point of a mixture of potassium bichromate and sodium carbonate, shows that temperature has an important influence on the reaction.

A strong solution of ammonium bichromate was neutralised with potassium carbonate at the ordinary temperature, the liquid filtered and mixed with a large excess of 95 per cent. alcohol, and the crystalline precipitate rapidly filtered and washed with alcohol, and purified by solution in a little water and reprecipitation by alcohol. The yellow, very soluble fine needles thus obtained have a specific gravity of 2·403, and give figures indicating the formula $3K_2CrO_4 + 2(NH_4)_2CrO_4$. On boiling their solution in water, ammonia is given off, and a mixture of potassium chromate and bichromate remains.

Substituting sodium for potassium carbonate in the last case, small prismatic readily soluble crystals, of sp. gr. 1·842, are obtained, the formula of which is $NaNH_4CrO_4 + 2H_2O$. This salt loses all its water *in vacuo* over oil of vitriol, and the dried salt is stable in the air at the ordinary temperature, but, on heating, readily gives off water and ammonia, leaving sodium bichromate.

Lithium carbonate, added at the ordinary temperature to potassium or to ammonium bichromate, gives in neither case any double salt. But when lithium carbonate is used instead of sodium carbonate in the first-described experiment, there separate after a certain amount of potassium chromate and bichromate, golden-yellow needles, so unstable as to be very difficult to dry. The analytical figures agree very closely with the formula—

$$K_2CrO_4 + Li_2CrO_4 + \tfrac{1}{2}HO,$$

and the specific gravity of the salt is 2·539.—J. T. D.

Cyanides, Conversion of Nitrites into. W. Kerp. Ber. **30**, 610—612.

Conversion of nitrite into cyanide was observed during a separation of cobalt and nickel by means of potassium nitrite in acetic acid solution when the alkaline filtrate of cobalti-potassium cyanide was accidentally evaporated to dryness over the open flame.

The formation of cyanide was traced to the interaction of acetate and nitrite, and the author has accordingly studied the decomposition which occurs when these salts are heated together.

He has found that a mixture in molecular proportions of fused sodium nitrite and sodium acetate explodes violently when heated, but that the reaction proceeds quietly when to this mixture is added an equal weight of soda. Such a mixture (12 grms. sodium acetate, 10 grms. sodium nitrite, and 22 grms. of sodium carbonate), when heated, begins to soften and swell out at 300°; at 375° it loses water and considerable quantities of hydrocyanic acid. So soon as the temperature of reaction is reached a strong glowing is observed, but there is neither explosion nor spurting. The product of the reaction is porous and friable, and, owing to separated carbon, is dark-coloured. The cyanide of sodium may be obtained pure by extraction with 70 per cent. alcohol and recrystallisation.

The reaction is represented by the following equation: $CH_3COONa + NaNO_2 = NaHCO_3 + NaCN + H_2O$, which the author considers to occur with the intermediate formation of nitroso-acetic acid, $NO.CH_2.COONa$.

The yield obtained varied with the manner of heating, but in no case amounted to more than 25 per cent., as calculated from the above equation.

Experiments in which sodium nitrite was heated with organic salts other than the acetate, showed that both sodium propionate and neutral sodium tartrate gave rise to cyanide, but that in the case of sodium formate the product consisted entirely of sodium carbonate.

With potassium nitrate, sodium acetate reacts violently when the two salts are melted together. On moderating the reaction by the addition of the double amount of sodium carbonate, a product consisting essentially of carbonate, is obtained, but mixed with this are small quantities of cyanide and larger quantities of cyanate. In this and other cases in which hydrocyanic acid results during the oxidation of organic substances with nitric acid, the author considers the result to be due to the intermediate formation of nitrous acid.—J. T. C.

Bromides and Iodides of the Alkalis, Preparation of. J. Knobloch. Pharm. Zeit. **42**, 190—191; Chem. Centr.-Bl. 1897, [1], 850—851.

See under XX., *page* 553.

PATENTS.

Caustic Alkalis, A Process for Obtaining or Extracting from Alkaline Aluminates, and Hydrate of Barium and Alumina from Barium Aluminate. D. A. Péniakoff, Huy, Belgium. Eng. Pat. 10,252, May 13, 1896.

Clear aluminate lyes are briskly agitated for a considerable time, whereby about four-fifths of the alumina present is precipitated. The lye drawn off, containing all the alkali, is then treated with a "quantity of alkaline earths equivalent to the amount of alumina remaining behind in the solution." The cleared caustic alkali solution is then decanted, evaporated, and melted down. Barium hydroxide may in like manner be obtained from barium aluminate.—E. S.

Sulphur, Impts. in Recovering from Sulphuretted Hydrogen. J. Brock and F. Hurter, Liverpool. Eng. Pat. 13,844, June 23, 1896.

Sulphuretted hydrogen is mixed with sulphurous acid in the proportions indicated by the equation—

$$2H_2S + SO_2 = 2H_2O + 3S,$$

and the mixture is passed into a Claus kiln, previously strongly heated. In practice, an impure hydrogen sulphide, such as that produced by treating alkali waste with lime kiln gases, is mixed with sulphurous acid obtained by the

combustion of iron pyrites or zinc blende, in such proportions that the free and combined oxygen present may be just sufficient to combine with the hydrogen of the hydrogen sulphide. If the sulphurous acid gas contain arsenic, it is purified before use by cooling, and then passing through a coke scrubber washed with sulphuric acid. The kiln is fed at first with a mixture of hydrogen sulphide and air, until the requisite temperature is reached, when the above described gaseous mixture is introduced. The kiln needs to be protected against loss of heat by radiation during the reaction. The sulphur produced, flows out from the Claus kiln into the sulphur pan.—E. S.

Brine and other Solutions, Impts. in Evaporating, and in Apparatus therefor. E. J. Duff and J. Brock, Liverpool. Eng. Pat. 14,951, July 6, 1896.

The brine is pumped into an upright tubular heater, supplied with steam from the evaporator, and passes into a second similar heater, divided by a vertical partition plate into two compartments, in one of which the brine descends and in the other ascends, and passes from the latter into a chamber having a perforated plate at the bottom, this chamber being on the top of the evaporator—a tall vertical cylinder down which the heated brine passes in thin streams. A current of air is forced through a horizontal tubular heater into a jacket surrounding the lower part of the evaporator, into which it passes through a series of holes, meeting the descending spray of brine, and passing from the upper part of the evaporator into the first of the tubular heaters described, thus "securing a second effect," as stated in one of the claims. The second tubular heater is heated from a separate source, as by the waste steam of an engine. The concentrated brine is received in a tank at the bottom of the evaporator, the tank extending outwards open to the air, so that the salts deposited therein may be withdrawn by hand; but the outer part of the tank is sealed off by a descending flange from the part immediately under the evaporator. The cooled brine is led into a second tank, from which, after addition of fresh brine, it is pumped through the system of heaters to be again concentrated.—E. S.

[*Alkali Percarbonates.*] *Chemical Products, Manufacture or Production of, by Electrolysis.* E. J. Constam and A. von Hansen, Zürich. Eng. Pat. 19,218, Aug. 31, 1896.

The method consists in electrolysing a saturated solution of a carbonate of an alkali metal or of ammonium at a temperature below 0° C. If a saturated solution, say of potash, made at − 10° C. and cooled down to − 16° C., be electrolysed, with or without a diaphragm, a bluish salt is obtained which is found to consist of percarbonate of potassium mixed with more or less potash or potassium carbonate, and to possess oxidising properties. The current strength employed may vary between 1 to 300 ampères per square decimetre of anode surface.—G. H. R.

Ammoniacal Preparations [*Liquid Ammonia and Camphor*], *Improved.* J. Pattison, Glasgow, N.B. Eng. Pat. 23,245, Oct. 20, 1896.

Camphor, either in lumps, ground, or in alcoholic solution, is added to liquid ammonia to render the latter more detergent, and to confer upon it a more agreeable odour, &c. —E. S.

Glover Towers, Gay-Lussac Towers, and the like; Impts. in, used, for instance, in the Manufacture of Sulphuric, Muriatic, and Nitric Acids. P. Jensen, London. From G. Lunge, Zürich, Switzerland, and L. Rohrmann, Krauschwitz, Germany. Eng. Pat. 6916, March 16, 1897.

The object of the invention is to compel the greatest possible amount of liquid to remain as long as possible in the apparatus, thus increasing the effect of the desired reaction, and reducing the height of the apparatus required, without diminishing the yield. To effect this, two new forms of insertion bricks are used. Each brick has a vertical through-slot surrounded on the top side by a channel recess, the inner edge of the channel being shallower than the outer edge, so that, when the bricks are built in, merely the outer edge shall be touched by the brick lying thereon, but not the inner edge. There will thus be a free running over of the liquid from the channel, over the inner edge, down on to the next lower brick. To prevent the acid adhering to the under side of the bricks, they are formed with a V-groove or recess all round the slot. Both sets of bricks are of the same outer dimensions, but one set has a longer and narrower slot than the other, so that the length grooves are wider in the one set and the end grooves in the other. The two sets of bricks are placed in alternate courses, with ordinary acid-proof bricks between, in such manner that the liquid trickling from the upper course "runs down upon firm surfaces of the next following course."—E. S.

Glover Towers and the like, Impts. in. P. Jensen, London. From L. Rohrman and H. H. Niedenführ, Krauschwitz, Germany. Eng. Pat. 7127, March 18, 1897.

In order to arrest the dust which deposits in a Glover tower where it first meets with solid surfaces and liquid, there is constructed above the gas inlet, and upon suitable girders, a nest of acid-proof bricks of about three courses, over which is a free space, and above that the girders on which the real filling devices are piled. Entry openings are provided in the part of the wall surrounding the free space, so that the dust-arresting bricks or other objects may be removed for cleansing, "without, as heretofore, removing the whole of the filling devices."—E. S.

VIII.—GLASS, POTTERY, ENAMELS.

Hollow Glassware, Decorating and Colouring. Central-Bl. f. Glas-Ind. u. Keramik, **12**, 51 and 71.

Gilding was practised as far back as the 16th century, but the gold, being merely fixed on a substratum of sodium silicate by burnishing, was not durable. The modern method of applying the gold in the form of precipitate along with calcined borax and oil, and then firing at a suitable ("gold fire") heat, is superior, but still the decoration is incapable of resisting various acids. The firing temperature is a most important factor, since if too low, the decoration is not fixed, and if excessive the gold is dull. Instead of burnishing the gilded surface with agate, the design is drawn on the glass with a flux of glass, minium, borax, and oil of turpentine, on which, when dry, the precipitated gold is applied, fired, and polished with a cloth.

Painting.—In this branch the sole progress is in the artistic character of the decorations, the colours and method of employment being the same as of old, *viz.*, a glass flux coloured by metallic oxides and applied by a brush in a turpentine medium, the operation being completed by firing in the kiln. Grey is obtained by means of a mixture of iron oxide and cobalt oxide; green, by the addition of chromium oxide; azure blue, by cobalt oxide, chromium oxide, and aluminium hydroxide; coffee-brown, by iron oxide, zinc oxide, cobalt oxide; orange, by red iron oxide; yellow, by sulphur preparations; red, by gold.

Engraving.—The older industry of grinding glassware has greatly declined, most of the modern ground glassware being of ordinary glass (not lead glass, as formerly), and artistic designs have been abandoned as less profitable than simple work. Engraved work commands a higher price for the finer specimens. The sand-blast is also used both for fine designs and ordinary engraved work.

Spun glass decorations can be applied to all descriptions of glassware, the articles being covered by threads of spun glass of various colours, applied by a machine which can be adjusted so as to lay the threads on at any desired distance apart. This method, combined with cameo decorations, is capable of wide application for glass services. The cameos are prepared by mixing equal parts of unburned porcelain earth and burned Rever clay in warm water to a paste, which is then left for three or four weeks (but stirred three or four times a week to insure intimate mixture), and then cast into the cameo mould. After removal from the mould, the cameo is slowly dried by

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exposure to the sun for a week, and gradually fired, commencing in the cooling kiln. The cameo may be coloured by the application of finely powdered coloured glass to the fresh, moist surface.

Opal Glass.—A mixture of barium nitrate, strontium nitrate, and stannous chloride is applied to the surface of a red hot glass plate, and the glass to be opalised, being held over the evolved fumes, assumes a number of beautiful blue-red shades. If previously converted into ice-glass by quenching in water, the effect of opalising will be heightened.

Ice-flower glass is prepared by dull-grinding the surface of the vessel, applying a coating of glue mixed with resin, and leaving to dry in a cool place; the contraction of the glue in drying, producing fractures in the dulled surface which resemble ice crystals.—C. S.

Pottery and Porcelain, Researches on. E. Damour. Bull. Soc. d'Encouragement, 1897, 191—215.

Some of the greatest of the practical difficulties encountered in the potter's art are due to the difference of the coefficients of expansion of the biscuit and the glaze. If the coefficient of the glaze be greater than that of the biscuit, the ware is liable to become crackled or "crazed"; if, on the other hand, the coefficient of expansion of the glaze be less, it is apt to scale. There are two possible means of obviating these defects, *viz.*: *First*, to use glaze and biscuit of nearly the same expansibility; *secondly*, to strengthen the glaze—*e.g.* by means of boric acid. The author considers that the true remedy for these difficulties is to be found in improved knowledge of the expansion coefficients of the materials used in pottery, and proceeds to describe the methods employed and the results obtained in his investigation. The preparation of prisms for measurement presents no difficulty in the case of biscuit, but it is otherwise with the glazes, which attack both platinum and porcelain vessels. Finally, the refractory material of the "Pasteur filter" was adopted, with the addition of a thin film of glaze. The expansion was measured by Fizeau's method (the displacement of Newton's rings) as modified by Le Chatelier. Figures are given in the paper showing the apparatus. In order that the rings may be visible, the bottom of the prism must be polished—a condition easily complied with in the case of the glazes, but which, with the biscuit, necessitates either the attachment of a microscope cover-glass by means of a film of balsam, or the addition of a thin layer of glaze. The author satisfied himself by trial with mixtures of known properties that it is sufficient to determine the law of expansion between 15° C. and 100° C., the values calculated by extra-polation being apparently correct to the limits of temperature attained in the kilns.

It has been found that the dilatation of the faience biscuit depends to a preponderating extent upon the fineness of grain and the physical condition of the materials. Small variations of chemical composition have, as a rule, but a trifling effect. The range of expansibility of the different materials employed, amounts to as much as 100 per cent.

In the case of the faience glazes also the range of expansibility is very great. The coefficient is, as a rule, greater than that of the biscuit. Alkalis, which increase the fusibility of the silicates, also increase the coefficient of expansion. The alteration of the superficial layer of the biscuit by the action of the glaze is *not*, in the author's opinion, an important factor in "crazing." He considers that this evil is to be combated in the porcelain manufacture "by the empiric study of the question under industrial conditions," and particularly the determination of the coefficients of expansion of the biscuit and of the glaze and the temperature of the kiln. In the case of *faience*, the greater range of variation of expansibility necessitates a more complete control of the operations through purely scientific study.—V. C.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

PATENTS.

Vulcanite, Hard Woods, or other Hard Materials; The Manufacture of a New or Improved Substance or Product suitable for Use as a Substitute for; or for other Uses or Purposes for which same may be applicable. H. Birkbeck, London. From P. W. Wierdsma and J. Kuipers, Leeuwarden, Holland. Eng. Pat. 2677, Feb. 5, 1896.

This invention relates to a product made from the refuse of potato-flour manufacture. The refuse consists of 85 per cent. of water, 5 per cent. of peel, and 10 per cent. of cellular and fibrous material, and only the last is useful for this purpose. It is freed from the peel, dirt, &c., by washing, grinding, and sifting; is bleached or dyed, if required; and finally, it is moulded under pressure into sheets or blocks.—R. B. B.

Cement, Plaster, Chalk, and the like; Improved Process and Apparatus for the Treatment of Articles of. A. J. Boult, London. From C. Tortorici and R. Grasso, both of Catania, Italy. Eng. Pat. 18,990, Aug. 27, 1896.

The articles are covered with warm tar or asphalt, and heated until the tar, &c. boils. The receptacle is finally closed hermetically. The volatile hydrocarbons are liberated in the process, and the articles become permeated with those which are less volatile.—V. C.

X.—METALLURGY.

Aluminium, Method of Browning. [*Protective Coat.*] Illustr. Zeit. f. Blechind. **26**, [13], 397.

To produce a brown surface on commercial aluminium, which is thereby protected against the action of moist air, water, and weak acids (*e.g.*, acetic acid), Göttig employs a 1 per cent. solution of ammonia, either alone or in conjunction with about 4 per cent. of chloride, acetate, or or other suitable salt of ammonium. In the former case the solution is rubbed over the aluminium, with which it combines, and in conjunction with the silica, iron, &c. present in the metal, forms a resistant surface (hydrogen being evolved). In the second method the metal is immersed for 1 to 3 hours, the aluminium and iron dissolved, being reprecipitated as hydroxides by the ammonium salts employed, the colour of the deposit in this case being darker than in the other.—C. S.

Gold Mines of Galicia, in Spain, The. Eng. and Mining J. 1897, **63**, 400.

The gold is found in the north-western part of Galicia, between the slate and granite formations. The ore is an iron pyrites containing a little arsenic. The lodes run generally with the formation, north and south; they are well defined, and average about 14 ft. in thickness.

The following are the principal mines which are being developed:—

Sagasta Mines.—About 2,000 m. of levels and crosscuts have been driven to fully prove the lodes. Last year, 1,095 m. were driven and 1,600 tons of ore extracted, equal, according to the assays, to 1,425 oz. of gold.

Fosas de Vila Mines.—These mines have been proved by sinking pits. The ore assays to 0·8—1·0 oz. of gold and 20 oz. of silver to the ton, besides a considerable quantity of lead.

Josefina Mines.—These are of arsenical iron pyrites. The assays give over 1 oz. of gold and 2 to 30 grains of silver to the ton.

Buena Esperanza.—The main lode is over 2,000 m. long, about 4 ft. wide, and more than 100 m. deep. The assays give 0·45 to 0·5 oz. of gold per ton, with less arsenic and more iron than the Josefina ore.

Virgen y S. Victor.—These are two mines on the same lode, in which the ore, though very rich, is of a complex nature.—A. S.

Gold from Cyanide Solutions, Precipitation of. L. Ehrmann. Chem. and Metall. Soc. of S. Africa. April 17, 1897, 3—7.

The precipitation of gold from cyanide solutions by means of zinc, is found to be greatly assisted by converting the zinc shavings into a copper-zinc couple by immersing them

for a short time in an ammonio-cupric solution, and by using this couple at a temperature of 80° C. With a ratio of cyanide to gold ranging from 14:1 to 16:1 the cold-extraction in 24 hours increased from 61—93 per cent. to 95—98·8 per cent. of the gold present in the liquors by the substitution of the copper-zinc couple for zinc. The original solutions in this case assayed from 2 to 7 dwt. of gold per ton. As large a percentage of the gold was precipitated in two hours at 80° C. as in 24 hours in the cold by the zinc and the copper-zinc couple respectively. Even when the ratio of KCy:Au was 205·5:1, extractions of 86·3 and 84·2 per cent. of the gold present in a solution containing 1 dwt. 17 grs. per ton were obtained by means of the couple, acting for 24 hours in the cold and for two hours at 80° C. respectively.—W. G. M.

Platinum-Silver Alloys; their Solubility in Nitric Acid. J. Spiller. Proc. Chem. Soc. 1897, [180], 118.

Referring to the published statements in the text-books, and particularly to those in Percy's *Metallurgy* and Bloxam's *Chemistry*, according to which 5 or even 9 per cent. of platinum followed the silver into solution when their alloys were treated with nitric acid, the author investigated the properties of 10 graduated alloys, constituted as follows:—Series I., containing 12, 9, and 5 per cent. of platinum; series II., containing 2, 1·5, 1, and 0·75 per cent. of platinum; series III., containing 0·5, 0·4, and 0·25 per cent. of platinum. These alloys were prepared by fusion of the requisite proportions of silver and platinum under a gas-air blow-pipe flame in shallow porcelain cups and then attacking with nitric acid of three different strengths; it was thus found that the ordinary concentrated acid of 1·42 sp. gr., warmed, proved the best solvent, but that even under the most favourable conditions no more than 0·75 to 1·25, mean 1 per cent. of platinum, could be dissolved along with the silver.

When diluted nitric acid of 1·2 sp. gr. was employed, the maximum amount of platinum taken up was only about 0·25 per cent.; whilst the highly concentrated acid of 1·50 sp. gr. proved altogether inappropriate, giving a bulky insoluble product, consisting of platinum black, intermixed with nearly the whole of the silver nitrate formed.

It would appear, then, that Berthier's account, quoted by Percy, and the statement in Bloxam's *Chemistry* are incorrect.

Iron Pyrites, On the Decomposition of. W. A. Caldecott. Proc. Chem. Soc. 1897, [179], 100.

In Watt's dictionary (1892 edition, vol. iii, page 64) it is stated that ferrous sulphide "is formed by the reduction of Fe_2O_3 on ferric salts by decomposing organic matter in the presence of sulphates," also that "finely divided yellow pyrites (FeS_2) oxidises in air forming chiefly $FeSO_4$." T. K. Rose (*The Metallurgy of Gold*, 1896, page 343) states that "FeS_2 is oxidised by air and water, $FeSO_4$ and free H_2SO_4 being formed."

In the treatment of auriferous pyritic Witwatersrand conglomerate, a large percentage of the ore is reduced by wet crushing in the battery to an impalpable powder. This fine material constitutes "slimes," and is carried in suspension by water into extensive dams, where it settles. The slimes leaving the battery are free from ferrous sulphide, but this compound may be detected in them a few days after deposition in the dams. The settled slimes form a clayey mass, practically impermeable to air and water, and consequently subsequent oxidation of the ferrous sulphide to ferrous sulphate proceeds extremely slowly.

When iron pyrites are crushed to an impalpable powder in an iron mortar ferrous sulphide is formed.

It thus appears that ferrous sulphide and not ferrous sulphate may be the first product of the decomposition of iron pyrites.

Owing to the almost total absence of acidity in slimes deposited as above, even when containing 0·89 per cent. ferrous sulphide, it is probable that under these conditions FeS_2 undergoes dissociation, and the sulphur is separated as such; the author is engaged in further investigating this subject.

Lead and the Oxides of Sulphur, The Reactions between. H. C. Jenkins and E. A. Smith. Proc. Chem. Soc. 1897, [179], 104—105.

The authors draw attention to the fact mentioned by one of them during the discussion on Mr. Hannay's paper (this Journal, 1894, 812, also 639), that when lead is heated to high temperatures and a current of sulphur dioxide passed through it, lead sulphide and oxide are formed. They have conducted experiments with a view to see whether the observations accounted for the anomalies hitherto met with whenever a full explanation of the chemistry of lead smelting in the reverberatory furnace has been attempted. Mr. Hannay had sought to explain these anomalies by the assumption that a volatile compound, PbS_2O_2, was formed, basing his evidence on an observation that in some experiments of his where air was passed over heated galena, he only obtained one-half of the lead in the metallic state. He gave the equation $2PbS + O_2 = Pb + PbS_2O_2$ as representing what occurred. The experiments have been carefully repeated by the authors, who find that the ratio of lead obtained to lead volatilised is not constant but varies, and is entirely dependent on the velocity of the current of air, indicating that no such reaction exists and that the ratio found in Mr. Hannay's experiments was the result of the accidental coincidence of his experimental conditions.

The authors, on continuing the experiments, found on heating lead sulphide and lead sulphate in a vacuum, that a reaction having sulphur dioxide as a product occurred, and they found that the amount of residual lead sulphide and oxide was dependent on the duration as well as on the temperature of the experiment. They then proceeded to separately test the pairs of bodies supposed to be present, with a view to discover if secondary or reversed reaction occurred. On heating lead and lead sulphate, they always obtained lead sulphate in the product in amounts varying with the duration of the experiment and the length of time that it occupied.

On heating lead and sulphur dioxide together, they found that at moderately elevated temperatures lead sulphide and sulphuric anhydride are formed in each other's presence, and that this leads to the formation, first of lead oxide and then of lead sulphate, the action being represented more or less completely by the equations $Pb + 3SO_2 = PbS + 2SO_3$, $Pb + 2SO_3 = PbSO_4 + SO_2$, or more simply—

$$2Pb + 2SO_2 = PbS + PbSO_4,$$

the main determining condition as to the relative amount of the products being that of the temperature at which the experiment is performed. One of the authors is continuing the investigation of the exact condition of the equilibrium.

The last of these equations occurring in the presence of excess of sulphur dioxide, is the exact inverse of the main ones of the metallurgy of lead as stated by Dr. Percy, and requires a supply of air by which to sweep away the sulphur dioxide as soon as it is liberated. The authors submit that, as the new equations quite account for the anomalies to which attention has been drawn, there is now no reason whatever to doubt that the equations given by Dr. Percy, $Pb + PbSO_4 = 2PbO + SO_2$, $PbS + PbSO_4 = 2Pb + 2SO_2$, as well as $PbS + 2PbO = 3Pb + SO_2$, represent the facts and form the basis of the metallurgy of lead.

Solid Alloys, X Ray Photographs of. C. T. Heycock and F. H. Neville. Proc. Chem. Soc. 1897, [179], 105—106.

In a previous communication (Trans. Chem. Soc. 1889, **55**, 666), the authors discussed the behaviour of gold in sodium, and the state of the gold in the solid alloy. They have lately examined alloys of gold and sodium by cutting thin sections from cylinders of the alloy of various concentrations, placing these on a photographic plate, and photographing them by means of the X rays.

On account of the much greater transparency of sodium to these rays as compared with gold, the individual crystals of the alloy were clearly shown. The plates were about 12 mm. thick, and the alloy had been allowed to cool and solidify very slowly. Pure sodium shows no crystalline structure, but an alloy containing 3 per cent. of gold

shows a mass of transparent sodium crystals, with dark spaces between the crystals where the gold has concentrated during the process of solidification.

A 10 per cent. alloy of gold shows the same phenomena, but the crystals of sodium are narrower, and the dark spaces occupy a larger area.

The appearance of the sodium crystals strongly recalls the fern-like pattern seen when ammonium chloride is crystallised on a microscopic plate, doubtless due to the fact that sodium, like so many other metals, crystallises in the regular system. An eutectic alloy of gold and sodium (23·1 per cent. gold) shows, as one would expect, scarcely any structure, because the crystals of gold and sodium separate out simultaneously, and are too minute to be detected by such a method.

A solution obtained by saturating sodium with gold at a temperature considerably above the melting point of sodium, shows a net-work of black, opaque needles, which are no doubt crystals of gold which have separated and grown to a considerable size as the liquid cooled.

No sodium crystals are here visible, as the groundwork consisted of the eutectic alloy. The authors have been able to demonstrate the internal structure of a solid alloy, and to show that the process of solidification is strictly comparable to that of a saline solution, the details being perfectly visible to the naked eye.

Gold, Liquation in Cyanide Bars. F. Stockhausen. Chem. and Metall. Soc. of S. Africa, April 17, 1897, 10—13.

The irregularity of assays when drill- or cutting-samples are taken from cyanide bars, is dwelt upon. The variation is sometimes due to imperfect mixing, sometimes to liquation. A dip-assay (from molten metal) alone is to be relied upon with this material. In the case of two bars exhaustively examined by means of carefully checked assays of the dip sample and of drillings and cuttings taken from different parts of the bars, the assays gave the following results :—

—	Bar 1.		
	Average Assay.	Extremes.	Maximum Difference.
Dip sample	809·0	808·9—809·6	0·7
Drillings	810·3	810·0—811·0	1·0
Cuttings	807·2	804·0—810·9	6·9

—	Bar 2.		
	Average Assay.	Extremes.	Maximum Difference.
Dip sample	679·5	679·1—680·3	1·2
Drillings	680·0	679·5—680·2	0·7
Cuttings	677·5	676·3—678·9	2·6

—W. G. M.

Iron, Specific Heat of, at High Temperature; Note on. W. N. Hartley. Iron and Steel Inst., May 1897. Ironmonger, 1897, **79**, 358.

The specific heat of iron increases at high temperatures in so remarkable a degree as to indicate a change in the molecular condition of the metal. From 0° to 660° C. the formula for the specific heat γ at temperature t is—

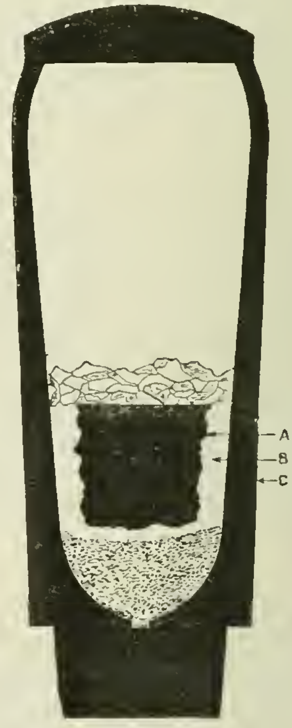
$$\gamma^t = 0{\cdot}1012 + 0{\cdot}000050666t + 0{\cdot}000000163998t^2$$

At the ordinary temperature, Regnault and Pionchon have found the specific heat to be 0·11379; but at 750°—1000° C. it is 0·213, at 954°—1006° C. it is 0·218, and at 1050°—1200° C. it is 0·19887. These numbers, however, are not in accord with Byström's, which was 0·4031 for 1400° C.

—W. G. M.

Crucible for Steel Melting, The Permeability of. J. O. Arnold and F. K. Knowles. Iron and Steel Inst., May 1897; Ironmonger, 1897, **79**, 317—318.

Pure Swedish iron was melted in clay pots, from 1 to 3 per cent. of aluminium was added, and the charges were then teemed. The resulting ingots were then broken up and re-melted, analyses of the metal being made before and after this treatment. It was found that the carbon percentage was increased from 0·04 to from 0·6 to 1 per cent., the silicon from 0 01 to from 0·04 to 0·5, and the sulphur from 0·01 to from 0·03 to 0·06 per cent. (the last-named proportion of sulphur being due to the use of a specially sulphury coke in melting), whilst the proportion of phosphorus had fallen from 0·02 to from 0·017 to 0·019 per cent., and that of aluminium from the percentage employed to from 0·07 to 0·2 per cent. At the same time the crucible became lined with a layer of alumina, shown at B in the annexed sketch, in which A represents the



steel, and C the clay wall of the pot. The explanation of the change is that the walls of the clay crucible are permeable at high temperatures, and that carbon monoxide penetrating into the metal became decomposed by the aluminium, which was thus separated as oxide, whilst the carbon of the gas served to carburise the iron; the sulphur contents of the steel were also increased owing to the penetration of sulphur dioxide into the crucible through the walls. The silicon was reduced from the clay of the pot. The loss of phosphorus is to be ascribed to the action of the basic (alumina) lining which had formed within the crucible. The oxidation of aluminium by carbon monoxide according to the equation $2Al + 3CO = Al_2O_3 + 3C$ had been shown by Arnold in 1891.—W. G. M.

Steel, The Carbide in. F. Mylius, F. Foerster, and G. Schoene. Ber. **29**, 2991—2996.

The authors have examined cast and forged steels containing from 0·1 to 3·0 per cent. of carbon, with the view of settling the composition of the contained carbide of iron, as a preliminary to work upon the changes which take place during the hardening of steel. They prepared steels from electrolytic iron and carbon from acetylene, which were melted together in little porcelain retorts by an oxygen

blowpipe. The steels were heated for four hours to low redness, cut into strips about 2 mm. thick, and submitted to a process of solution.

Electrolytic solution, making the metal the anode in a solution of zinc sulphate, was not found suitable; it is difficult to avoid electrolysis of the carbide itself, and oxidation easily takes place, either atmospheric or through too high current density, contaminating the residue with carbonaceous substances.

The same oxidation readily takes place when the iron is attacked by chromic and sulphuric acids; and the best method is to use dilute acid.

The following results were arrived at by using dilute hydrochloric, sulphuric, or acetic acid :—

1. Cast and forged steel contain the same carbide, which forms gleaming needles and plates, up to 1 mm. long. Arnold and Read's amorphous carbide was never seen.

2. The whole of the carbon may be present as carbide.

3. The residue of carbide is slowly soluble in dilute acids.

4. The yield depends on the nature and concentration of the acid, as the following figures (steel containing 1·3 per cent. of carbon) show :—

Acid.	Yield of Carbide per Cent. of Steel.	Percentage of Carbon in Carbide.
4 × normal HCl..........	5	6·42
2 × " "	8	6·50
¼ × " "	15	6·49
4 × " H_2SO_4........	12	6·71
¼ × " "	17	6·64
1 × " $HC_2H_3O_2$.....	19—20	6·30

5. The carbide is a definite compound, Fe_3C, existing as such in the steel, and which undergoes no change during isolation. Its constancy of composition was proved by fractional solution of the steel.

6. The carbide dissolves completely in warm hydrochloric acid, evolving gas, which is chiefly hydrogen (92·3 H, 1·4 N, 6·3 CxHy in one case: the hydrocarbon probably pentane). Liquid hydrocarbons may be also separated, but any solid residue means impurity in the carbide.

7. The carbide is not acted on by water at the ordinary temperature. Steam at 140° and above, forms Fe_3O_4, and gives off combustible gases; at a red heat, hydrogen and oxides of carbon are found in these gases, but no marsh gas.

8. The dry carbide is unalterable in the air (though under circumstances not yet defined, it is pyrophoric).

9. The moist carbide is easily oxidised by free oxygen or other oxidising agents, giving ferric hydroxide and a brown amorphous substance containing carbon hydrogen and oxygen.

10. The carbide melts unchanged, and, at a higher temperature, breaks up into carbon and a carbide containing about 4·6 per cent. of carbon.

The authors consider they have proved the identity, and the existence in steels, of Fe_3C, a body analogous to, but more stable than Mn_3C. They consider any statement as to its constitution premature. They mention that crystals found and described by Weinschenk in 1889 in a meteoric iron from Magura have been recently analysed by Cohen, and found to have the formula Fe_3C and the properties above described, and point out that microscopic research on steels is leading to the opinion that the carbide in all steels is identical. They suggest tentatively that the breaking up of the carbide above its melting point, and the consequent formation of lower carbides, may play a part in the hardening of steel.—J. T. D.

Cast-iron, Malleable. G. P. Royston. Iron and Steel Inst., May 1897. Ironmonger, 1897, **79**, 338—340.

Hard, brittle, white-iron castings may be softened either by oxidising the carbon or by changing it into the graphitic state. The former (Reaumur's) method is most generally used, and consists in heating the castings in oxide of iron (or in lime, sand, or bone-ash, in the presence of air); but in this case manganese and sulphur must be absent or else the elimination of carbon will be delayed. When oxide of iron is used for annealing, it is in part reduced to the metallic state. The process is necessarily slow, because the carbon has to travel to the outside of the bar before it becomes oxidised; this it does by diffusion, and more rapidly as the temperature is raised. The temperature must not exceed 1,000° C. lest the castings become deformed; and, on the other hand, as carbon does not exist in solution at temperatures below 660° C. any oxidation of the carbon would then be accompanied by oxidation of iron. The quickest result obtained was with a bar of white iron ¾-in. in diameter, which was decarburised after heating to 900° C. for 22 hours. The second process consists in merely heating the iron continuously to from 850°—650° C. It is accidentally applied, sometimes, in the cooling of large pieces of iron which have escaped complete decarburisation by the Reaumur process. The rationale is explained in a second paper on the Relation of Carbon to Iron (see succeeding abstract). Castings produced by this method are as soft as those obtained by Reaumur's process, but the presence of the free carbon as finely divided graphite prevents bending. Here the action is independent of the size of the casting, and large masses may be softened as rapidly as smaller pieces, the quickest result being the conversion of a white iron containing 3·8 per cent. into one with only 0·6 per cent. of combined carbon, within five hours. A new method of annealing is also described. The material is simply packed in a non-oxidising substance, and is thus heated to the freezing point of the metal (which is about 35° C. below the fusing point); it is thus instantaneously converted into a steel containing 1·5 per cent. of carbon, the remainder of the carbon being separated throughout the mass in the form of graphite. Care must, of course, be taken not to raise the temperature too high, or the iron will melt. The presence of manganese, tin, or chromium interferes with this reaction, but that of silicon, aluminium, or nickel is without effect. The author hopes shortly to be able to produce a soft iron containing graphite with the same speed as in the case of the steely material just described.—W. G. M.

Iron, Relation of, to Carbon at High Temperatures. G. P. Royston. Iron and Steel Inst., May 1897. Ironmonger, 1897, **79**, 340—343.

When high and low carbon steels, placed in contact, were heated to 900° C. for 12 hours in a vacuous porcelain tube, it was found that carbon had been transferred from the former to the latter in such a way that the loss of weight of the one corresponded exactly to the gain of the other, whilst the variations in the carbon percentages agreed exactly with the alterations in weight. This tends to confirm the view that the carbon is present in iron at a red heat in a state of simple solution. The explanation of the behaviour of white iron in the malleable cast-iron process would then be as follows:—White iron in the cold is a mixture of mass cementite and pearlite (cementite and ferrite). When heated to the recalescence point Ar_1 the cementite begins to decompose, and the iron dissolves the released carbon to the extent of 0·9 per cent. The pressure of this dissolved carbon then prevents the further dissociation of the carbide. As the temperature rises the solvent power of the iron increases, and at 1,030° C. it can dissolve 1·5 per cent. of C. But at this critical temperature the whole of the residual cementite decomposes, in spite of the carbon pressure around: the iron cannot, however, dissolve more than 1·5 per cent. of C at this point, and the remainder of the carbon is therefore deposited as graphite as fast as it is separated. When fluid (white) cast-iron is quenched, the bulk of the carbon forms cementite, and 1·5 per cent. remains as a homogeneous alloy with the rest of the iron; but when the iron is heated to 700° C. and quenched, the well-recognised alloy corresponding in composition to $Fe_{24}C$ (containing 0·9 per cent. C) is formed. In white iron at a red heat there are two opposing pressures: the dissociation pressure of the carbide, and the solution pressure of the carbon. The latter is normally the greater; but if the latter be removed, or if the former be aided, the decomposition may be continued. The tendency to crystallisation appears to be the only other force operative at this temperature. The crystallising point of pure iron is 750°; in

0·2 per cent. C steel it is 735°; and in 0·8 per cent. C steel it is 685° C. (AR_1). It would appear, therefore, that AR_2 in low carbon steels marks the crystallising point of the pure iron, while the presence of carbon in solution prevents crystallisation of the remaining iron until the point AR_1 is reached, at which the carbon passes out of solution into combination with iron as Fe_3C. Hence, in 0·2 per cent. C steels at 720° C., the mass should consist of pure crystallised ferrite intermixed with amorphous masses of iron containing carbon. The solution pressure of carbon in iron appears to exceed the "crystallisation pressure" of the iron. To sum up:—The pressure of carbon in solution is greater than either the dissociation pressure of Fe_3C or the crystallisation pressure of Fe alone, but less than that of the two pressures acting together. In the absence of tendency to crystallisation of Fe (*i.e.*, above 750° C.), slow cooling will not cause any deposition of graphite. In the absence of dissociation pressure (that is, if the iron contain less than 0·9 per cent. C) graphite is not produced by annealing. Below 620° C. it may be assumed that carbon is unable to combine with iron, for a malleable cast-iron containing the whole of its carbon as graphite, underwent no change even when heated for three hours to that temperature. And below AR_1 all the carbon other than graphite exists as Fe_3C, so that no diffusion of carbon can occur, and hence no oxidation of carbon can take place without a simultaneous oxidation of carbon.—W. G. M.

Open-Hearth Process, Combined, of Bertrand and Thiel; Notes on. E. Bertrand. Iron and Steel Inst., May 1897. Ironmonger, 1897, **79**, 329—332.

In this process (this Journal, 1897, 243) two furnaces are employed at different levels. In the first much, but not all, of the phosphorus is eliminated from the charge; the fact that it is not necessary to remove the whole in this furnace permits the use of less lime, and therefore of less slag in the first hearth, so that the flame acts upon the metal with greater intensity than it could otherwise do. The scrap is for the most part charged into the second, and the silicious and phosphoric pig iron into the first furnace, ore and lime being added to each as required. It is even possible to work with pig iron alone, whether high or low in silicon, the only provision being that the material should contain but little sulphur. When large proportions of pig iron are used, the hearth should be deeper than usual, because the ore and basic addition are more bulky, and because the boiling of such a charge is more lively than under the older system of working. Under somewhat unfavourable conditions, several charges of grey forge-pig, containing 3·8 per cent. C, 1·0 per cent. Mn, 1·6 per cent. P, and 1·0 per cent. Si have been worked by this process, magnetic Gellivara ore, with about 65 per cent. of Fe, being used in boiling. The finishing furnace was used only for finishing the heat, a small quantity of scrap being melted in it merely that it should not be empty whilst heating up. After treating several 12-ton charges of the forge-pig in the primary furnace, the average composition of sample ingots run as the metal was being transferred to the second hearth, was 2·2 per cent. C, 0·4 per cent. P, 0·05 per cent. Si, and 0·05 per cent. Mn. Hence one-third of the carbon and three-fourths of the phosphorus, with practically the whole of the silicon and manganese, are removed in the primary furnace, the behaviour of the phosphorus being the reverse of that in the basic Bessemer process. Owing to the length and low gradient of the lauder connecting the two furnaces, it was unsafe to reduce the proportion of carbon and phosphorus below that given above, as the metal would then have flowed too sluggishly. The time required in the primary furnace was about $4\frac{1}{2}$ hours, and in the finishing furnace about 2 to $2\frac{1}{2}$ hours; and there was, in the majority of cases, a gain in the actual weight of metal, ranging from 0·1 to 6·2 per cent. In no case was there a loss of more than 2·4 per cent.; this is due to the fact that the metalloids in the pig iron reduce more than their own weight of iron from the ore during their oxidation. It is believed that working with two primary furnaces to one finishing hearth, with the same pig as in these experiments, nine 15-ton heats could be made in 24 hours; and that by charging liquid pig iron the output could be still further very greatly increased. The slags from the primary furnace are very rich in silica (25—30 per cent.), and contain also about 13—14 per cent. P_2O_5, whilst those from the finishing hearth contain a larger proportion of lime in order to ensure complete dephosphorisation. Tables of analyses and tests of the charge and of the finished steel are supplied. An addendum has also been given, showing the result of working two heats of basic pig, containing 3·8 per cent. of C, 2·5 per cent. of P, 1 per cent. of Si, and 0·6 per cent. of Mn, with 2 tons of Gellivara ore and 1 ton of lime. After treatment in the primary furnace, the metal contained 1·2 per cent. of C, 0·55 per cent. of P, 0·03 per cent. of Si, and 0·05 per cent. of Mn; whilst the slag contained 19·6 per cent. of SiO_2 and 21·88 per cent. of P_2O_5, with only 6·34 per cent. of Fe. This slag could evidently be employed as a fertiliser.—W. G. M.

[*Iron.*] *Cold-Shortness, Influence of Phosphorus on.* H. Jüptner von Jonstorff. Iron and Steel Inst., May 1897; Ironmonger, 1897, **79**, 347—351.

The author seeks to account for the varying influence of phosphorus on iron under different conditions. Pig irons containing phosphorus evolve a portion of this element in the form of hydrogen phosphide (PH_3) on treatment with dilute acids, but much is at the same time left in the residues as iron phosphide (Fe_3P). When manganese is present in considerable quantities, however, the proportion of phosphorus as insoluble phosphide rapidly increases with that of manganese, in accordance with the formula Mn_3P_2. The phosphorus from pig iron is left by cupric chloride solutions as a pulverulent crystalline admixture, whilst the ground-mass is poor in phosphorus. Thus the phosphorus favours crystallisation in the less fusible constituents by the formation of easily fusible compounds, and these, being the last to solidify, do not separate in large crystalline planes. The author has now investigated the gases evolved by the action of dilute H_2SO_4 (sp. gr. = 1·1) at 18° C. on samples of steel, by passing them through Peligot tubes containing neutral silver nitrate solution; after acting in the cold, the solution was raised to the boiling point, and then air was drawn through the whole. It is thus shown that the cold-shortness of the metal increases with the proportion of the phosphorus that is evolved as hydrogen phosphide. Many specimens of steel showed no trace of cold-shortness, although their total percentage of phosphorus was higher than that of other steels which were markedly cold-short; in such cases the proportion of phosphorus given off as PH_3 was highest in the more brittle metals. It would seem that the phosphorus present as Fe_3P or Mn_3P_2 is practically without influence upon the properties of the metal, being distributed through the mass in the form of crystal grains, which only affect to some extent the machining of the metal owing to their hardness. Manganese, by assisting the formation of these crystals, therefore tends to counteract the prejudicial influence of phosphorus. The phosphorus, on the other hand, that is present in the ground mass in solution, and that is evolved as PH_3, is in a position to affect the character of the whole of the material. Further experiments with weld-iron confirmed this observation, and seemed to establish the fact that the quantity of separated phosphide (Fe_3P) in the metal is greater in the case of annealed metal, but less in the hardened material, than in the normal or "natural hard" iron. It would thus appear that, like carbon, phosphorus exists in iron in two conditions—as "hardening" (that which gives rise to PH_3) and as "phosphide" phosphorus, and that the inter-conversion of the two varieties takes place in a manner similar to that of the two carbon modifications, only that it is slower and less far-reaching. Behrens and Van Linge's view that the deposition of the phosphide takes place within the carbide is also supported. Since the hardening capacity of steel is reduced by an increase in the percentage of phosphorus, it appears that the latter element favours the separation of the carbide; but, on the other hand, the influence of the phosphorus increases with the proportion of the combined carbon. Conversely, however, carbon would seem to favour the separation of phosphide; and the phenomenon of cold-shortness is not solely dependent upon the quantity of hardening phosphorus present. It is necessary to take into

account the joint quantity of the carbides and phosphides present, or, in other words, the quantity of the eutectic alloy of the metal cooling from fusion, inasmuch as with an increasing quantity of the mother-metal the formation of crystals is facilitated in the portion of the iron that is first to solidify. Following up the idea of eutectics in the solidification of steel ingots, the material consists of crystals deposited from without inwards, with interspaces and cavities filled with the portion last separated. But on further cooling, there separates from this solidified solution either the solidified solvent, if the solution be dilute, or the dissolved material (carbide, phosphide, &c.) if it be concentrated. Thus each constituent of iron may exist in at least two different forms: carbon, phosphorus, and sulphur occurring in two, chromium in three, and manganese, tungsten, &c. in several different forms. The chief of these are:—(1.) *Dissolved Substances separated out from Concentrated Solutions*—*e.g.* graphite, ferrochrome, carbide, &c., which are always characterised by a more or less well-defined crystalline form. (2.) *The Solvent separated out from Dilute Solutions*—*e.g.* pure granular iron; these are always crystalline, but less definitely so than the members of Class 1. (3.) *Solidified Mother-Metal*, in which secondary separations occur. If Fe_3P and Mn_3P_2 have higher melting points than the final residue of the mother-metal, and are relatively but little soluble in this, the process of solidification may again reverse, and phosphides may separate once more, together with some carbide (corresponding to water of crystallisation in saline solutions); but as the whole mass must solidify shortly afterwards, the phosphide would not have time to crystallise. In this case, phosphorus would favour the separation of carbide and diminish the hardening capacity of the iron, because carbide separates simultaneously with phosphide, and, like salt crystals in supersaturated solutions, these secretions favour the further separation of the carbide (mother-liquor).—W. G. M.

Waste Products in Iron Manufacture. Eng. and Mining J., June 5, 1897, 566.

Americans are accustomed to claim for their iron and steel plants superiority over those of European countries in many points. In their blast furnaces they claim to have increased the capacity to a point far beyond anything known abroad, and they have been able to make iron at a low labour cost, in spite of higher average rates of wages. This has been secured largely by the use of machinery wherever possible, and by cutting down the amount of labour required to the lowest possible point. To a certain extent also, economy has been secured in fuel and materials; and all this without in the least reducing the standard of quality in the product.

In some respects, however, they have much to learn yet from their competitors. It is, perhaps, a consequence of having abundant supplies of raw materials that they have been careless about their use, and indeed rather inclined to wastefulness. This is especially apparent in the iron manufacture, where they have, as a rule, entirely neglected the by-products which, in other countries, form so important an element, that a German writer has suggested the possibility that before long the pig-iron made will become a by-product, and the blast furnace will be valued chiefly for its output of chemicals. They are considerably behind the English and very far behind the Germans in this respect, and there is corresponding room for improvement.

The most important element of saving in European practice is found in the use of the by-product coke oven, which is almost universal in Germany, and is gradually making it way in England. In Scotland, where the majority of the furnaces use raw coal, and not coke, for fuel, plants are found at a number of the iron works where the gases escaping from the furnace are treated for the purpose of saving the ammonia sulphate, tar, and other chemical products, while the purified gas is used for fuel in boilers. These plants, of course, serve the same purpose as the by-product oven, and are, perhaps, not especially to be recommended except under the peculiar conditions presented in Scotland.

In Germany the slag from the furnaces is used for making bricks, for roads and similar purposes, and new applications for this material are constantly being found. In America such uses are still comparatively rare.

One of the latest applications of a by-product is found at the Cockerill Works at Seraing, in Belgium, where the waste gas from a blast furnace has been for a year past used experimentally to run a gas engine. This has proved so successful—though coke is used for fuel in the furnace—that the company is now putting up two more gas engines of 150 horse-power each, and anticipates in time supplying a large part of its motive power from this source. Blast-furnace gas has also been recently utilised in the same way in Great Britain by Mr. James Riley, the well-known ironmaster. Every great iron and steel works should investigate possible uses for what are now their waste products, and, through the applications of science to industry, turn them into profits. The careful saving of by-products will soon be a necessity if iron-men are to hold their own and to build up the foreign trade which is within their reach.

Aluminium Bronze Colours, Danger of Explosion and Fire in the Manufacture and Storage of. R. Kayser. Zeits. f. öffentl. Chem. 3, 93; Chem. Centr. Bl. 1897, 833.

See under XVIII. B., *page* 551.

Telluride Gold Ores. R. Pearce. Eng. and Mining J., April 17, 1897, 376.

See under XXIII., *page* 561.

Steel, Hardening Carbon and Carbide Carbon in; Determination of. H. Jüptner v. Jonstorff. Iron and Steel Inst., May 1897; Ironmonger, 1897, 79, 351.

See under XXIII., *page* 561.

Calcium Carbide, New Economical Preparation of. H. Marx. Monit. Scient. 1897, 11, [4], 276.

See under II., *page* 524.

Blast Furnaces, Agricultural Value of Sulphate of Ammonia from. F. J. R. Carulla. J. Iron and Steel Inst., May 1897.

See under XV., *page* 547.

PATENTS.

Blast Furnaces, Impts. in. W. Gibbings, Widnes. Eng. Pat. 7436, April 8, 1897.

To keep the fuel away from the sides of the furnace and to confine the melting zone to the centre thereof (thereby rendering the furnace more durable), the fuel is fed from a receptacle supported on the top of the furnace at the centre. Such receptacle may form part of the flue of the furnace or be independent thereof.—R. S.

Sulphide Ores of Lead, Impts. in the Treatment of, Preparatory to Smelting the same, and Incidentally to obtaining Sulphurous Acid. T. Huntington and F. Heberlein, Pertusola, near Spezzia, Italy. Eng. Pat. 8064, April 16, 1896.

An intimate mixture of galena and lime (or other alkali earth) is heated to 700° C., and the temperature is allowed slowly to fall to about 500°, when the mixture is removed to a receptacle through which a current of air is passed. Much heat is generated, and the sulphurous acid given off is collected. This mixture fuses and is then ready for reduction of the metal in a suitable furnace. The reactions assumed to take place consist in the oxidation at the higher temperature of lime into calcium dioxide, which at the lower temperature reacts on the lead sulphide to reproduce lime, and lead sulphate. When the heated mixture, now containing lead sulphate with lead sulphide, is subjected to the action of air, they react to form lead oxide and sulphur dioxide, as shown in the equation, $PbS + PbSO_4 + O_2 = 2PbO + 2SO_2$.—E. S.

Manganese-Steel Castings, An Improved Method of Toughening. R. A. Hadfield, Sheffield. Eng. Pat. 11,833, May 30, 1896.

In toughening manganese-steel castings by water-quenching, there has always been a liability to the production, during the heating, of internal ruptures which might escape subsequent detection. This difficulty is overcome by heating the castings very slowly, placing them, after cleansing from sand, in a cold furnace, and then raising the temperature so slowly that thick castings ($1\frac{1}{2}$—2 ins. in thickness) shall have attained a temperature of 400° F. only after 1 hour, 800° F. after 2, and 1,200° F. after 3 hours' heating. The temperature may then be raised more rapidly to 1,700° F. —an orange heat—when they are removed quickly from the furnace and at once plunged into water.—W. G. M.

Iron and Steel, Carbonising; Impts. in. A. Wilson and F. Stubbs, both of Sheffield. Eng. Pat. 12,782, June 10, 1896.

The plate to be treated is covered with a layer of calcium (or other similar) carbide, when resting horizontally in a muffle; a second plate is then placed over this, and a current is passed through the carbide by connecting the plates with the poles of a dynamo, as soon as they have attained a red heat. For plates to be deeply carburised a layer of carbide 3 or 4 ins. thick should be used, and the voltage of the dynamo must be sufficient to overcome the resistance of the carbide when hot, but not necessarily when cold. The greater the quantity of the current, the quicker the result. The plant described allows for 100 ampères at 90 volts being applied to thick plates for 3 or 4 days.
—W. G. M.

Ores, Pyritic; Improved Process for Extracting Metals from, by Treatment with Chlorine, and for Recovering the Chlorine employed. O. Frölich, Steglitz, Berlin. Eng. Pat. 13,371, June 17, 1896.

Crushed sulphide ores, unroasted, are treated at the ordinary temperature with chlorine, either in the dry state or after being slightly moistened with suitable liquids. Soluble chlorides are then leached out, and the metals and chlorine are recovered by electrolysis, the halogen being used for the treatment of a fresh batch of ore. Thus zinc blende is treated with dry chlorine until no more of the gas is absorbed. The zinc chloride is then extracted with a moderate quantity of water containing a little hydrochloric acid or sodium chloride to hasten the solution of the zinc. When galena and silver are present, both lead and silver remain in the insoluble residue, when they may be extracted afterwards. In the case of auriferous antimony sulphide, the chlorine is first applied dry; the ore is then washed twice with water, dried, and re-chlorinated. The residue is next treated with hydrochloric acid to give antimonious chloride, and the washings are treated for the recovery of gold. Or the crushed ore is moistened and chlorinated, and is treated with dilute tartaric acid solution, which dissolves the antimony, but no gold. The residue is then moistened, re-chlorinated, and treated with water or dilute hydrochloric acid in order to obtain the gold in solution. Pyritic ore containing precious metals is moistened with hydrochloric acid, and is then chlorinated, and the product is treated with hydrochloric acid, giving a solution which contains almost all the gold and silver together with the common metals.—W. G. M.

Precious Metals, Impts. in or relating to the Precipitation of, from their Cyanide Solutions. M. B. Zerener, London. Eng. Pat. 12,255, June 4, 1896.

Alkali-metal amalgam, in the form of spray or of fine streams, is passed through the cyanide solutions containing the gold and silver in a vertical chamber so arranged that the mercury collecting at the bottom is carried up into an elevated tank, where it is again charged with alkali-metal and allowed to flow through the chamber. The cyanide liquor enters the chamber at the bottom from one tank, and, after the removal of gold and silver by the alkali-metal, overflows above into another.—W. G. M.

Alloys [Bronze], Impts. in. J. C. Bull, Erith. Eng. Pat. 12,328, June 5, 1896.

The purest copper obtainable (preferably the best brands of electro-deposited metal) is alloyed with from 2 to 5 per cent. of pure tin, with or without (at most) 0·1 per cent. of phosphorus or 0·2 per cent. of manganese added at the end for the purpose of deoxidation; and the fused mixture is run into ingots, which may then be worked at a red heat into bars, sheet, tubes, or wire.—W. G. M.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(A.)—ELECTRO-CHEMISTRY.

Standard Cell, The Cadmium. W. Jaeger and R. Wachsmuth. Ann. der Phys. und Chem. 1896, **59**, 575; Proc. Inst. Civil Eng., 1897, **127**, [1], 73—74.

The authors recommend a cell similar to the H-form of Latimer-Clark cell with platinum electrodes sealed into the glass. In one limb is placed cadmium amalgam (1 of Cd to 6 of Hg) covered with crystals of cadmium sulphate, and in the other, pure mercury beneath a layer of mercurous sulphate, the tube being filled with saturated solution of cadmium sulphate and sealed with paraffin and cork, with shellac over all. The electro-motive force of the cell at 20° C. is 1·019 volt, and its temperature co-efficient is about one-twentieth of that of the Latimer-Clark cell. The following formula is given:—

$$E_t = E_{20} - 3{\cdot}8 \times 10^{-5} \times (t - 20) - 0{\cdot}065 \times 10^{-5} \times (t - 20)^2,$$

where $E_{20} = 1{\cdot}019$ volt. This formula holds good between 5° C. and 25° C., but between 0° C. and 5° C., some cells were found to have an electro-motive force about 0·001 volt higher than the calculated value. The cadmium cell quickly recovers its normal electro-motive force after being heated or cooled through 16 or 17 degrees. The addition of 2 per cent. of zinc to the cadmium amalgam only raised the electro-motive force about 39×10^{-5} volt. An amalgam containing 1 per cent. more cadmium than usual had an electro-motive force about 0·001 volt too high. To ensure absence of free acid, the cadmium sulphate should be digested with cadmium hydroxide, filtered, warmed with mercurous sulphate and again filtered; and the mercurous sulphate should be washed with distilled water till it begins to turn yellow from the formation of basic sulphate.

The maximum variation from the normal electro-motive force of a number of cells which have been under observation since July 1894, was 8×10^{-5} volt.—A. S.

PATENTS.

Electrical Accumulators [Plates of compressed Spirals of Lead or Alloy], Impts. in. F. W. Greengrass, Epsom, and S. R. Docking, Croydon. Eng. Pat. 13,644, June 20, 1896.

The claims relate (1) to the manufacture of plates composed of entangled spirals of lead or lead alloy gently compressed and having the margins consolidated by melting or burning so as to form a solid edging all round the plate; (2) supporting the negative plates in the cell on lead feet burned upon their bottom corners and suspending the positive plates on tubular rods supported by lugs within the vessel; and (3) methods of sealing the cell by a plate.
—J. C. R.

Electrolytic Anodes, Impts. in or connected with the Arrangement and Attachments of. [Carbon.] P. E. Secrétan, Paris, France. Eng. Pat. 14,584, July 1, 1896.

The surface of the anodes is increased by providing them with ridges or wings, and to provide better connection between the carbon anodes and the metallic conductors, the irregular upper ends of the former are turned and provided with a thread which screws into a metallic socket preferably made of aluminium bronze, and this socket also screws on to the end of the red copper conducting rod. Discs of red copper are placed against the connecting bar at each end of the

part of the socket into which the bar is screwed, or the carbon anode may be held by a grip consisting of several rigid or elastic parts completely insulated from each other, or simply separated by a partial longitudinal slot. This grip is compressed into the anode by means of a nut acting directly on these parts or by the intermediary of elastic jaws, or of a tightening ring, absolute contact being obtained by metal washers interposed between the carbon and these parts.—G. H. R.

Electrolysis, Impts. in. G. Poore, London. Eng. Pat. 16,852, July 29, 1896.

In this process for producing electrotypes, electro-plating, decomposing metallic salts, and so forth, the cathode which is unsubmerged, is placed within a tank on a movable table, and the electrolyte is played on it in a jet from a suitably shaped nozzle, which may or may not be constructed of the metal to be deposited. The jet constitutes the only electrolytic connection between the anode and the unsubmerged cathode, and the electrolyte falling off, the latter passes through a tray containing the salt of which it is composed, so as to maintain its strength, and collects in the lower portion of the tank, from which it is pumped back to the jet. Both the anode and the cathode may be moved relatively to one another.—G. H. R.

[*Alkaline Percarbonates.*] *Chemical Products, Manufacture or Production of, by Electrolysis.* E. J. Constam and A. von Hausen, Zurich. Eng. Pat. 19,218, Aug. 31, 1896.

See under VII., *page* 537.

Electrical Furnaces [*Carbides, &c., under Vacuum or Pressure*], *Impts. in or relating to.* A. J. Boult, London. From O. Patin, Paris. Eng. Pat. 19,290, Sept. 1, 1896.

See under II., *page* 528.

Electric Furnaces [*Arc moved by Magnets*], *Impts. in or connected with.* F. J. Patten, New York, U.S.A. Eng. Pat. 4138, Feb. 16, 1897.

See under II., *page* 528.

Storage Battery [*Peroxide on Plate of Pumice*], *An Improved.* J. Entwisle, Manchester. From C. T. Barret, New York, U.S.A. Eng. Pat. 6818, March 16, 1897.

The improvements relate to the use of a cell having a porous plate of pumice stone, in and upon which the peroxide is applied, the said porous plate and peroxide being enclosed in a leaden box (faced interiorly with a layer of coke dust) which secures the parts in intimate contact, whilst providing for the admission and circulation of the acidulated solution." The perforated lead support is supported again with perforated insulating material, such as rubber, vulcanite, or their equivalent.—J. C. R.

Primary Electric Batteries or Generators [*Heating the Electrolyte*], *Impts. in.* S. Van B. Essick, Yonkers, U.S.A. Eng. Pat. 8131, March 30, 1897.

The positive and negative (zinc-copper or zinc-carbon) elements are in contact with a single battery liquid (20 parts of water and 1 each of sulphuric acid and copper sulphate) which is "subjected to heat sufficient for depolarisation." Means are provided for automatically keeping the liquid supply in the generator.—J. C. R.

(*B.*)—ELECTRO-METALLURGY.

PATENTS.

Electrolytic Depositions, An Improved Apparatus for Producing Galvanic or. [*Rotating Drum.*] W. P. Thompson. From The Elektro Galvanoplastische Anstalt, H. Feith and A. Floeck, Cologne, Germany. Eng. Pat. 11,730, May 29, 1896.

The rotating drum is lined with sheets of the metal to be precipitated, which are insulated from each other, and are so connected by means of a commutator to the dynamo that those plates which, for the time being are uppermost, serve as anodes, whilst the lower plates on which the work rests, form the cathodes.—G. H. R.

Electro-plating Apparatus, Impts. in. [*Electro-plating Workholder.*] R. J. Marks, Connecticut, U.S.A. Eng. Pat. 2470, Oct. 12, 1896.

The apparatus is designed to distribute the current evenly to all the work to be plated which it contains, and consists of a tray, formed of an insulated frame, containing a series of permeable, conductive floor sections insulated from one another, and the alternate sections at either end are connected to hangers, by means of which the workholder is suspended from a rod connected with the negative terminal of a dynamo. The workholder may be provided with a permeable cover.—G. H. R.

Metals, Impts. relating to the Coating or Covering of, and other Substances with, and other Materials by the aid of the Electric Arc. [*Ships' Hulls, &c.*] F. H. Snyder, Westcombe Park, England. Eng. Pat. 25,360A, Nov. 11, 1896.

The metallic or other structure is plastered over with a mixture made of a glutinous or binding material and a finely divided metal or alloy which it is desired to use as a coating, and this metal is then fused on by the arc. If a vitreous covering be required, the surface of the body is smeared with a plastic mixture of a suitable silicate, or of powdered glass, and a binding material, with a substance which is a good conductor of electricity, and the coating is fused on as before.—G. H. R.

Electric Furnaces [*for treating Ores*], *Impts. in.* E. F. Price, Niagara Falls, U.S.A. Eng. Pat. 27,301, Dec. 1, 1896.

The claims are for combinations of parts in furnaces which are described with drawings, and relate principally to the construction of the bottom or hearth [slab of carbon] of the furnace forming one electrode, and "a range of electrodes [carbon] substantially perpendicular to the bottom." Means of feeding the material and for the escape of the gases produced, are also the subjects of claims. The object of the invention is "to decompose the ore or other material, or melt the same under an electric current, and the material decomposed or melted is caused to pass or run off the hearth which is set at an inclination, and it is to be adjusted to greater or less inclination according to the material operated upon, so that the electric treatment can be substantially continuous."—J. C. R.

XII.—FATS, OILS, AND SOAP.

Residues from the Distillation of Starchy Substances Treatment of. [*Oil Extracting.*] Bull. Soc. d'Encouragement, **96**, 1897, 161—165.

The process elaborated by Donard and Boulet for extracting the oil from distillers' and brewers' grains, starch-makers' residues, &c. consists in drying the material under a pressure of 40 mm. of mercury and extracting by petroleum spirit (boiling point 60°—100° C.).

The drying apparatus consists of a horizontal revolving cylinder $2\frac{1}{4} \times 2\frac{1}{2}$ metres containing a number of tubes into which steam is introduced through one of the hollow axes, and exhausted by a double-acting pump *viâ* the other. The charge (2,500 kilos. of grains, containing 55 per cent. of moisture) is introduced through manholes and, the apparatus being rotated at a speed of three revolutions per minute during $3\frac{1}{2}$ hours, the moisture is reduced to 15 per cent. (equivalent to the evaporation of 1,000 kilos. of water) with a consumption of 3 cwt. of coal.

The extracting apparatus consists of a pair of vertical boilers $2\frac{1}{2} \times 2\frac{1}{2}$ metres and two vertical extractors, 120 × 310 cm. with a common condenser, one-half of the apparatus being worked at a time.

The extractor is charged with 2,200 kilos. of dried material and the boiler with 2,800 to 3,000 kilos. of petroleum spirit. Heat being then applied, the latter is volatilised

and ascends to the top of the extractor, where it condenses and passes down through the contents into the other boiler, the operation taking nine hours to complete. The residual spirit is driven out from the mass by the steam from water at the bottom of the boiler, and is passed through the condenser into the second boiler, the contents of the latter being then employed to extract the charge in the second extractor. Not more than 0·43 to 0·75 per cent. of spirit is lost.—C. S.

Glycerin, The Quantitative Estimation of. F. Bullnheimer. Forsch-Ber. ü. Lebensm. u. ihre Bez. 3, Hyg. &c. **4**, 12 and 31; Chem. Centr. Bl. 1897, 522 and 773.

See under XXIII., *page* 565.

Cholesterin in Fats, Detection of. A. Forster and R. Riechelmann. Zeits. für öffentl. Chem. **3**, 10; Analyst, 1897, **22**, 131.

See under XXIII., *page* 565.

Lard, Detection of Vegetable Fats in. Forster and R. Riechelmann. Zeits. für öffentl. Chem. 1897, 10; Pharm. Centralhalle, 1897, **38**, 151.

See under XXIII., *page* 565.

Fat in Oilcakes, Estimation of, by Carbon Tetrachloride. A. Bilteryst. Bull. Assoc. Belge des Chimistes, **10**, [11], 406.

See under XXIII., *page* 565.

PATENT.

Cleaning Agent [Saponaceous Mixture], Process of Manufacture of a. J. C. F. Müller, Hamburg. Eng. Pat. 1554, Jan. 20, 1897.

Five kilos. of oleic acid are saponified with $1\frac{1}{2}$ kilos. of caustic soda, $1\frac{1}{2}$ of potassium carbonate, and 3 or 4 litres of water. 25 kilos. of Iceland moss are made into a mucilage with the smallest possible amount of water, mixed with 5 kilos. of "linseed mucilage produced by boiling," and 125 kilos. of salicylic acid added as an antiseptic; the whole being finally incorporated into one. The mixture is specially suited for cleaning ceiling-paintings, work done in wax colours, and oil paint generally, as it is "neutral," and does not damage the objects; but in the case of articles which have to be freed from old oil colour, it may with advantage be made more strongly alkaline.—F. H. L.

XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

(*A.*)—PIGMENTS, PAINTS, &c.

Red Lead, Variations in the Composition of. D. Woodman. J. Amer. Chem. Soc. 1897, **19**, 339—341.

In the manufacture of red and orange lead, colour is primarily of first importance for pigment purposes, whilst the degree of oxidation, apart from its influence on colour, is not esteemed of much importance. For certain purposes, however, the degree of oxidation is important, and the author having occasion to examine a large number of samples from various sources, adopted the method of digestion with a saturated solution of normal lead acetate, by which the litharge in excess of that in combination as Pb_3O_4 (or $2PbO.PbO_2$) is dissolved out. In this way a series of results was obtained, showing a variation from 11 to 92 per cent. of actual red lead.

The interesting point was the large amount of monoxide in some samples which had escaped oxidation, and was separable by the acetate treatment, and which, from the ease with which it was separated, showed that it played no part in the chemical constitution of red lead, but was simply an admixture, and, as such, an indication of the completeness or otherwise of the oxidising process.

The separation of the litharge from the red lead was quite sharp, and prolonged digestion failed to effect any alteration in the weight of the actual red lead.

By the usual methods of analysis depending on the decomposition of the red lead into peroxide which remains insoluble, no distinction is made between the monoxide in combination as $2PbO.PbO_2$ and that present in excess and extracted by acetate solution, analyses usually being stated in such a form as:—Peroxide, PbO_2 = 26·4 per cent.; litharge, PbO = 69·7 per cent.; impurities, 3·5 per cent. Total = 99·6 per cent. However, it would be more satisfactory for commercial work to state it as, peroxide of lead (26·4 per cent.) equivalent to red lead = 75·6 per cent.; lead monoxide = 20·5 per cent.; impurities, 3·5 per cent. = 99·6 per cent.—O. H.

PATENTS.

Ferric Oxides [Pigment], Impts. in the Manufacture of. A. Crossley, Cwmavon, Monmouth, and H. A. Allport, Barnsley. Eng. Pat. 13,244, June 18, 1896.

Ferrous sulphate is mixed with about 1 per cent. of an oxidising agent, such as sodium or potassium nitrate or chlorate, in a wet state, and the mixture is charged into the calcining furnace as a stiff paste. It is stated that the sulphuric acid is liberated by this process at a lower temperature than otherwise, and that the colour of the oxide obtained, is improved. Reference is made to Eng. Pat. 2618, 1894 (this Journal, 1895, 157).—E. S.

Colours for Water-Colour Painting, A Process for Making. F. Beindorff, Hanover, Germany. Eng. Pat. 28,950, Dec. 17, 1896.

A process for making water colours, consisting in adding to the binding medium used, a mixture of "an aqueous solution of dichlorhydrin fat, acid-soda, and a preparation of pigs' gall."—R. B. P.

Pigments, Printing Inks, or Paint Mixtures; Impts. in and in the Manufacture of. E. O. S. and H. R. Gentele, Stockholm. Eng. Pat. 7399, March 22, 1897.

"Lakes" are produced by staining wood-pulp, cellulose, &c., either before or after grinding, with the desired dye; the result being that a given amount of colouring matter can be distributed over a larger quantity of base than when alumina and similar substances are employed. The manufacture of printers' inks and paints from pigments made in this manner is also claimed.—F. H. L.

(*B.*)—RESINS, VARNISHES.

Gum Arabic and Frankincense, some Indian Substitutes for; Report on the Examination of. Imp. Inst. J. Research Dept. **3**, [28], 123.

See under XVI., *page* 548.

(*C.*)—INDIA-RUBBER, &c.

Caoutchouc, Valuation of Raw and Manufactured. R. Henriques. Zeits. für öffentl. Chem. **3**, 37; Analyst, 1897, **22**, 134.

See under XXIII., *page* 566.

XIV.—TANNING, LEATHER, GLUE, SIZE.

PATENTS.

Glue Compounds, Impts. in, and in Applying the same to Barrels and the like. J. Howden, Glasgow. From Julius Tyciak, Olszancia, Galicia. Eng. Pat. 3073, Feb 5, 1897.

The improved glue is made by combining ordinary glue, 640 parts; Pecher's natural gum, 210 parts; glycerin, 85 parts; ferrous sulphate, 57 parts; tannic acid, 5 parts; and soda crystals, 3 parts. It is used for coating the interior of barrels for containing light hydrocarbons, rendering them fluid and vapour tight.

"The hot compound is put into a barrel, the bung closed, and the barrel rolled about." The air pressure forces the compound into the pores of the wood.—J. T. W.

Tanning, Arrangement for Performing a continuous Mode of. H. Schmidt and J. Landini, Hamburg. Eng. Pat. 13,686, Feb. 12, 1897.

The tanning is conducted in a series of four drums connected with each other by pipes in such a way that the extract solutions used can be forced from one drum to the other by means of compressed air; the liquor used for finishing the tanning being the strongest, passing back to the next drum, and so diminishing in strength until it can be used on the freshly introduced hides. Heaters are arranged for raising the temperature of the liquor before its entry into the drums. Water can also be supplied whilst the drums are rotating. Illustrations show the arrangement of pipes, &c.—J. T. W.

Hides or Skins for Transport or Manufacturing Purposes, A Process for Preserving. J. W. Scott, Albionville, Queensland. Eng. Pat. 8168, March 30, 1897.

The hides, after washing free from blood, are treated with a mixture of molasses and water instead of salting them. The liquid ferments. With a mixture of 10 per cent. strength, the hides are cured in about seven days; they are then hung up to dry, and may afterwards be folded up and shipped.

None of the insects that attack salted hides touch hides treated by this process. The action is due to a "lactous or butyrous" fermentation.—J. T. W.

XV.—MANURES, Etc.

Manure, New versus Old. Agricult. J. 10, 315; Pharm. J. 1897, [1405], 472.

Experiments made in Canada last year showed that 8,000 lb. of manure placed under shelter and weighed once a month was reduced to 2,600 lb. between March and December. The manure was at its best at the end of four months, weighing 3,480 lb. The experiments of eight years appear to show that the action as between fresh and rotted manure is equal, ton for ton. In England, where the soil is stiff, fresh manure would, perhaps, be more beneficial, as the soil is lightened by its use; but in Canada, taking into consideration the undigested seeds in fresh manure, the small rainfall, and the outlay in the daily carting of the manure, it would certainly not be economical.—A. S.

Sulphate of Ammonia from Blast Furnaces, Agricultural Value of. F. J. R. Carulla. J. Iron and Steel Inst., May 1897.

The object of the paper is to call attention to the manurial value of sulphate of ammonia, which the author considers is neglected, not on account of any inherent deficiency, but owing to the persistency with which nitrate of soda is pushed to the front. Results of Wagner's experiments with grass, rye, and buckwheat are quoted, showing the equal manurial value of $\frac{3}{4}$ cwt. of sulphate of ammonia and 1 cwt. of nitrate of soda. (See this Journal, 1897, 110, and 1896, 863.)—N. H. J. M.

PATENTS.

Superphosphates and the like, Impts. in the Manufacture and Storing of. F. M. Mooney and A. Dickson, Dublin. Eng. Pat. 9789, May 8, 1896.

Phosphatic materials are mixed with acid to form "superphosphate" at such an elevation as to admit of running the compost while it is yet fluid into large stores, used as "dens," where it may be kept for a considerable time, till the reaction is completed and the heat fully utilised in evaporating moisture. Instead of elevating the mixer, it may be made portable; or horizontal or slightly inclined conveyors may be employed to carry the material from the mixer to the stores.—E. S.

Phosphates and other Substances, Impts. in Dissolving and Drying. J. Milne, Inverurie, N.B. Eng. Pat. 12,765, June 10, 1896.

Phosphates mixed with acid are discharged from the mixer through a hopper on to the uppermost of a series of double rows of plates, fixed to traction chains, arranged as an endless apron and moved by slow driving gear, within a closed chamber through which hot air or gases of combustion from a furnace are caused to pass, cold air being also admitted so as to avoid overheating the manure. The material, as it is discharged from the upper row of plates, is carried successively upon a series of other superposed rows, until it falls into a screw conveyor or other means of discharge. It is stated that the mixture of phosphate and acid charged into the apparatus, is mechanically and chemically changed during its passage through the same, from a semi-liquid material to a dry powder; or from tribasic phosphate of lime and sulphuric acid to monobasic phosphate of lime and calcium sulphate.—E. S.

Moss-Litter Manure, Treatment of, for Obtaining Products therefrom. E. van de Griendt, Rotterdam, Holland. Eng. Pat. 17,883, Aug. 12, 1896.

Moss litter that has been used for stable purposes is treated with steam in a boiler while being agitated. The steam passing from the vessel "is allowed to percolate through sulphuric acid" to obtain ammonium sulphate. The liquid portion in the boiler is run off and evaporated to obtain "a mixture of nitrates, phosphoric acid, and alkali" for use as a manure. The residual mass is dried for application as a fuel.—E. S.

XVI.—SUGAR, STARCH, GUM, Etc.

Starch Manufacture, Organisms in. Dr. Saare. Zeits. für Spiritusind. 1897, Supplement II., 4—7.

In the Halle or acid process for making wheat starch, organisms set up fermentation of the steeped and bruised wheat and thus dissolve the albuminoid integument of the starch granules. This process is, however, being gradually abandoned in favour of others, in which the gluten is recovered in the residuals. In the production of cereal starches, sulphurous acid or alkali is used, and suppresses organisms; though in drying, mould or fermentation may develop and cause discolouring of the product. Organisms abound in the manufacture of potato starch, many being derived from diseased potatoes. *Phytophthora infestans*, the fungus of potato disease, spreads through the tuber, and makes the starch granules either dissolve or assume a more or less acicular form, so that they settle badly; or particles from the rind pass the sieves and discolour the starch. Scab and dry rot are due to organisms, and cause specks in the starch. Wet rot is a more formidable disease, and gives rise to bacteria which cause putrefactive fermentation of the liquor and loss of starch. The starch will also be affected if the water used in its production contain algæ, diatoms, decaying vegetable matter, or certain bacteria, especially those common in chalybeate waters.

If the temperature of the factory do not exceed 10° C., bacteria do not occur in large numbers in the liquors, but if the sieves be not frequently cleansed, a mould forms on and chokes them, and a loss of starch results. With proper cleansing and sound potatoes, few bacteria or germs will be found in the starch milk at that temperature; but if, on the other hand, the washing take place at 20°—26° C., bacteria will multiply and the liquors will become acid. The starch produced therefrom will retain traces of acid and a rank acetous smell, whilst its colour will be injured through albuminoid particles and fungoid growths which will pervade it. It was found by experiment that an addition of 12 litres of sulphurous acid ($2\frac{1}{4}$° B.) to a tank of 4·5 cb. m. capacity almost wholly suppressed the bacterial fermentation at these higher temperatures and diminished the amount of scum formed. It did not, however, entirely destroy the sporules from diseased potatoes. When fermentation has been permitted, the dried scum is brown, but when it has been suppressed by sulphurous acid the scum is yellow. Fermentation prevents rapid and complete settling of the starch. The manufacture should be pursued as much as possible in the winter; at warmer times, or when diseased potatoes are treated, an addition of sulphurous acid increasing with the temperature should be made. Rapid drying at a comparatively high temperature is less favourable to the spread

of bacteria than prolonged drying. The complete aeration by centrifugal drying machines greatly checks the bacteria. The presence of bacteria is of no moment in many of the applications of starch, but it is important in the case of the starch used in the production of pressed yeast, as the bacteria may set up a putrefactive fermentation which is very injurious.

The presence of bacteria in the waste pulp is beneficial, as, after removal of water, lactic acid fermentation sets up, and improves the value of the waste as fodder for cattle. The milk of the cattle fed thereon is improved both in quality and quantity. The effluent liquors, if applied to land, are also benefited by the presence of bacteria, which convert the nitrogenous constituents into forms from which the nitrogen can be assimilated by plants.—J. A. B.

Starch, Transformation [into Soluble Starch and Dextrin] by Sulphur Dioxide. A. Bergé. Bull. Assoc. Belge des Chimistes, **10**, [12], 444—447.

Below 80° C. the action of sulphur dioxide, whether liquid or gaseous, on starch is almost *nil*, but between that temperature and 115° C., soluble starch is formed. On the other hand, above the last-named temperature, conversion (in the absence of moisture) into a very pure dextrin occurs, though beyond 140° C. the product turns yellow, and the author has prepared dextrin—containing not more than 0·95 per cent. of glucose—on an industrial scale by heating dry potato starch with sulphur dioxide at 140° C. for seven hours in a revolving cylinder.

With sulphurous acid in solution the action is extremely feeble below 45° C., but at 100° C. saccharification to glucose begins, and is complete between 135° and 140° C. The most favourable conditions for this conversion are: Temperature, 135° to 140° C.; pressure, 6 atmospheres; proportions of the mixture, 25 per cent. of starch and 75 per cent. of water containing 3 to 6 per cent. of sulphurous acid. The reaction is complete in about an hour.

In the case of potato starch, the granulose is rapidly converted, but the starch cellulose is attacked much more slowly. The presence of extraneous substances retards the operation.—C. S.

Gums for Thickening. C. Gassmann. Färber Zeit. **8**, [9], 135—136, and [10], 153—155. (See also this Journal, 1897, 237.)

The author describes certain insoluble gums derived from Africa, which can be rendered soluble by boiling with water under pressure, and may then be used as thickeners. These solutions are bleached and evaporated to dryness by well-known means, and the gums thus obtained are put on the market in broken flakes about 0·5 to 1·0 cm. thick, as *gommes crystallisées* or *gommes solidifiées*,—which are all soluble.—I. S.

Gum Arabic and Frankincense, Some Indian Substitutes for; Report on the Examination of. Imp. Inst. J., Research Dept. **3**, [28], 123—124. (See also this Journal, 1892, 403.)

Three gums and one gum-resin, received from the Conservator of Forests, School Circle, N.W. Provinces and Oudh, were examined.

I. Simla Gum (*Bauhinia retusa*).—The sample consisted of large rounded tears, &c., which broke easily with a vitreous fracture and were brown in colour. The fragments were translucent, and varied in colour from yellow to brown. The gum was odourless, and bland and mucilaginous in taste. It contained 13·5 per cent. of moisture and 3·18 per cent. (on the dried gum) of ash. A 10 per cent. solution, made for determining its viscosity, yielded a mucilage so thick that it could not be manipulated. A 5 per cent. solution was therefore employed. Even with this dilution, a considerable quantity of the gum remained insoluble, and was removed by straining through muslin. The solution was not precipitated by neutral lead acetate, but with basic acetate it formed an opaque white jelly. It was also immediately gelatinised by the addition of ferric chloride. The gum contained no starch and only a trace of dextrin or sugar.

II. Jingan Gum (*Odina Wodier*).—The gum had very little taste, a slight odour, and varied from white to yellowish-white in colour. It contained 12·3 per cent. of moisture and 8·73 per cent. of ash (on the dried gum). It was completely soluble in twice its weight of water, forming a rather thin mucilage of light brown colour, which possessed considerable adhesive power. The solution behaved like that of the *Bauhinia retusa* gum towards neutral and basic lead acetate and ferric chloride. It contained no starch, but had a marked reducing action on Fehling's solution, indicating the presence of dextrin or sugar.

III. Pial Gum (*Buchanania latifolia*).—This consisted of large irregular masses, tears, and small fragments. The tears were fissured and opaque; the fragments were clear and glassy, as also were the larger masses. The latter contained considerable quantities of impurity, pieces of bark, &c., and the whole sample was contaminated with vegetable *débris*. It had little taste or odour, and the fragments varied in colour from yellow to reddish-brown. It contained 14·2 per cent. of moisture and 6·27 per cent. (on the dried gum) of ash. The gum was not entirely soluble when mixed with twice its weight of water, a portion (about 10 per cent.) swelling up and forming a gelatinous mass which remained undissolved. The mucilage obtained by treating the gum with twice its weight of water was thick, dark in colour, and possessed strong adhesive properties. The solution gave the usual reactions of gum-acacia, behaving with neutral and basic lead acetate and ferric chloride exactly as the two preceding samples. It contained no starch and only a trace of dextrin or sugar.

Comparative Viscosities.—The viscosity of the solutions of these gums was compared with that of a solution of the best gum arabic, by noting the time taken by 50 c.c. of the solution to flow from a burette fitted with a fine nozzle.

——	Strength.	Burette Time in Seconds.
	Per Cent.	
Gum arabic	10	78
Odina Wodier	10	58
Buchanania latifolia	10	184
Bauhinia retusa	5	200

IV. Salhar Gum (*Boswellia serrata*).—The specimen of this gum-resin, which is closely related to ordinary frankincense, consisted of small rounded tears and larger irregular masses. The odour and taste were strongly terebenthinous. The gum-resin readily burned, and on analysis gave the following figures:—Resin, 53·2 per cent.; gum, 36·45 per cent.: volatile oils, 7·0 per cent.; ash, 1·55 per cent. The gum was completely soluble in water, and the solution gave the ordinary reactions of gum-arabic. The greater part of the volatile oil boiled at 157° C., after which the temperature rose to over 170° C.

Analyses of true frankincense have been published by Braconnot (Ann. de Chim. [2], 68, 60), who found—resin, 56 per cent.; soluble gum, 30 per cent.; insoluble gum, 6 per cent.; volatile oil, 8 per cent.: and by Kurbatow (Zeits. für Chem. [2], 7, 201), who found—resin, 72 per cent.; gum, 21 per cent.; volatile oil, 7 per cent.—A. S.

Residues from the Distillation of Starch Substances, Treatment of. [*Oil extracting*] Bull. Soc. d'Encouragement, **96**, 1897, 161.

See under XII., *page* 545.

Sugar Cane, A New. Bd. of Trade J., June 1897, 711.

See under Trade Rep., *page* 573.

XVII.—BREWING, WINES, SPIRITS, Etc.

Alcoholic Fermentation without Yeast Cells. (*II.*) E. Buchner. Ber. 1897, **30**, 1110—1113.

In continuation of his former experiments (this Journal, 1897, **16**, 156), the author now gives some new results to disprove the view that the fermentative effects described in

his former paper might be attributed to the action of minute particles of living protoplasm. He finds that the stability of the yeast extract is not affected by the presence or absence of air, and the destruction of its activity is probably due to the presence of peptic enzymes. The loss of activity is thus accompanied by a diminution of the coagulable albumin. This hypothesis also explains the preservative action of a concentrated solution of cane-sugar, which it is thought would destroy the activity of the peptic enzyme. The addition of antiseptics, such as chloroform, benzene, and sodium arsenite, does not destroy the fermentative activity of the yeast extract, and as these substances hinder the growth of living yeast cells, they would be expected to act much more injuriously upon suspended particles of plasma deprived of their protective membrane. It is further found that the yeast extract may be evaporated to dryness without loss of activity; this may be effected in about 6 hours by exposing it in thin layers to a temperature of 30°—35° and under a pressure of a few millimetres. The dry product may be redissolved in water, and the solution, after filtration, gives a bright filtrate, which, when mixed with a 75 per cent. solution of cane sugar, will produce a distinct fermentation in 6—10 hours, and the evolution of gas will continue for days. The dried residue retains its activity certainly for 20 days, and probably much longer. The author has also succeeded, after several unsuccessful attempts, in isolating an active substance by precipitation with alcohol. In further proof of the existence of zymase, it is stated that when well washed and dried (at 37°) yeast was heated for 6 hours at 100° so as to kill all the yeast cells, the product still gave rise to a vigorous fermentation when added to sterilised cane-sugar solution. When, however, the yeast was heated for 1 hour to 140°—145° the zymase was destroyed and its fermentative power was lost.

—A. K. M.

Diastase, Action of Light on, and its Biological Significance. J. R. Green. Proc. Royal Soc. 1897, **61**, 25—28.

According to the observations of Brown and Morris (J. Chem. Soc. 1893, **63**, 604), the quantity of diastase in foliage leaves undergoes considerable variations during the 24 hours of the day, being greatest in the early morning and least in the evening, particularly after several hours' sunshine. During the past three years the author has conducted a series of experiments to ascertain whether the diminution in quantity is due to a destructive influence of the light upon the enzyme, similar to that observed with micro-organisms and other low forms of vegetation.

The investigation has been carried out by exposing various solutions containing diastase to the action of light for several hours, either that of the total spectrum or selected portions of it, and after each such exposure testing the hydrolysing power of the solutions upon a weak solution of soluble starch, or upon a 1 per cent. starch paste. Controls were carefully kept during each experiment, so that any difference shown in diastatic power could be only attributed to the illumination. The diastatic solutions consisted of malt extract, solution of diastase precipitated from such extract by alcohol, diluted human saliva freed from mucin, and extracts of foliage leaves. The solutions were preserved by the addition of 0·2 per cent. of potassium cyanide. The sources of illumination were bright sunshine, diffused daylight, or the light from a naked electric arc. The diastatic power has been ascertained by titrating the results of the digestions with Fehling's solution, burning the filters with the precipitates, and weighing the resulting cupric oxide. When all the rays of the spectrum were under investigation, the solutions of diastase were exposed to the light, either in quartz-fronted cells, or mixed with films of agar-agar, the former method giving the most satisfactory results.

From many experiments it was found that exposure to the whole of the spectrum for several hours caused the destruction of from 20 to 60 per cent. of the diastase. When the ultra-violet rays were eliminated by the use of glass vessels there was at first a considerable increase in the diastase, but this was succeeded, on an exposure lasting for several days, by a gradual and almost complete destruction of the enzyme.

By the use of a series of screens, arranged according to the directions of Landholt (Ber. 1894, 2892), the spectrum was divided into red, orange, green, blue, and violet. The infra-red, red, orange, and blue regions gave increases of diastatic power of 10·8, 53·5, 4·75, and 20·8 per cent. respectively; the green gave a diminution of 15·7 per cent. The violet also gave a marked diminution. The effects of the illumination were found to be progressive, the increase or diminution of the diastase continuing after the solutions were removed from the access of the several rays. The deleterious rays were found to be absorbed by the solution, this being partly effected by the diastase itself, whether boiled or unboiled, and partly by the proteids present in the extracts. After removal of the latter by boiling and filtration, the solution was still opaque to the rays. The screening influence of proteids was examined separately, by adding small quantities of egg albumin to the extracts. The degree of protection was, however, only roughly proportional to the amount of albumin present. The colouring matter of the barley grain was found to act as a screen to the deleterious rays.

The living leaf was examined by the same method as the extracts, and the diastase in it was found to undergo a similar destruction under the influence of the light. It was found impossible to investigate the possible protective influence of chlorophyll in the leaf, as all the solvents of the latter were found to be opaque to the ultra-violet rays. They confirmed the view that the violet rays, and possibly the green, have a destructive effect upon the enzyme.

The author concludes from his experiments that there exists in the leaf, and in the various extracts examined, a certain amount of zymogen which is converted by the infra-red and the red, orange, and blue rays into active diastase. This conclusion is supported by experiments upon the effect of keeping the solutions for several days at a temperature of 38° C. The violet and ultra-violet rays, on the other hand, cause a destruction of the diastase, or at least such a change in the configuration of its molecule that it is unable to effect the hydrolysis of starch. The following three conclusions have also resulted from this investigation:—1. That the enzyme is not located in the chlorophyll grain, but in the protoplasm of the cell. 2. That the suggestion of Pick (Bot. Central. **16**, 9—12) and Johow (Pringsheims Jahrb. **15**, 299), that the red colouring of certain leaves is a material help to the translocation of starch in them is probably sound, as such colouring matters screen off the rays which destroy the diastase. 3. That there exists in plants a power of absorbing and utilising the radiant energy of light, sometimes to a considerable extent, without the presence of a chlorophyll apparatus. The last conclusion is of far-reaching importance, supplementing other observations already published by Engelmann (Bot. Zeit. 1888), Winogradski (Ann. de l'Institut Pasteur, 1890–91), Speschnew, and other observers, none of whom, however, have indicated such utilisation of the rays of the visible spectrum.—J. L. B.

Barley [Malting], Influence of Drying, on Germination. Behrend. Zeits. f. d. ges. Brauwesen, **20**, [16], 218.

Samples of wet barley were dried by storage for five weeks, and the percentage of moisture was then found to have receded from 20·88 (maximum) to 12·13 per cent. Concurrently the percentage of germinating corns rose from 67·8 to 91·2 per cent.—counted on the third day—and from 79·7 to 94·8 per cent. on the tenth day after steeping, —an increase in germinating power of the dried over the undried grain of 23·4 and 15·1 per cent. respectively.

The inference drawn from these results is that in the case of wet barley, growers can improve the quality for malting purposes, by storing the grain on airy floors.—C. S.

Beer, Mexican. A. Laug. Zeits. f. d. ges. Brauwesen, **20**, [13], 179.

A sample of beer (from the Oaxaca brewery) prepared from Bavarian malt (from Moravian barley) and Spalt and Saaz hops gave on analysis: saccharometer indication, 3·49° Balling; alcohol, 4·01; extract, 5·25; maltose,

1·114 : total acid (lactic acid), 0·124 : protein (N × 6·25), 0·5 ; ash, 0·203 ; P_2O_5, 0·089 ; glycerol, 0·151 ; and CO_2, 0·235 per cent. The original gravity was calculated as 12·98° Balling, the apparent attenuation being 73·11, and the actual 59·55.

The beer was pale in colour and apparently modelled on the Bavarian lager type, but of much lower original gravity. A considerable deposit and characteristic flavour, due to pasteurisation, were apparent.—C. S.

Analysis of English, Munich, and Vienna Beers. Dömens. Wochenschr. f. Brau. 1896, **13**, 1343.

The author has analysed some English and foreign beers, the results of which are shown in the following tables :—

—	Bass and Co. Special Stout.	Bass and Co. Pale Ale.	Guiness and Co. Extra Stout.	Bass and Co. Strong Ale.	Light Porter Ale.	Allsopp and Co. Luncheon Stout.	Allsopp and Co. Light Dinner Ale.	Tottenham Lager Beer.	Tottenham Pilsener Beer.	Tottenham Munich Beer.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Apparent extract	2·55	1·82	4·50	8·77	1·25	2·97	1·97	2·42	1·27	5·12
Real extract	5·27	4·32	7·03	11·8	2·82	5·37	4·05	4·45	3·02	7·32
Extract of original wort	16·80	14·96	17·61	24·2	9·65	15·58	13·01	13·16	11·55	16·64
Apparent attenuation	84·80	87·8	74·8	63·8	87·0	80·93	84·8	81·61	89·0	69·23
Real attenuation	68·6	71·1	60·1	51·2	70·8	65·53	68·8	66·18	72·3	56·01
Alcohol	6·11	5·58	5·64	6·85	3·49	5·35	4·65	4·52	4·30	4·92
Sugar	0·8	0·76	1·03	3·81	0·6	1·53	0·70	1·08	0·71	1·84
Fixed acidity (as lactic acid)	0·432	0·234	0·477	0·288	0·09	0·504	0·198	0·155	0·126	0·216
Volatile acidity (as acetic acid)	0·034	..	0·082	0·017	0·031	0·3	..	..	..	..
Depth of colour (expressed as c.c. of $\frac{N}{10}$ iodine solution).	..	..	..	2·6	1·0	24·0	0·9	1·6	0·46	3·8

—	Munich Lager Beers.						Vienna March Beer.	Vienna Lager Beer.
	Dark.	Dark.	Dark.	Dark.	Dark.	Light.		
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Apparent extract	5·55	5·57	5·59	6·30	5·50	3·60	4·17	3·77
Real extract	7·22	7·23	7·61	7·80	7·27	5·50	6·29	5·52
Extract of original wort	14·35	14·33	14·71	14·21	14·83	13·60	15·33	13·0
Apparent attenuation	61·3	61·1	59·5	55·7	62·9	73·5	72·8	71·0
Real attenuation	49·7	49·5	48·3	45·1	51·0	59·6	59·0	57·5
Alcohol	3·7	6·69	3·7	3·33	3·93	4·20	4·75	3·89
Sugar	2·52	..	2·87	2·92	2·38	1·30	1·45	1·25
Acidity (as lactic acid)	..	0·16	0·15	0·16	0·20	0·17	0·15	0·15

—J. L. B.

Citric Acid, Preparation of, by Fermentation. Zeits. f. Spiritusind, 1897, **20**, 149.

See under VII., *page* 535.

Saccharification of the Mash, Testing the. J. Fuchs. Zeits. f. des Brauwesen, **20**, [15], 203.

See under XXIII., *page* 563.

Glycerin, Estimation of Small Quantities of. M. Nicloux. Bull. Soc. Chim. 1897, **17**, 455.

See under XXIII., *page* 565.

Glycerin, The Behaviour of Metallic Oxides towards; A Contribution to the Quantitative Estimation of Glycerin. F. Bullerheimer. Forsch.-Ber. ü. Lebensm. u. ihre Bez. z. Hyg., etc. **4**, 12 and 31 ; Chem. Cent. Bl. 1897, 522 and 773.

See under XXIII., *page* 565.

Phosphoric Acid in Wine, Simple Method for the Estimation of; especially in Sweet Wine. W. Thörner and R. Uster. Forsch.-Ber. ü. Lebensm. u. ihre Bez. z. Hyg., etc. **4**, 55 ; Chem. Centr. Bl. 1897, 825.

See under XXIII., *page* 567.

Colouring of Wines; Distinguishing Coal Tar Colours from Caramel. A. d'Aguiar and W. da Silva. Bull. Soc. Chim. 1897, **17**, 443.

See under XXIII., *page* 563.

Residues from the Distillation of Starchy Substances, Treatment of. [*Oil Extracting.*] Bull. Soc. d'Encouragement, **96**, 1897, 161.

See under XII., *page* 545.

PATENTS.

Grape Juice, Wines, Spirits, and the like ; Impts. in Means for Treating and Improving. P. W. May, Child's Hill, and B. E. R. Newlands, London. Eng. Pat. 16,747, July 28, 1896.

The substance to be treated is heated in a closed vessel to a temperature sufficient to effect sterilisation. The temperature employed may vary, but 71° is generally sufficient. The heat is applied gradually and maintained for a sufficient time, usually about four hours, after which time the product is allowed to cool gradually, the vessel being kept closed during the whole operation.—A. K. M.

Crude Alcohol and other Alcoholic Mixtures, Process and Apparatus for Purifying. C. Killing, Düsseldorf. Eng. Pat. 28,713, Dec. 15, 1896.

The apparatus described, is an upright vessel of cylindrical or other shape, containing a steam coil, at the lower end a stirrer, and is also provided with two adjustable openings at or near the top. For the purification of crude alcohol, the mixture is heated to about 37° to 50°, when mixed vapours will be given off ; the alcohol vapour will, however, gradually condense as it reaches the higher portions of the apparatus, and practically pure aldehyde can be led away through one of the openings at the top, air being admitted by the other.—A. K. M.

XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

(*A.*)—FOODS.

Amylaceous Substances, The Economic Treatment of the By-Products of Distillation of. Report by M. de Luynes, Bull. Soc. d'Encouragement, 1897, 166.

MM. Donard and Boulet, at their works situated at Bapeaume-les-Rouen have treated during the last seven

years about 13,000,000 kilos. of maize cake from which 3,500,000 kilos. of oil were extracted. Incidentally they have also produced in one year 20,000 kilos. of wheat oil, 7,000 kilos. of rye oil, and more than 200,000 kilos. of rice oil. They avoid the resinification of the oil, and obtain complete extraction from the cake, by—

1. Drying at a low temperature under a pressure of 10 mm. in a rotatory apparatus.
2. Extracting the oil with boiling petroleum spirit.

—V. C.

Gingers, Commercial, and Essence of Ginger. W. S. Glass. Pharm. J. 1897, **54**, 245.

Of the three commercial varieties of ginger, Jamaica, Cochin, and African, the last is found to be richest in oleo resin, yielding 6·3 per cent. compared with 5·0 per cent. from the Jamaica variety, and 4·6 from Cochin ginger. African ginger is therefore recommended for the preparation of the soluble essence of ginger, which may be obtained by adding to 8 fluid parts of ordinary essence, 3 parts of powdered pumice, shaking occasionally for 12 hours, then adding gradually 24 parts of distilled water, shaking after each addition, allowing to stand for six hours and filtering.

—J. O. B.

Caffeïne, Notes on. G. L. Spencer. J. Amer. Chem. Soc. 1897, **19**, 279.

See under XXIII., *page* 568.

Caffeïne in Tea, Estimation of. C. C. Keller. Ber. Pharm. Ges. 1897, **7**, 105; Chem. Zeit. Rep. 1897, **21**, 102.

See under XXIII., *page* 568.

Caffeïne in Coffee and Tea, Estimation of. A. Hilger and A. Juckenack. Forsch. Ber. ü. Lebensm. u. ihre Bez. z. Hyg., &c., **4**, 49; Chem. Centr. Bl. 1897, 775.

See under XXIII., *page* 567.

Chocolate, Detection of Earth Nuts [*Arachis Hypogæa*] *in.* A. Bilteryst. Bull. Assoc. Belge des Chimistes, **10**, [12], 447.

See under XXIII., *page* 567.

Butter Analysis, Contribution to. L. Drumel. Bull. Assoc. Belge des Chimistes, **10**, [11], 411.

See under XXIII., *page* 568.

Fats, Note on Weighing out. C. E. Cassal. Analyst, 1897, **22**, 113.

See under XXIII., *page* 565.

PATENTS.

Preservation of Foods and other Perishable Goods, Impts. in or relating to. H. Higgins, Cambridge. Eng. Pat. 8232, April 18, 1896.

The object of the invention is to hermetically seal vessels containing perishable substances, which have been sterilised, without admitting unsterilised air. The vessel is fitted with a cork, which is pushed down to below the upper edge of the neck. A funnel, containing cotton wool, with a tube of fine calibre which is drawn out to a solid point, but which is perforated just above the point, is now pushed through the cork. The cork is covered with melted paraffin wax or any suitable material which melts at the temperature employed in sterilising. When the vessel has cooled, a hot fluid substance is poured into the space above the cork, so that, as the funnel tube is withdrawn, the liquid flows into the hole, thereby at once sealing the opening without unsterilised air having had access.

A suitable cement can be made of india-rubber or gutta-percha (2 parts), paraffin wax (6 parts), resin (1 part), and magnesium silicate (2 parts). Fusible metal may also be employed.

Other modifications of the sealing process are described.

—N. H. J. M.

Smoking Salmon and Similar Fish, An Improved Process for, and Means therefor. C. Waldemann, Coslin, Germany. Eng. Pat. 12,904, June 11, 1896.

The fish is prepared and cut in the usual manner, and the exposed portions evenly covered with animal bladder, vellum, or other suitable material. The pieces of fish are then placed on stands, formed of triangular plates and an overcoating of wire gauze, and exposed to the smoke of a wood fire. The advantages claimed for the process are the prevention of flaws and cracks in the inner side of the fish, which are apt to become mildewed, and the shortening of the smoking process, owing to the fish drying more rapidly when kept in a horizontal position than when hung in the ordinary manner.—N. H. J. M.

Drying Fruit and the like, by Air and Furnace Gases, Impts. in Apparatus for. O. Schmeisser, Landsberg-on-the-Warthe, Germany. Eng. Pat. 28,414, Dec. 11, 1896.

A process of fruit drying is described such that, in the case of stone fruit from which the stones have not been separated, the heat is so applied that whilst the fruit itself is dried, the stones also become dry. The stones have, as a rule, to be heated below 100° C., whilst the flesh of the fruit may, at first, be exposed to a higher temperature. The material is fed in a long continuous stream in the same direction as the hot gases from a fuel furnace. The drying gases pass into different compartments, to prevent their being mixed, so arranged that the fresh fruit receives the greatest heat, and that the heat becomes less as the fruit approaches dryness. In order that the fruit may be uniformly heated during its passage, it is mixed by means of a rotating perforated drum. Small fruit, which dries more quickly than larger fruit, and would, therefore, be liable to become overheated, passes through holes arranged at convenient distances in the drum, into suitable receptacles.—N. H. J. M.

(*B.*)—SANITATION; WATER PURIFICATION.

Aluminium Bronze Colours, Danger of Explosion and Fire in the Manufacture and Storage of. R. Kayser. Zeits. öffentl. Chem. **3**, 93—94. Chem. Centr.-Bl. 1897, [1], 833.

The author observed repeatedly that the aluminium bronze colours became oxidised in the settling vessels, to aluminium hydrate, with evolution of hydrogen. This decomposition by water only took place if the temperature of the rooms in which were the settling vessels rose above 25° C. The evolution of hydrogen begins slowly, but in consequence of the heat of the reaction becomes very vigorous. As there may be present in a settling vessel 6 kilos. or more of the bronze colour, capable of evolving at least 7 cb. m. of hydrogen, there is the possibility of a dangerous explosion, by the formation of considerable quantities of inflammable gas in the rooms. By admission of moisture and heat to packed aluminium bronze, evolution of hydrogen may take place, and if the packing material be inflammable, may lead to fire. Spontaneous combustion of bronze colours was also caused by strong blows or friction, due probably to small quantities of adhering fat or benzene. These phenomena were not observed with bronze colours made from copper-zinc and copper-tin alloys.—A. S.

Waste Water from Print Works, The Purification of. R. J. Flintoff. J. Soc. Dyers and Colourists, 1897, 99—104.

This paper contains a criticism of the circular of the Mersey and Irwell Joint Committee, a general discussion of the purification of a print work's effluent, and a description of a plant and process for the purpose.

The waste waters first enter a deep "sludge tank," where a partial subsidence of suspended matter, as well as of any precipitates formed by the mixing of different waste liquors, takes place. Here also fatty matters separate and collect on the surface as a thick scum, which is prevented passing into the next tank, as it would soon choke up the filters. The partially clarified water is run into one of the precipitating tanks, is well mixed, and, if acid, is made alkaline by adding some lime. As a precipitant, the author favours a

basic ferric sulphate containing from 5—10 per cent. of aluminium sulphate. A solution of this precipitant is added to the now alkaline water, and the precipitate is allowed to settle for 16—36 hours. The water finally passes through filters composed of cinders, gravel, and sand. —I. S.

Filtration, An Improved Method of. U. S. Consular Reps. 1896, **53**, [197], 164—167.

The following process, devised by Fischer, is used for filtering Rhine water at Worms. Clean sharp sand, when mixed in the proper proportions with finely powdered glass, forms a porous mass, which by roasting at a high temperature may be hardened into any desired form. At present, plates are made 40 ins. square and 8 ins. thick, with a hollow space of 2 ins. in the centre These plates are placed upright in batteries, and are covered to a depth of 3 or 4 ft. with the water to be filtered. The water is forced by its own pressure through the porous walls of the plate into the internal hollow space, where it trickles down and is drawn off through pipes laid at the bottom of the tank to the reservoir. The discharge pipes are fitted with cocks, so that each plate and group of plates may be isolated for cleaning purposes whilst the adjacent batteries are in operation. For greater economy of space and tubing two tiers of plates may be set one above the other, one discharge pipe serving for both. The plates can be easily and quickly cleaned by reversing the current of water, whereby it percolates outwards through the porous walls and discharges the dirt which has collected on the outer surface. The mud is drawn off and the operation finished by flushing with a jet of water. The head of water necessary to make the filter act with sufficient rapidity, is from 3 to 4 ft., and the reverse head for cleaning should be about 6 ft. The water delivered by the above-described filter is said to be thoroughly purified and suitable for drinking, as well as for culinary and manufacturing purposes.—J. L. B.

PATENTS.

Softening Water for Industrial and other Purposes, An Improved Method of and Apparatus for. E. D'Huart and M. and G. Glaesener, Luxembourg. Eng. Pat. 9646, May 6, 1896.

This process depends on the insolubility in water of the oleates of the alkaline earths and the heavy metals, and the ease and rapidity with which the precipitates rise to the surface. The water is agitated with the necessary amount of oleic acid, and sufficient caustic soda to throw down the lime, &c. is added; care being taken to avoid excess. Or sodium oleate may be prepared in a separate vessel from the carbonate, by the aid of steam, and then introduced into the water. For many industrial purposes the opalescence of the softened liquid has no deleterious effect; but it may be clarified, after determination of its alkalinity, by treatment with the equivalent quantity of aluminium or iron salts, or by hydrochloric or sulphuric acid. The latter reaction must also be accompanied by agitation, and it may appropriately take place in the presence of "frictional agents, such as sawdust and paper material"; the water being finally passed through a sand filter.

The specification includes a description of, and a claim for, a suitable plant for dealing with comparatively large volumes of liquid in a short time.—F. H. L.

Extracting Grease and other Impurities from Feed Water for Steam Boilers and other Purposes, Impts. in Filters for. E. C. Mills, Manchester, and J. G. Chamberlain, Tipton. Eng. Pat. 11,136, May 22, 1896.

Inside a cylindrical vessel a perforated D-shaped metal plate is fixed eccentrically, so as to leave an "annular" space between it and the casing on one side, and a small space on the other. It is bolted to the walls at its corners, also making a tight joint with the top of the vessel when this is in place, and with the bottom, except in its straight portion, opposite which is the inlet for water. The plate is covered with layers of wire gauze or netting and canvas or asbestos, whilst the inner cavity is filled with wood shavings or the like. The outlet is in the annular space. Suspended from the cover by bolts is an inverted wooden cone, which can be driven downwards to compress the filtering material. The apparatus is provided with an inlet for steam and a blow-off cock, so that the whole may be cleared from deposit in a direction opposite to the normal flow. —F. H. L.

Town and other Refuse, An Improved Method of and Process for Utilising. L. Cséry, jun., Budapest. Eng. Pat. 29,607, Dec. 23, 1896.

The process consists in separating and sorting, by suitable mechanical means, the different constituents of the refuse, and utilising each according to its properties. Thus, the comparatively light and combustible bodies, such as paper, wood, straw, and the like, are separated and used as fuel for working the machinery; the manure substances proper, (*e.g.*, ashes, earthy matter, &c.) are separated from fragments of metal, glass, and clay, the latter being likewise sorted; and finally the substances remaining unsorted (principally organic matter) are subjected to dry distillation, the volatile products being collected, whilst the residue from the distillation is added to the manure products.—A. K. M.

Water (Drinking) Free from Germs, An Improved Process for Rendering. P. Altmann, Pankow, Germany. Eng. Pat. 5793, March 4, 1897.

A measured quantity of bromine is added to the water, for the purpose of destroying the germs present, and the bromine is then rendered innocuous by the addition of ammonia. The bromine is preferably employed in the form of a potassium bromide solution containing 20 per cent. of bromine, 0·2 c.c. of this solution being sufficient to sterilise a litre of river water in five minutes. For the subsequent removal of the bromine, an equal volume of 9 per cent. ammonia is added. If the water to be treated is strongly ammoniacal, or contains much lime, sufficient bromine must be added until a slight yellow colour is produced and lasts about half a minute.—A. K. M.

(C.)—DISINFECTANTS.

PATENT.

Sterilising Apparatus, Impts. in Steam-. S. Delépine, Manchester. Eng. Pat. 10,934, May 21, 1896.

The apparatus described is intended for submitting articles of clothing, sheets, blankets, &c. to the action of a current of steam for their thorough disinfection; and it is claimed to be cheap, portable, and simple. It consists of a steaming chamber provided with a valve which blows off at a pressure of 2 lb. The steam is generated in the boiler beneath this chamber, and passes into the latter through an annular opening round the false bottom. The steriliser is jacketed, and water from the boiler is forced in the jacket by the pressure of the steam.

The apparatus may also be used for drying the articles after they have been sterilised. In order to do this, the steam is prevented from entering the inner chamber, but passes through an outer jacket, and escapes through a pipe near the top of the apparatus.—A. K. M.

XIX.—PAPER, PASTEBOARD, Etc.

Vulcanised Fibre and other Cellulose Products. C. Hofmann's Prakt. Handbuch der Papier fabrikation, [44], 1703.

"*Vulcanised fibre*" is manufactured according to the German Patent, No. 3181 of 1878, by treating cellulose (paper) with zinc chloride solution of 65°—75° B. The gelatinisation of the cellulose is taken advantage of in welding into masses of any required thickness. After removing the zinc chloride, the masses are dried and rendered waterproof by treatment with "nitrating" acid.

Viscose.—The sulphocarbonates of cellulose (this Journal, 1893, 516; Eng. Pat. 8700 of 1892) are briefly noticed; also the nitrates of cellulose, as used in the production of *celluloid* and *artificial silk* (lustracellulose).—C. F. C.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

Bromides and Iodides of Alkalis, Preparation of. J. Knobloch. Pharm. Zeit. 1897, **42**, 190.

The reactions upon which the process is based consist in first combining the haloids with iron in the form of ferroso-ferric bromide or iodide, which is then decomposed with an excess of milk of lime, the iron being completely precipitated, and a solution of calcium bromide or iodide obtained, which in its turn is decomposed with a solution of an alkaline sulphate. The details of the method may be illustrated by the example of *Potassium Bromide*. Eight parts of iron filings are introduced into a flask with 20 parts of water, to which 16 parts of bromine are gradually added with constant agitation and careful cooling. When action is complete, and the green solution of ferrous bromide is obtained, it is decanted, and another 4 parts of bromine are added to it. The solution is then treated with a slight excess of milk of lime, by which means the whole of the iron is precipitated as magnetic oxide. 21 parts of potassium sulphate are now added, and the mixture is heated on the steam-bath. The hot solution is filtered off, the precipitate washed with hot water, only the first part of the washings being added to the filtrate, the subsequent portions being reserved for a subsequent operation. The small amount of sulphate in the filtrate is then precipitated by the cautious addition of a little barium bromide solution, any slight excess of this salt being decomposed with a very slight excess of potassium carbonate. The solution is then filtered. The filtrate is finally neutralised with a trace of hydrobromic acid, concentrated and crystallised. The bromides and iodides of the other alkalis are prepared in a precisely similar manner, substituting their respective equivalents for the quantities employed in this instance.
—J. O. B.

Mercurous and Mercuric Salts, The Interconvertibility of. S. Hada. J. Coll. Science. Imp. Univ. Japan, 1897, **9**, [2], 161—194.

The author establishes the following facts:—All mercurous salts dissociate, in presence of water, into mercury and mercuric salts, and reciprocally, all mercuric salts combine with mercury, in presence of water, to form mercurous salts, when these are able to exist. Some, and probably all mercurous salts are oxidisable by air, in presence of water, when the temperature is much above 100° C.; but at and below that temperature, mercurous salts are not oxidisable in the air, or are oxidised so slowly that the effects of oxidation are not recognisable, owing to the effects of dissociation. Mercurous oxide is oxidisable at the ordinary temperature. It was also found that mercurous nitrate, in presence of water, is converted into mercuric nitrite and nitrate by the action of heat and light.—A. S.

Glycerophosphates, A Simple Method of Preparing. Delage. Bul. gén. de thérap. (Sec. Pharm. J. **1**, 225).

One part by weight of phosphoric acid, sp. gr. 1·454, is mixed in a flask with one and a half parts of glycerin, sp. gr. 1·242. The flask is furnished with a double-bored cork provided with a thermometer and an exit tube. It is then gradually heated in the ordinary way, over a Bunsen flame. At 120° the liquid assumes a straw colour, and as the temperature slowly rises, it gradually darkens, until 190° is reached, when the colour is that of dark beer, and vapours of acrolein are given off. The heat is removed, and the vessel allowed to cool, a viscous mass resulting. 100 grms. of phosphoric acid and 150 grms. of glycerin should require about 40 minutes to complete the reaction.

To obtain glycerophosphate of lime, the resulting liquid is poured, in small quantities at a time, into an excess of a mixture of 50 grms. of lime to 250 grms. of water. The mixture is stirred, and when effervescence ceases, allowed to stand for six hours and then filtered. To the faintly yellow clear filtrate, half its volume of 90 per cent., alcohol is added, which throws down a flocculent precipitate of glycerophosphate of calcium. This is collected, washed with alcohol of the same strength, re-dissolved in water, re-precipitated with alcohol, and dried at as low a temperature as possible in a desiccator. In this manner a white powder is obtained, consisting of masses of microscopic crystals, soluble in about 20 parts of water, 100 grms. of phosphoric acid yielding about 6 grms. of this salt. From the mother-liquors another salt, having the composition of an acid glycerophosphate, is obtained. This is not precipitated by alcohol.

The glycerophosphates of sodium and potassium can only be obtained in solution. Those of magnesium, strontium and lithium are readily obtained from their carbonates in a similar manner to the lime salt. The iron salts are easily prepared, ferric glycerophosphate being obtained by the action of glycerophosphoric acid on moist ferric hydrate, and precipitating the solution with alcohol. The ferrous salt is prepared in a similar way, using the ferrous carbonate.

The author gives the following reactions for the pure salts:—In aqueous solutions they are precipitated by heat. Alcohol and ether precipitate them. They give no immediate precipitate with ammonium phosphomolybdate, nor with magnesium mixture, nor with uranium acetate. The white silver nitrate precipitate is soluble in an excess of water. The white precipitate given by lead acetate is soluble in acetic acid. When treated with absolute alcohol, no residue should be obtained on evaporation.—J. O. B.

Hydroxylamine Sulphate, Economic Preparation of. E. Divers and T. Haga. J. Coll. Science, Imp. Univ. of Japan, 1897, **9**, [2], 291—293.

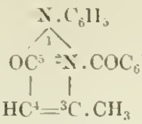
A concentrated solution of commercial (95 per cent.) sodium nitrite (2 mols.) and sodium carbonate (1 mol.) is treated with sulphur dioxide till just acid, the liquid being kept well agitated at −2° to −3° C. A few drops of sulphuric acid are added and the mixture gently warmed, when the oximidosulphonate formed at first, rapidly hydrolyses into oxyamidosulphonate and sodium bisulphate. The solution of these salts is kept at 90°—95° C. for two days, at the end of which time all oxyamidosulphonate will have been converted into hydroxylamine sulphate and sodium bisulphate, only a very small trace of ammonium salt being produced. At 80°—85° C., five days are necessary, but practically no ammonia is formed; at 70° C. three weeks at least are necessary; whilst at the ordinary temperature, much oxyamidosulphonate remains after several months. If the solution be kept boiling, seven or eight hours are sufficient to decompose all sulphonate, but at least one-third of the hydroxylamine is converted into ammonia, whilst another third is wasted as a practically inseparable mixture of its sulphate with ammonium sulphate.

To ascertain whether all the sulphonate is decomposed, excess of barium chloride is added to a little of the solution which is then filtered and boiled with potassium chlorate, thus converting any sulphonate into sulphate. When sulphonation is complete, the solution is neutralised with sodium carbonate, using methyl orange as indicator, evaporated till it weighs 10·5 to 11 times as much as the sodium nitrite taken, and then cooled to 0° C. or lower, when nearly all the sodium sulphate will crystallise out. The mother-liquor evaporated sufficiently and cooled to the ordinary tempercture, yields hydroxylamine sulphate, the mother-liquor from which, very slightly diluted and cooled below 0° C., gives a little more sodium sulphate and can be worked for hydroxylamine sulphate as before. The hydroxylamine sulphate is purified by re-crystallisation. Sodium nitrite on the small scale yields, by this method, nearly its own weight of pure hydroxylamine sulphate, and on the large scale, the theoretical yield of 118·84 per cent. could no doubt be more nearly approached.—A. S.

1-Phenyl-3-Methyl-5-Pyrazolone, Constitution of Acid Derivatives of. Fr. Stolz. J. prakt. Chem. **55**, 145—171.

According to Nef (Annalen, **266**, 155) the action of benzoyl chloride and soda lye on pyrazolone or of benzoyl

chloride on silver pyrazolone yields phenylmethyl-benzoylpyrazolone melting at 75° C. and having the formula—



This formula is based on the assumption that the silver in silver pyrazolone is in the position 2, and he further states that this latter compound when acted on by methyl iodide yields only antipyrine. The author's investigations show that not only is this latter reaction incomplete but also that the action of benzoyl chloride on silver pyrazolone does not yield exclusively pyrazolone. As a matter of fact this and other acidyl derivatives which have been prepared by Autenrieth (Ber. **29**, 1659), Himmelbauer (J. prakt. Chem. **54**, 177 *et seq.*), and by the author are oxygen esters, *i.e.*, are 5-derivatives of pyrazolone, and for the following reasons :—The action of methyl iodide on benzoylphenylmethylpyrazolone gives an iodomethylate which on warming with dilute alkali decomposes quantitatively into antipyrine, benzoic acid and hydriodic acid.

Antipyrine, (1-phenyl-2-alkyl-3-methyl-5-pyrazolone), gives characteristic reactions with ferric chloride and nitrous acid which are not shown by 1-phenyl-3-methyl-5-alkyloxypyrazolone, and further, the antipyrines are easily soluble in water but sparingly so in ether and petroleum spirit, whereas the acid derivatives obtained by Himmelbauer as analogues of the alkaloxy derivatives are to be considered as oxygen esters, since they are insoluble in water but easily so in ether and petroleum spirit and do not give the ferric chloride or nitrous acid reactions of the antipyrines. The alkyl carbonic esters are similarly constituted, since they correspond in their properties (solubility and reactions, with nitrous acid and ferric chloride) to the pyrazole-5-oxyacetic esters but differ from the pyrazolone acetic esters. The former are obtained by reacting with sodium pyrazolone on chloracetic ester, whilst the pyrazolone 2-acetic acids are produced by the addition of halogen acetic ether to 5-alkyloxypyrazole and subsequent removal of the alkyl group. Himmelbauer states that the methyl carbonic ester when distilled *in vacuo* yields antipyrine (about 40 per cent of theory) together with methoxypyrazole, and he gives this in support of his view that the compound is a 2-derivative but in reality a molecular transformation takes place, and, for instance, 1-phenyl-3-methyl-5-methoxypyrazole is converted into the 2-derivative like the transformation of *p*-methoxyquinaldine into *m*-methylquinaldone. Hence this is a further proof that the compounds are 5-derivatives which are converted first into alkyloxypyrazoles and subsequently into antipyrines.—T. A. L.

Phenacetin brominated in the Benzene Nucleus. W. Vaubel. J. prakt. Chem. **55**, 217—219.

In a previous paper (J. prakt. Chem. **52**, 421) the author described a bromo-derivative of phenacetin produced by direct bromination, as an oil probably identical with that obtained by Staedel (Annalen, **217**, 73). The substance is now found to be a solid, melting about 20° C., but frequently remaining superfused. On hydrolysis, and subsequent removal of the amido group, a viscid oil is obtained, having a peculiar odour, and capable of taking up more bromine. The probable formula of bromophenacetin is $1.2.4.C_6H_3.NHC_2H_3O.BrOC_2H_5$.—T. A. L.

Bases, Organic ; The Use of Potassium-Bismuth Iodide in the Preparation of. E. Jahns. Arch. der Pharm. **235**, 151—156 ; Chem. Centr.-Bl. 1897, [1], 817.

The author recommends potassium-bismuth iodide solution as a means of precipitating bases soluble in water, the separation from the extract in this way often being quantitative. The bismuth-iodide solution is prepared according to Krauts' prescription. 80 grms. of bismuth sub-nitrate are dissolved in 200 grms. of nitric acid (sp. gr. = 1·18), the solution poured into a concentrated aqueous solution of 272 grms. of potassium iodide, and, after crystallising out the potassium nitrate, is made up to a litre. This solution gives precipitates with dilute sulphuric acid solutions of narceïne, solanine, veratrine, creatinine, &c., and also with bitter principles, glucosides, and albumins. It is best, in the case of solutions of vegetable matters, to first separate the colouring matters and albumins by precipitating with lead acetate. The excess of lead is precipitated from the filtrate by sodium phosphate, the filtered liquid evaporated, mixed with sufficiently dilute sulphuric acid, and precipitated with the bismuth iodide solution. The red precipitate is washed, and whilst still moist, triturated with silver carbonate till the red colour disappears, and the filtrate ceases to give the iodine reaction. A solution of the pure base or its carbonate is thus obtained, the dissolved traces of silver being removed by sulphuretted hydrogen. The bismuth precipitate may also be decomposed by boiling with barium carbonate and water, the barium accurately precipitated from the filtrate by H_2SO_4, and the HI removed by saturating with silver carbonate. By this method, cholin was detected in *Flor. chamomill. vulg.*, *Herb. millefol.*, *Herb.*, *meliloti*, *Fol. malvæ*, *Herb. cochlear*, *Fruct. anisi*, *vulg.*, *Cort. sambuci*, and *Sem. robiniæ pseudacaciæ*. The author also found that the alkaloid, bursine, discovered by Bombelon in *Capsella bursa pastoris* is identical with cholin. Cholin was found to be present in tolerably large quantities (up to 0·2 per cent.), together with somewhat less betaïn, in the seeds of *Lathyrus sativus* and *L. cicera*.—A. S.

Aloin, Separation of, from Aloes. G. L. Schäfer. Pharm. Zeits. für Russl. 1897, **36**, 65.

The author avails himself of the fact that aloin forms insoluble compounds with the alkaline earths in the presence of ammonia, from which it may subsequently be liberated by treatment with an acid. 50 grms. of aloes are dissolved in 300 c.c. of hot water, acidulated with a few drops of hydrochloric acid ; after cooling, the liquid is poured off from the deposited resin ; 50 c.c. of 20 per cent. ammonia solution are then added, followed by a solution of 15 grms. of calcium chloride in 30 c.c. of water ; the mixture is thoroughly agitated and allowed to stand for 15 minutes ; the precipitated calcium aloin is then collected and pressed, or drained on a centrifugal machine. The drained mass is triturated in a mortar with a slight excess of hydrochloric acid, the mixture dissolved in a minimum quantity of boiling water, filtered, the filter washed with a little boiling water, cooled, and crystallised at a low temperature by means of ice. From 15 to 30 per cent. of well-crystallised light yellow aloin is thus obtained from various kinds of commercial aloes.—J. O. B.

Scammony, a Sample of Spurious. I. W. Thompson. Pharm. J. 1897, **54**, 245.

The specimen consisted of irregular broken pieces, apparently portions of a cube, about half an inch in thickness, greenish black, hard and horny, breaking with a resinous fracture, and very difficult to powder.

The following figures were obtained on analysis :—

Soluble in ether, 0·4 ; in alcohol, 2·0 ; in water, 42·6 ; starch and cellular tissue, 43·0 ; and moisture, 12·0 per cent.

It yielded 2·12 per cent. of ash. The portion soluble in water was evidently gum, apparently gum acacia. The insoluble portion consisted very largely of starch. The sample was marked "Scammony," accompanied by a statement that it contained 84·864 per cent. of scammonin. It was said to be of German origin.—J. O. B.

Kryofine, A New Antipyretic. Pharm. Zeit. 1897, **42**, 283.

A new derivative of paraphenetidine, phenetidine-methyl-glycollate has been introduced to medicine under the name of kryofine. It is a condensation product obtained by heating together paraphenetidine with methyl-glycollic acid as shown by the equation—

$$CH_3OCH_2COOH + NH_2C_6H_4OC_2H_5 = CH_3OCH_2CONHC_6H_4OC_2H_5 + H_2O.$$

It forms white crystalline needles which melt at 98° to 99° C. These are odorless and tasteless, very sparingly soluble in cold water (1 in 600), but more readily so in hot water (1 in 52). The solution is bitter and burning in taste. In therapeutic effect, according to Eichhorst, 0·5 grm. of kryofine is equivalent to 1·0 grm. of phenacetin. If kryofine fail in certain cases, it has been found that the other synthetic antipyretics are also without reaction. Kryofine possesses useful anti-neuralgic properties as well.—J. O. B.

Pilocarpine and Pilocarpidine, Further Contribution to the Study of. A. Petit and Polonovski. J. Pharm. Chim. 1897 (10), **5**, 475; (see also this Journal, 1897, 461).

Commercial Salts of "Pilocarpine."—These almost invariably consist of the mixed salt of the two bases, pilocarpine and pilocarpidine; this is notably the case with the nitrate, specimens of which are not unfrequently met with containing 50 per cent. of pilocarpidine. This impurity may be detected by the crystals being ill-defined, instead of definite, and by the lowering of the melting point and of the optical rotation. Pure pilocarpine nitrate melts at 177°—178° C., while the specimens containing 50 per cent. of pilocarpine soften at 140° C. and are completely decomposed at 150° C. The influence on the optical rotation is so definite that by the data obtained by its observation, the respective amount of the two nitrates present may be calculated by Landolt's formula.

Pilocarpidine not a Decomposition Product. — The authors consider that pilocarpidine exists naturally in the plant, and not as a decomposition product, as stated by Hardy and Calmels. Boiling water alone, in the absence of strong acids and alkalis, cannot be capable of forming large quantities of pilocarpidine, its action upon pilocarpine being but slight. Even when the extraction and separation of the alkaloids is conducted wholly without heat, notable quantities of pilocarpidine are obtained; again, operating under the same conditions with different species of jaborandi, the yield of pilocarpidine is very variable, oscillating between 5 and 75 per cent. of the total alkaloids, according to the variety of leaf employed. It is found also that the stems usually give markedly more pilocarpidine than the leaves of the same plant.

Pilocarpic and Pilocarpidic Acids.—The two bases are considered to be the anhydrides of pilocarpic and pilocarpidic acids respectively. Upon the addition of dilute alkali to a solution of pilocarpine, hydrolysis takes place; if phenolphthalein be used as an indicator, the colour gradually disappears in the cold, and rapidly on warming. In this way one equivalent of alkali is found to combine with each equivalent of pilocarpine. On exactly neutralising, the optical rotation is found to remain identical with that of the sodium compound, and notably lower than the figure for pilocarpine. On adding an excess of acid, however, gradual dehydration of the pilocarpic acid takes place, pilocarpine is regenerated and ultimately the rotation is brought back to that of pilocarpine. In this manner the progressive hydration of the base by alkaline solution, and the subsequent dehydration in an acid solution of the acid at first formed, may be followed by polarimetric observation.

Although these acids have hitherto been regarded as hypothetical, the authors have succeeded in preparing them in a free state, by decomposing the barium salts with an exact equivalent of sulphuric acid. They are thus obtained in the state of amorphous varnishes, very soluble in water but insoluble in chloroform or in ether; on heating, partial dehydration takes place, with partial regeneration of the alkaloids. In the presence of free acid this is more rapid and, on heating, is complete. The only observed difference in the behaviour or composition of the two acids or of their salts, is the fact that pilocarpidic acid and its compounds are levogyrate.

The authors conclude that pilocarpine and pilocarpidine are isomers, both having the formula $C_{11}H_{16}N_2O_2$. The assumption that pilocarpidine differs from pilocarpine in having a methyl group less, giving it the composition $C_{10}H_{14}N_2O_2$, is not borne out by the figures obtained by analysis, nor by theoretical considerations. If the constitution assumed for the two bases by Hardy and Calmels be correct, it is impossible for pilocarpidine to possess an anhydride group, although the authors now show that in its behaviour towards alkalis it is exactly similar to pilocarpine; in fact, the only marked differences observed between the two alkaloids and their products lie in their physical constants.—J. O. B.

Holocaïne, A New Anæsthetic. G. Gutmann. Deutsch. Med. Wochenschr. 1897, [11], 165—167; Chem. Centr.-Bl. 1897, [1], 875.

According to Täuber, holocaïne is *p*-diethoxyethenyl-diphenylamidine and is prepared by the combination of molecular quantities of phenacetin and *p*-phenetidine, with separation of water—

$$C_2H_5O.C_6H_4.NH.CO.CH_3 + NH_2.C_6H_4.O.C_2H_5 =$$
$$C_2H_5O.C_6H_4.NH.C(CH_3):N.C_6H_4.OC_2H_5 + H_2O.$$

The beautiful crystalline base is insoluble in water, volatilises at 121° C., and forms soluble salts. The hydrochloride forms minute white needles soluble in boiling water. The cold saturated aqueous solution of the hydrochloride contains 2·5 per cent., tastes faintly bitter, reacts neutral and is not altered by boiling. On boiling the solution in glass vessels, it sometimes becomes turbid, owing to a small quantity of the alkali of the glass being dissolved, and the insoluble amidine base set free. Porcelain vessels should therefore be used when dissolving the hydrochloride. The author considers holocaïne better than cocaïne as a local anæsthetic, as it acts efficiently in one to two minutes.—A. S.

Cedar-Wood, Essence of. L. Rousset. Bull Soc. Chim. 1897, **17**, 485—489.

Pure essence of cedar-wood was subjected to fractional distillation *in vacuo*, 80 per cent. of a hydrocarbon boiling at 125°—130° under a pressure of 9 mm., and a solid product distilling at 149°—155° under 8 mm. were obtained, the intermediate portion being insignificant.

Cedrene, $C_{15}H_{24}$. — This hydrocarbon, distilled over sodium, boils at 131°—132° under 10 mm. It is optically active, $\alpha_D = -47° 54'$. The hydrocarbon absorbs bromine, hydrochloric and hydrobromic acids, but the compounds spontaneously decompose and on distillation yield cedrene. Oxidised by chromic acid in acetic acid solution, the ketone $C_{15}H_{24}O$, cedrone, is obtained. This distils at 147°—151° under 7·5 mm., does not combine with sodium bisulphite, but yields an oxime boiling at 175°—180° under 8 mm.

Cedrone on reduction gives the alcohol isocedrol $C_{15}H_{26}O$, a viscous liquid of boiling point 148°—151° under 7 mm. A benzoic ether was obtained. Cedrone treated with sodium hypobromite and potassium iodide gives iodoform, and hence probably contains the acetyl group.

Cedrene, by further oxidation, gives a viscous monobasic acid $C_{12}H_{18}O_3$, and some dimethylketone. Oxidation by nitric acid or alkaline permanganate does not give favourable results. By the action of sulphuric or glacial acetic acid cedrene gives no hydrate.

Cedrol, $C_{15}H_{26}O$.—The solid product of the distillation after repeated recrystallisation from methyl alcohol forms fine white needles melting at 84°. A molecular weight determination and analysis, point to the formula given. Cedrol is optically active. Treated with acetic anhydride at 100° it is transformed into an ester and partly into a hydrocarbon $C_{15}H_{24}$. Benzoyl chloride gives rise to no ester, but produces the same hydrocarbon. Cedrol has thus properties very different from those of the alcohol obtained by reduction of cedrone. Oxidised by chromic acid, a sesquiterpene boiling at 115°—117° under 6·5 mm. is obtained, no aldehyde or ketone being formed. Cedrol, therefore, appears to be a tertiary alcohol.—A. C. W.

Geranium Essence, Researches on. E. Charabot. Bull. Soc. Chim. 1897, **17**, 489—492.

The author finds that the rotatory power of geranium essences is decreased by saponification, hence they must contain a levo-rotatory ester. This ester is almost entirely contained in the part of the essence which is most easily volatile with steam; essence of palmarosa contains no similar levo-rotatory ester.—A. C. W.

Tea, Essential Oil of. Schimmel's Rep., April 1897, 40.

The essential oil of tea leaves which has so far been obtained in the proportion of only 0·006 per cent. had the following characters. The specific gravity was 0·866, and the optical rotation slightly levo-rotatory, 0° 11′ in a 200 mm. tube. Fractional distillation separated the oil into two parts, one boiling below 170° C. and the other above that temperature. The lower boiling fraction contained a body boiling between 153° C. and 154° C., having a pungent fusel-oil like odour; the formula for this was found to be $C_6H_{12}O$. It formed a benzoyl compound and an acetic ester, being evidently of an alcoholic nature; by oxidation with bichromate of potassium and sulphuric acid a liquid was obtained which appeared to contain butyric acid.—J. O. B.

Camphor Wood Oil, Venezuelan. Schimmel's Rep., April 1897, 45.

The botanical source of the "camphor wood" from Venezuela, which was distilled, has not been determined. It gave 1·15 per cent. of volatile oil, which solidified to a crystalline mass at the normal temperature; the specific gravity was 1·155, and the optical rotation 2° 40′ in a 100 mm. tube (temperature not stated). The crystals drained from the liquid and recrystallising from alcohol were large, well-formed prisms melting at 28·5° C. With alcoholic potash a compound crystallising in plates which melt at 55·56° C. was obtained. This was evidently apiol, which was present in the oil to the extent of 90 per cent. Its characters agreed in every particular with apiol derived from parsley oil.—J. O. B.

Wormwood Oil. Schimmel's Rep., April 1897, 17.

Hitherto only one definite constituent of this oil, thujone, has been known. A pure sample of the oil was therefore examined; this had a specific gravity of 0·932. After removing the greater part of the thujone by means of sodium bisulphite and saponifying the residue with alcoholic potash, the residue of steam distillation was found to contain palmitic acid; acetic and isovalerianic acid were also found to exist as esters in the oil. The lowest fractions boiling between 158° C. and 168° C. were found to contain phellandrene; pinene, if present, existed in so small a trace that it could not be isolated. It is evident that an oil containing much pinene can only be adulterated. A ready test for the freedom of wormwood oil from adulteration with turpentine consists in distilling off about 10 per cent. of the sample. One part of this distillate should give a clear solution with 40 parts of 80 per cent. alcohol. Besides thujone a higher boiling body distilling between 210°—215° C. was obtained; this proved to be thujyl (tanacetyl) alcohol. By quantitative saponification of the oil before and after acetylation, the amount of thujol free and as acetate was found to be 24·2 per cent. Several commercial specimens of wormwood oil were found to be adulterated with turpentine.—J. O. B.

Goldenrod, Canadian; Essential Oil of. Schimmel's Rep., April 1897, 46.

The oil of *Solidago canadensis* has been recently found to contain 85 per cent. of terpenes, mainly pinene, with a little phellandrene and dipentene, possibly also a little limonene. The higher fractions contain borneol, 5·8 per cent., and bornyl acetate, 3·4 per cent., with some cadinene. The close analogy in chemical composition between this oil and that of some of the pine needle oils is remarkable, considering the widely different botanical position of the two sources.—J. O. B.

Schinus Molle (Pepper Tree), Essential Oil of. Schimmel's Rep., April 1897, 44.

The oil was distilled from the berries imported from Mexico; a yield of 5·2 per cent. of a light oil being obtained. This had an odour of phellandrene; the specific gravity was 0·850 at 15° and the optical rotation + 46° 4′; with sodium nitrite and glacial acetic acid, it gave the characteristic reaction for phellandrene nitrite. Only traces of phenols could be found.—J. O. B.

Mandarin Oil, Chemical Composition of. Schimmel's Rep., April 1897, 21.

In endeavouring to prepare citryl-β-naphthocinchonic acid from the fractions of mandarin oil which contain citral, although the characteristic precipitate was obtained, it did not give, even after repeated crystallisations, the correct melting point for the pure substance. It commenced to melt at 197° C., but was not fully melted until the temperature reached 222° C. It is, therefore, probable that this oil, like lemon oil, contains both citral and citronellal. The presence of limonene in the fraction boiling between 175° and 179° was indicated by preparing the dichlorhydrate, melting at 49° C., and the tetrabromide melting at 104°—105° C. This fraction was strongly dextro-rotatory (+ 77°) and consisted mainly of dextro-limonene.—J. O. B.

Lemon Oil, Influence of Climate on. Schimmel's Rep., April 1897, 18.

The influence of climatic variations on the constitution of lemon oil has been markedly shown during the last season in the northern and eastern districts of Sicily. The summer there has been cool with an abnormal rainfall. The oil produced in this part of the island was found to have an optical rotation two or three degrees below the normal standard, while that derived from the southern district, where the weather was hot and dry, retained its normal optical activity. Careful observations of the produce of each month during the season, has shown that atmospheric conditions have a distinct influence, not only on the optical behaviour of lemon oil, but also on the ester percentage of bergamot oil.—J. O. B.

Lemon Oil, Absence of Pinene in. Schimmel's Rep., April 1897, 20.

In order to determine definitely if pinene could be regarded as a normal constituent of lemon oil, 50 kilos. of it were submitted to fractional distillation; after re-fractionation, the lowest boiling portion, which distilled at 172° C., amounted to only 8 c.c., or 0·016 per cent. of the whole, and had none of the characters of pinene. If, therefore, that body be detected in a sample of lemon oil, it may with certainty be pronounced to be adulterated with turpentine. Recent investigations show that this form of sophistication is again being largely practised, specimens containing as much as 60 per cent. of oil of turpentine having been met with.
—J. O. B.

Dill Oil. Schimmel's Rep., April 1897, 15.

German dill oil, although differing markedly in odour from carraway oil, resembles it very closely in chemical constitution, and at present, no definite substance accounting for the variation in odour has been isolated. The peculiar dill-apiol detected in East Indian dill oil by Ciamician and Silber, is not found, it is said, in the German oil; no constituents of any importance with a boiling point above that of carvol having been isolated. The residue, on fractionation, gave only a small quantity of a paraffin, melting at 64° C. A specimen of English dill oil recently examined, was found to give, with sodium nitrite and glacial acetic acid, a marked reaction for phellandrene. With German oil this reaction was not obtained.—J. O. B.

Damiana Oil. Schimmel's Rep., April 1897, 15.

Leaves of damiana (derived from several species of *Turnera*) distilled in Leipsic last year yielded about 1 per cent. of oil. This had a specific gravity of 0·943, which was much lower than that of a specimen distilled in 1888, which had a density of 0·970 and 0·980. The new sample was levo-rotatory, the index being −23° 25′; the saponification figure was 41·8. On standing, it deposited crystals resembling in appearance those of the stearoptene of rose oil; it is possible, therefore, that damiana oil may contain a paraffin. The oil has at present, no practical application.
—J. O. B.

Culilaban Oil. Schimmel's Rep., April 1897, 14.

From culilaban bark, derived from *Cinnamomum culilavan*, 4 per cent. of oil was obtained. This had a specific gravity

of 1·051 and possessed a strong odour of eugenol; the oil was dark in colour. The phenol was isolated and proved to be eugenol, giving benzoyl-eugenol, melting at 70°—71° C. After the removal of the eugenol, the remainder of the oil boiled between 100° and 130° C., under a pressure of 10 mm.; the main fraction boiling at 125° to 130° had a specific gravity of 1·031, the fractions with a lower boiling point being lighter than water. Distilled under normal atmospheric pressure, the fraction distilling between 249° and 252° C., gave, upon oxidation with potassium permanganate, veratric acid, with a melting point between 170° and 180° C. Analysis of the silver salt gave figures corresponding with the formula $C_9H_9O_4Ag$. This result, as well as the boiling point of the fraction, indicate methyl-eugenol. By bromination, tribromo-eugenol was obtained. Culilaban oil, therefore, consists, it is stated, mainly of eugenol with some methyl-eugenol, and a small amount of constituents of a lower specific gravity than water. —J. O. B.

Champaca Oil. Schimmel's Rep., April 1897, 11.

Champaca oil, distilled from the fresh flowers of *Michelia champaca*, a native of Manilla, is a very fragrant, light-brown liquid, having a specific gravity of 0·938, the optical rotation is 52° 8′ in a 100-mm. tube, and a saponification factor of 77·3. The oil is soluble in absolute alcohol, but is not easily dissolved in weaker spirit. The odour somewhat resembles that of Ylang-ylang, and, like it, champaca oil contains benzoic acid.—J. O. B.

Anise Oil, Adulteration of, with Fennel Oil Stearoptene. Schimmel's Rep., April 1897, 6.

From the fact that pure oil of aniseed is always slightly levo-rotatory (−1° 50′), while certain specimens of low-priced anise oil met with in commerce are dextro-rotatory, it is suspected that these latter are adulterated with the stearoptene of the cheaper fennel oil. Since this always retains a considerable amount of fenchone, which is strongly dextro-rotatory, its presence imparts an abnormal optical rotation to a mixture of anise oil. Anise oil which is not slightly levo-rotatory should therefore be rejected. —J. O. B.

Valerian Oil, Mexican. Schimmel's Rep., April 1897, 44.

The root distilled was imported from Mexico, and was probably derived from *Valeriana mexicana*. On simple distillation no oil was obtained, and it was not until cohobation had been first performed that any product was yielded. The specific gravity of the oil thus finally obtained (an oil optically inactive) was 0·949 at 15° C.; it was entirely soluble in soda solution. The acid number, when titrated with alcoholic potash, was 415: equivalent to 89 per cent. of valerianic acid. There is, then, practically no true essential oil present in the root, but simply valerianic acid, which appears to exist in the free state.—J. O. B.

Bengal Cardamom Oil. Schimmel's Rep., April 1897, 43.

Bengal cardamoms yielded 1·12 per cent. of a light yellow oil having a specific gravity of 0·920 at 15° C., and an optical rotation of 12° 41′; the odour strongly recalled that of cineol. On distilling, the major fraction passed over below 220° C.; in this the supposed cineol was separated as hydrobromide, from which it was subsequently obtained in the pure condition by decomposing with water, when it was found to have all the physical and chemical characters of pure cineol, and to give, on oxidation, cineolic acid, melting at 197° C. Bengal cardamom oil has little of the characteristic aroma of cardamoms, and therefore is not likely to be useful commercially.—J. O. B.

Clove Oil. Schimmel's Rep., April 1897, 45.

The body which gives to clove oil its fruity ethereal odour, which is not found in pure eugenol, has been isolated, and proved to be normal amyl-ketone, $CH_3(CH_2)_4CO.CH_3$, which has not been previously observed as a constituent of essential oils. It was obtained from the first fraction of clove oil, boiling between 150° and 155° C., and was purified from furfurol by shaking with cold potassium permanganate solution, after which it had a constant boiling point of 151° to 152° C. It is only present in the oil in extremely minute quantities.—J. O. B.

Basilicum Oil Distilled in Grasse. Dupont and Guerlain. J. Pharm. Chim. 1897, 5, [9], 453.

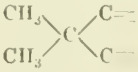
The oil was a somewhat thick yellowish fluid with a strong and characteristic odour. The specific gravity at 15° C. was 0·9154, and its rotation in a 100-mm. tube, 7° 40′. The chief constituents proved to be left-handed linalool and estragol. At ordinary atmospheric pressure, 80 per cent. of the oil distilled between 190° and 220° C. On re-fractionating this, two principal fractions were obtained, one boiling between 195°—200° C., and the other between 205°—215° C.

The former, which represented 60 per cent. of the original oil, proved to be levo-rotatory linalool, as shown by its physical properties, by its bromo derivative, and by the formation of linalyl acetate.

The second fraction, boiling between 205°—210° C., was found to be estragol. This was proved by transforming it into its propenylic isomer, anethol. This was effected by heating it on the water-bath with twice its volume of concentrated alcoholic potash for 24 hours. The mass was then heated with water, extracted with ether, the solvent removed, and the residual oil fractionated; the higher boiling fraction, distilling between 225° and 230°, was found to be anethol, solidifying below 0° C. and melting again at + 21°. —J. O. B.

Filicic Acid, Researches on. G. Daccomo. Apoth. Zeit. 1896, 41, 812; J. Pharm. Chim. 1897, 5, [9], 147.

From the behaviour of hydroxylamine with filicic acid and the action of barium hydrate on the copper salt, it appears that this acid does not belong to the aromatic series, but is a fatty acid. It seems to be a β-ketone-aldehyde containing the nucleus—



When a mixture of filicic acid in benzene is added to an alcoholic solution of hydroxylamine and heated with calcium carbonate, a reddish-brown body is formed which, on distillation in a partial vacuum, leaves a brownish residue, with an odour of pyridine. On washing this with dilute soda and recrystallising the insoluble portion from alcohol, a reddish powder composed of prismatic crystals is obtained. These have the composition $C_{14}H_{15}O_4N$; that is to say, they are the anhydride of filicic acid, $C_{14}H_{16}O_4NOH$, and not the oxime. When methylic ether is used as a solvent of the filicic acid in the above reaction, an isomeric modification of the same anhydride is obtained.

When the copper salt of filicic acid is heated with baryta water, decomposition takes place at ordinary temperatures; the copper salt gradually dissolves and is then reduced, a precipitate of cuprous oxide resulting, and a slightly aromatic acetone-like odour is given off. At a temperature of 50°—60° C. reduction is complete in two or three hours. By this reaction, acetone, isobutyric, normal butyric, and dimethylmalonic acids are all products, and are all members of the series of fatty acids.—J. O. B.

Acetone, Impt. of Squibbs' Volumetric Method for Estimating. L. F. Kebler. J. Amer. Chem. Soc. 1897, 19, 316.

See under XXIII., *page* 569.

Chloroform, Analysis of. A. Béhal and M. François. J. Pharm. Chim. 1897, 5, 417.

See under XXIII., *page* 566.

Marmé's Reagent, Limit of Sensibility of, to certain Alkaloids. S. Verven. Ann. de Pharm. 1897, 13, 145; Chem. Zeit. Rep. 1897, 21, 116.

See under XXIII., *page* 567.

Filicic Acid, Various Methods of Determining, in Extract of Male Fern. J. Pharm. Chim. 5, [6], 443.

See under XXIII., *page* 568.

Carvacrol, Determination of. E. Kremers. Pharm. Rundschau, 1896, 221; Schimmel's Rep., April 1897, 41.

See under XXIII., *page* 568.

Thymol, Determination of, in Thyme Oil. Kremers. Pharm. Rundschau, 1896, 221; Schimmel's Rep., April 1897, 41.

See under XXIII., *page* 568.

Lavender Oil, Detection of Adulteration of. Schimmel's Rep., April 1897, 24.

See under XXIII., *page* 563.

Star Anise Oil. [*Testing for Kerosene.*] Schimmel's Rep., April 1897, 37.

See under XXIII., *page* 563.

Sandal-Wood Oil, East Indian; Analytical Data of. Schimmel's Rep., April 1897, 36.

See under XXIII., *page* 568.

PATENTS.

Xanthine Derivatives, Impts. in the Manufacture of, from Alkylated Uric Acids. [*Synthesis of Caffeine.*] E. Fischer, Berlin. Eng. Pat. 10,004, May 11, 1896.

The process consists in the preparation of the halogen derivatives of alkylated xanthines by treating tetra-alkyluric acids with the halogen compounds of phosphorus, and the production of trialkylated xanthines by reducing, in the usual manner, the halogen derivatives so obtained. By means of this process it is possible to prepare caffeine from tetramethyluric acid, and incidentally from uric acid. To convert tetramethyluric acid (the method for the preparation from uric acid is well known) into chloro-caffeine, it is heated in a closed vessel with five times its weight of phosphorus oxychloride for 10 hours at 160°—165° C. A clear brown liquid is formed, which, when evaporated and treated with water, gives a crystalline powder containing the chloro-caffeine. This is purified by heating with five times its weight of fuming hydrochloric acid at 130° C. for three hours; the hydrochloric acid solution is evaporated to dryness, the residue extracted with cold dilute caustic soda; the impurities having thus been converted into acids are removed, and chloro-caffeine remains behind, and may be obtained pure by a single recrystallisation from hot alcohol. Its reduction to caffeine is then accomplished in the usual way.—J. O. B.

Alkylated Uric Acids, Impts. in the Preparation of. E. Fischer, Berlin, Germany. Eng. Pat. 10,005, May 11, 1896.

Alkylated uric acids are prepared by the action of halogen alkyls upon the alkaline salts of uric acid, or on the alkyl derivatives of the acids. Details are given of the methods for (1) converting uric acid into alpha-monomethyluric acid; (2) converting alpha-methyluric acid into tetramethyluric acid; (3) converting alpha-dimethyluric acid into tetramethyluric acid; (4) converting beta-trimethyluric acid into tetramethyluric acid; (5) converting alpha-dimethyluric acid into benzyl-dimethyluric acid; (6) converting beta-trimethyluric acid into benzyl-trimethyluric acid; (7) converting alpha-dimethyluric acid into tetra-methyluric acid in the dry way.—J. O. B.

Aromatic Aldehydes, Manufacture of. O. Imray, London. From The Farbwerke vorm. Meister, Lucius, and Brüning, Hoechst a/Main, Germany. Eng. Pat. 10,689, May 18, 1896.

The process consists of the oxidation of benzylaniline, its homologues or nitro-substitution products, and of separating in the usual way, by means of diluted mineral acid, the benzylidene or nitrobenzyl thus obtained into aniline bases and aldehyde. The following process is followed for the manufacture of benzaldehyde:—100 kilos. of benzylaniline and from 500 to 1,000 litres of water are placed in a large retort fitted with an agitator; during agitation and boiling, the following mixture is run in gradually, during a few hours. Potassium or sodium bichromate, 50 kilos; water, 200 litres, acidulated with hydrochloric acid (21° B.), 165 kilos., or its equivalent of sulphuric acid. Distillation of water and benzaldehyde soon results. An equivalent proportion of benzyltoluidine may be substituted for the benzylaniline. By substituting 120 kilos. of *o*-nitro-benzylaniline or its equivalent of *o*-nitro-benzyltoluidine, *o*-nitro-benzaldehyde crystallises out from the distillate on cooling: by substituting the equivalent "para" modifications, the corresponding "paraldehyde" is obtained. From neutral solutions *o*-nitro-benzaldehyde may be made as follows:—23 kilos. of *o*-nitrobenzylaniline (or the equivalent of *o*-nitro-benzyltoluidine, or *o*-nitro-benzylxylidine) are dissolved in acetone and then a cold aqueous solution of 12·5 kilos. of potassium permanganate is run in, the temperature being kept at about 10° C. The solution is filtered and the acetone distilled off. Hydrochloric acid (15 kilos.) is then added to the aqueous residue, when *o*-nitrobenzaldehyde crystallises out; it is collected, washed, and purified by steam distillation.
—J. O. B.

Pharmaceutical Compounds [*Iodine Compounds from Sponges, Seaweeds, and similar Material*], *Manufacture of.* H. E. Newton, London. From The Farbenfabriken vorm. F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 11,458, May 26, 1896.

Medicinally active iodine compounds are obtained from sponges, laminariæ, fucus, and similar material by the action of dilute acids, followed by extraction with dilute alkalis, and then precipitating the alkaline extract by acids or by alcohol. The bodies so obtained are claimed as new, and available for medicinal purposes. Five parts of common sponge are heated with 20 to 30 parts of 5 per cent. sulphuric acid at 100° C. for 12 to 15 hours. The liquid is filtered when cold and the insoluble residue again treated with soda solution (containing 1 or 2 per cent. of NaHO) to extract the iodine compounds. The filtered alkaline liquid is made acid, when the iodine compounds are precipitated as a red-brown powder, which is collected, washed, and dried. After treatment with acid, the residue may, if necessary, be purified by extraction with boiling alcohol, in which the iodine compounds are insoluble. The residue is then extracted with the weak alkaline menstruum as described; or the material may be treated direct with alkali without the previous extraction with acid.
—J. O. B.

Albuminous Compounds, New [*with Formic Aldehyde*], *Manufacture of.* O. Imray, London. From The Farbwerke vorm. Meister, Lucius, and Brüning, Hoechst a/Main, Germany. Eng. Pat. 11,878, June 1, 1896.

By allowing aqueous solution of white of egg to act upon formic aldehyde and then boiling off the excess of the latter and evaporating the product to dryness, a modified albumin is obtained. This is soluble in water, does not coagulate on boiling, and is not precipitated by soda or ammonia; alcohol and acetone precipitate it, and the substance thrown down, when dried, is still soluble in water.

The new product is thus obtained. Four kilos. of white of eggs are mixed with about 25 grms. of formic aldehyde (40 per cent.), and left for several days. Water is then added and the solution boiled until the excess of aldehyde is driven off, more water being added if necessary. The solution is then filtered, evaporated at a low temperature to the desired concentration, or to dryness *in vacuo*.—J. O. B.

Salicin, Saligenin, and Free Hydrochloric Acid: A Compound of good keeping Property, of. W. P. Thompson, London. From Ludwig Sell and Co., Pasing-Munich, Germany. Eng. Pat. 12,434, June 6, 1896.

A fluid extract of horse-chestnut is mixed while hot with an equal weight of salicin, the mixture warmed on the water-bath for 20 minutes, or until a reaction for saligenin is obtained. Twice as much salicin is then incorporated with the warm mass, and the whole is kneaded thoroughly, spread on glass plates, and dried at 19° C. The resulting product is a composition which is said to keep well, and contains salicin, saligenin, glucose, and free hydrochloric acid.—J. O. B.

Tobacco, Impts. in Manufacture of [*Combination of Essential Oils and Aromatic Herbs with*]. R. Pserhofer, Vienna. Eng. Pat. 12,736, June 10, 1896.

The finely powdered leaves of *Mentha piperita, Mentha crispa, Eucalyptus* var. species, *Thymus vulgaris* or *Melissa officinalis*, are mixed directly with tobacco, or the tobacco is moistened with solutions or emulsions of the volatile oils, or the "stearoptenes" of the oils. Or the "stearoptenes" of menthol, eucalyptol, or of melissa oil "in a finely powdered state," are mixed with tobacco. Or the oils or stearoptenes are put into a distillatory apparatus, and the tobacco is impregnated with the vapours.
—J. O. B.

Artificial Musk, Impts. in Manufacture of. O. Imray, London. From "The Fabriques de Produits Chimiques de Thann et de Mulhouse," Thann, Alsace. Eng. Pat. 14,552, July 1, 1896.

The artificial musk is obtained by nitrating dimethyl-butyl-benzaldehyde, or its homologue, methyl butyl benzaldehyde, or its substitution product, methylbutyl-methoxyl-benzaldehyde. For instance, one part of dimethyl-butyl-benzaldehyde is put into 10 parts of nitric acid (99 to 100 per cent.) and kept cool; after standing for some time, the mixture is poured on ice, the precipitate collected, washed, and crystallised from alcohol, from which it separates in white plates melting at 112°—115° C. In composition it is dinitro-dimethyl-butyl-benzaldehyde; any of the above-mentioned aldehydes may be substituted for the dimethyl-butyl-benzaldehyde in this process.—J. O. B.

Artificial Musk, Improved Process for Manufacture of. Adolf Blaile, Zürich, Switzerland. Eng. Pat. 24,568, Nov. 3, 1896.

By dissolving terpenes in alcohol, pouring the solution into sulphuric acid, and then the latter mixture into nitric acid, and finally the whole into water, the crude musk-smelling substance is obtained as a brown amorphous mass.

100 parts by weight of terpinol are mixed with an equal weight of ethylic alcohol of 96 per cent. strength, to form a clear solution, which is poured into 1,000 parts of sulphuric acid (66° B.), with constant stirring and cooling, the mixture being stirred for 6 hours. The resulting compound is then poured into 1,000 parts of concentrated nitric acid, free from chlorine, with constant stirring, and the temperature not allowed to rise above 8° C. The mixture is then warmed to about 70° C. for 4 hours, then cooled, and poured into 20,000 parts of water, with vigorous stirring. The new perfume, in the crude state, then forms a brown deposit, which is collected. This may be further purified by washing with hot water until free from acid, then extracting with petroleum ether, or some other suitable solvent, until a yellow crystalline body is obtained, having a delicate musk-like odour. Instead of terpinol, eucalyptus or turpentine oils, or oils from fossil resins, such as oil of amber, retene oil, or other bodies having similar chemical properties, may be employed.—J. O. B.

Acetanilide or Acetoluides, Process for Producing. J. Corry Fell, London. From W. J. Matheson and Co., New York City, U.S.A. Eng. Pat. 6220, March 9, 1897.

By treating aniline or the toluidines, under pressure, with more dilute acetic acid than the glacial acid hitherto employed—*viz.*, containing 50 or 33 per cent. of real acid—or even by using crude pyroligneous acid, acetanilide or the acetoluides are obtained. When 1 part of aniline with $1\frac{1}{2}$ to $1\frac{3}{4}$ of 50 per cent. acetic acid are heated together under pressure to between 150° to 160° C. until acetylisation is complete, the resulting liquid, on cooling, solidifies into a mass of crude acetanilide, which may be purified by fractional distillation or by washing, and subsequent crystallisation. The same method and reactions apply to the toluidines. The quantity of acid employed should be in excess of the theoretical equivalents.—J. O. B.

Preserving Substances [*Vegetable, containing Aromatic Oils*], *Improved Process for.* H. Humbser, Fürth, Bavaria. Eng. Pat. 6064, March 8, 1897.

The process is intended for preserving vegetable substances, such as hops, which contain aromatic oils readily volatilising on heating. Air, dried by forcing it into a very cold chamber, where it deposits its moisture, is then warmed, so as to enable it to take up moisture again, and conveyed through storage chambers containing the substance to be preserved, where it again takes up moisture. The warming of the air can be effected by means of a stove pipe, or it can be passed through a coil of thin pipe exposed to the open air.—N. H. J. M.

XXI.—PHOTOGRAPHY.

Photography and Photo-Mechanical Processes, Recent Progress in. J. M. Eder and E. Valenta. Dingl. Polyt. J. 1897, **304**, 67 and 92.

Kogelmann has lately stated that the effect produced on bromide plates by ordinary white light may be removed to a certain extent by exposure to the red rays; whilst among new sensitisers for this end of the spectrum, Eberhardt recommends that after immersion in 1 per cent. ammonia for 1 minute, the plates should be dipped for 3 minutes in a bath containing 3 c.c. of a 0·2 per cent. solution of Beyer's Nigrosin B, 1 c.c. of ammonia, and 100 c.c. of water.

Teape prepares a collodio-chloride emulsion for positives by means of zinc chloride and silver nitrate, the developer being quinol. In the manufacture of bromide emulsions, Scheers makes use of urea in order to regulate the action of the ammonia and to hasten the ripening of the emulsion in the cold. Colby claims the addition of acetyl chloride, which increases the sensitiveness. Vogel holds that the character of the gelatin itself notably affects the keeping qualities of the plates. He tests it by dissolving a sample in 10 parts of water, heating the liquid with a 10 per cent. solution of ammoniacal silver nitrate; if a yellow or brown colour is produced, the gelatin is not adapted for the preparation of emulsions by the ammonia process.

Hauff states that the presence of thiosulphate in the metol developer increases its strength and makes its action more like that of pyrogallol. He gives the following formula:—(A.) Metol, 15 parts; sodium sulphite, 150; water, 1,000. (B.) Sodium carbonate, 350; "hypo," 1; water, 1,000. 20 parts of A., 10 of B., and 30 of water are used for development. Neuhauss advises a similar method of procedure in the case of amidol.

Anthion (potassium persulphate; this Journal, 1896, 50), which is frequently employed as a "hypo-eliminator," in Olbrich's hands produces markings on the film.

To remove the gelatin film from its support—an operation frequently involved in photo-mechanical work—Valenta suggests that the plate should be immersed for 10 minutes in a bath of 1 part of formalin diluted with 5 or 10 parts of water, then dried, levelled, and coated with gelatin, 75 grms.; water, 500; and glycerin, 10 (previously filtered through flannel). After being dried again, it is soaked for 10 or 15 minutes in a mixture of glycerin, 50 parts; alcohol, 50; and water, 1,000, when the film can be readily stripped off the glass. A clean glass plate is then levelled, coated with 5 per cent. plain collodion, and the solvents allowed to evaporate; the moist negative film is "squeegeed" on to it, gelatin side down, and after the whole has become dry, it may be stripped without danger of causing any distortion.

G. and H. Beneke have introduced a matt collodion paper for positives which, it is claimed, will not crack or peel off, and will lend itself well to retouching. It is made by coating baryta paper with an emulsion containing only a small amount of collodion. Two gelatin papers have been put on the market, called "gelatoid" and "universal" paper respectively, having the film rendered insoluble by treatment with formalin, so that they may be worked, like the collodion papers, in warm baths.

Among new toning agents for silver bromide, solutions are being sold of uranium nitrate, potassium ferricyanide, and ferric chloride in about 1 per cent. strengths, which, on being mixed together in varying proportions, yield brown, red, green, and blue pictures; the process is known as "virage panchromatic." For chloride prints, Barnstaple recommends a small addition of potassium platinous chloride to the ordinary gold and sulphocyanide bath.

According to Kuhn, by the partial or total substitution of potassium palladious chloride for the corresponding platinum compound in "platinotype," brown or sepia prints may be obtained, which gain considerably in depth and brilliancy during fixing in weak hydrochloric acid. Bank has described a process for the production of positives in platinum plus silver, as follows:—Thick Rives paper is painted with a solution of 20 grms. of ammonio-ferric oxalate and 5 c.c. of 2 per cent. potassium platinous chloride in 100 c.c. of water. When dry, it is exposed under a negative and developed in silver nitrate, 1 grm.; citric acid, 1: ammonio-ferric citrate, 2·5; oxalic acid, 0·5; 4 per cent. potassium bichromate, 10 drops; water, 250 c.c. After washing, it is fixed in ammonia, 4 grms.; sodium citrate, 4 grms.; water, 200 c.c. The prints can be finally toned in a platinum bath if desired.

Arndt and Troost have introduced a paper giving a perpermanent brown image on a white ground which only requires water to finish it. 80 to 100 grms. of ammonio-ferric citrate, 12 to 20 of silver nitrate, 15 to 20 of tartaric acid, and (in another portion) 10 to 15 grms. of gelatin are dissolved in water, and the whole is made up to 1 litre. Paper is coated and dried as usual. Valenta finds that Merck's green ammonio-ferric citrate is preferable to the ordinary brown salt, for it is eight times more sensitive, the coating fluid keeps better, and the prepared paper itself keeps quite as well. His formula is: (A.) green double citrate, 12·5 grms.; water, 50 c.c.: (B.) potassium ferricyanide, 4·5 grms.; water, 50 c.c.

Fisch has worked out Itterheim's "negrotype" again. A solution of 25 parts of gum arabic, 1 of potassium bichromate, and 1 of alcohol in 100 of water is spread on paper, dried, and exposed under a negative for $\frac{1}{4}$ to 4 minutes. The print is dipped in water to remove the unaltered portion of the coating, then dried, and a varnish of 5 parts of bleached shellac, 1 of Frankfort (vine) black, and 100 of alcohol, applied with a brush. Finally, it is washed in 2 per cent. sulphuric acid, which dissolves the rest of the bichromated gum, leaving behind the pigment that has settled on the exposed parts or shadows of the picture.

For the intensification of carbon transparencies, Burton makes use of a 0·5 per cent. solution of silver nitrate, followed by a pyro-silver intensifier. A permanent warm brown tone is thus produced.

In the domain of collotype, Classen claims the application of a substratum of rosin soap to the plate before coating with the gelatin solution, thus obtaining a specially strong film capable of yielding an unusually large number of impressions. The chief advance in this direction is in the employment of aluminium to replace the glass supports. The use of the metal not only removes all danger of breakage, but makes the process easier to work in a bad or artificial light, as the condition of the film when in the press can be more readily seen against the white metallic background. Albert cleans the aluminium plates when new by rubbing them with 1 : 3 ammonia and drying them with a cloth. Used plates are freed from gelatin by immersion in 1 : 30 sulphuric acid and then cleaned as before. During the drying process the plates carrying the sensitive film should be supported horizontally on glass, and the exposure to light should not be carried so far as is necessary with glass supports. While being printed from, they are moistened ("etched") with glycerin and water only; but if the high lights take the ink at all, a little ammonia or preferably acid, may be added to the "etching" liquid.

Moses has patented an etching process for gelatin plates, which is particularly suited for plans, &c. A gelatin film is cemented to a ground-glass support by means of some adhesive insoluble in water. A ground, consisting of wax, asphaltum, mastic, and turpentine is next applied, and after drying, the design is drawn with the needle. The plate is then brushed over with a dark water-colour (*e.g.*, Indian ink) rubbed down in a bichromate solution, and the glass side is exposed to light so as to render the gelatin insoluble where the ground has been perforated. The face is washed with a damp sponge, the ground removed by turpentine, and the whole treated with water containing glycerin, ammonia, and potassium nitrate, in order to swell the protected portions of the film, and to leave the lines sunk below the surface. It can then be printed from as if it were an ordinary collotype plate.

For producing negatives suitable for photo-lithography, very slow grainless plates are requisite. These may be obtained from bromide plates of commerce by treating them with a 0·1 per cent. solution of bichromate before use. As an etching ground for zinc plates Pope recommends a mixture of asphaltum, 2 parts; rosin, 2 parts; and dragon's-blood, 1 part. This is dusted on, and finally melted as usual. In the preparation of relief blocks by the gelatin process, Vallot prefers to employ "meta-gelatin," which he makes by boiling 500 grms. of glue in 800 c.c. of water and 125 c.c. of ammonia for an hour, the amount of liquid evaporated being constantly replaced. To coat copper plates he uses 10 grms. of meta-gelatin, 3 of ammonium bichromate, and 100 c.c. of water; or 5 grms. of meta-gelatin, 50 of albumin, 3 of bichromate, and 50 c.c. of water. The latter formula allows of a stronger heating of the plate, but with the former the temperature must not exceed 334° C. Steel plates may be etched by placing them in contact with zinc in a mixture of the nitrates of silver and mercury, prepared by dissolving 30 grms. of silver in 100 of nitric acid, and 30 grms. of mercury in 150 c.c. of the same, adding 150 c.c. of water to each, then mixing the two together, and diluting to a convenient strength.

In working the three-colour process, Elsden prepares colour screens in the subjoined manner:—For violet, strong solution of copper chloride, 7 c.c.; water, 17 c.c.; filtered and mixed with methyl violet B (concentrated solution), 3 c.c.; magenta S (ditto), 5 c.c. For orange-red, strong cobalt chloride, 15 c.c.; water, 35; 5 per cent. ammonium bichromate, 25; ammonia, 2—3 c.c., filtered. For green, a solution of nickel sulphate. These are placed in troughs with strictly parallel sides of about $\frac{1}{8}$-inch diameter.

—F. H. L.

Hydroxylamine Sulphate, Economic Preparation of. E. Divers and T. Haga. J. Coll. Science, Imp. Univ. of Japan, 1897, **9**, [2], 291.

See under XX., *page* 553.

XXII.—EXPLOSIVES, MATCHES, Etc.

PATENTS.

Explosives [Ammonium Nitrate], Impts. in the Manufacture of. G. G. André, Glenlean, Argyllshire; and C. H. Curtis, London. Eng. Pat. 11,842, June 1, 1896.

A SAFETY, or flameless, explosive consisting of ammonium nitrate, wood meal, and starch, or a certain proportion of potassium nitrate may be substituted for part of the ammonium nitrate. The following proportions are given: (*a*) wood meal, 4 per cent.; starch, 3·5 per cent.; ammonium nitrate, 92·5 per cent.; (*b*) wood meal, 4 per cent.; starch, 3·5 per cent.; ammonium nitrate, 90 per cent.; potassium nitrate, 2·5 per cent.—R. B. P.

Explosives [Ammonium Nitrate], Improved Manufacture of. G. Beneké, Southport, Lancashire. Eng. Pat. 13,922, June 24, 1896.

IN order to ensure the more thorough detonation of explosives compounded of ammonium or another inorganic nitrate and resins, the addition of bichromate of potassium, sodium, or ammonium, but preferably the potassium salt, is claimed. The necessity of a thoroughly even and intimate mixture of the ingredients is pointed out. The process of manufacture is briefly described, and the following given as the composition: ammonium nitrate, $92\frac{1}{2}$ parts; resin, 5 parts; and potassium bichromate, $2\frac{1}{2}$ parts.—R. B. P.

Explosives [Smokeless Powder], Impts. in. A. F. Hargreaves, Roslin, Scotland. Eng. Pat. 15,351, July 10, 1896.

SKEINS, or hanks, of cotton thread are nitrated, and then thoroughly washed and boiled until stable.

Whilst still moist, a suitable number of threads are twisted into a rope, which is then passed through a hot

water-bath in which any, or all, of the following substances have been dissolved :—Nitrates of barium, potassium, ammonium, and sodium, and a little starch or gum.

After passing through the bath, the "rope" is allowed to cool, and is then squeezed through rollers to remove excess of the solution. A number, preferably three, of these ropes are twisted together to form a still larger one, which, whilst still moist, is passed through a bath of acetone, or acetic ether, diluted with ether-alcohol, ether, benzene, or naphtha. Preferably, the bath consists of 1 part of acetone to 3 parts of benzene.

This stiffens the rope without destroying its fibrous nature, and causes the individual strands to adhere. Whilst still wet and in an uninflammable state, the "rope" is cut into suitable lengths, which, after drying, constitute powder pellets.—R. B. P.

Explosives [Smokeless Powder], Impts. in the Manufacture of. F. W. Jones, Barwick, Herts. Eng. Pat. 1154, Jan. 15, 1897.

This invention aims at regulating the densities and rates of combustion of smokeless powders, particularly that kind of which nitro-cellulose gelatinised by a suitable solvent, is a main ingredient.

The first process consists in treating the granulated powder in a "swelling-bath," in which it is soaked in an aqueous or alcoholic solution of a solvent for nitro-cellulose, such as ether, acetone, ketones, and acetic ethers of the paraffins, for one or more days, either with or without agitation and the application of heat.

The solvent is then extracted by washing or by passing through steam, &c. This bath causes the powder to swell, the increase in bulk being greater, the greater the strength of the solution, the higher the temperature, or the longer the action is continued.

The powder, when freed from the solvent, is then dried for use.

In the case of powders in which one, or more, of the solid ingredients melts below the temperature of boiling water, and, when melted, has a solvent action on the base, such as the di- or tri-nitro derivatives of benzene, toluene, &c. the grains are kept in a bath of water at a temperature above the melting point of the above-mentioned ingredient for one or more hours (they may or may not have been previously treated in the swelling-bath). The effect of this treatment is to make the rate of combustion vary at different points in each grain, so that "the burning surfaces increase as the combustion progresses," thus enabling low initial pressures to be obtained. The following are given as three general compositions between the limits of which, powders for every kind of firearm can be made by these processes :—

1st. Nitro-cellulose, 100 per cent. to 75 per cent.

Slowing material, such as the di- and tri-nitro-benzene, toluene, and zylene, 0 per cent. to 25 per cent.

2nd. Nitro-cellulose, 90 per cent. to 60 per cent.; nitro-glycerin, 10 per cent. to 40 per cent.

3rd. Nitro-cellulose, 90 per cent. to 50 per cent.; nitro-glycerin, 10 per cent. to 25 per cent.; slowing material as in 1st, 0 per cent. to 25 per cent.—R. B. P.

XXIII.—ANALYTICAL CHEMISTRY.

INORGANIC CHEMISTRY.—QUALITATIVE.

Carbonic Oxide, A Reaction of. A. Mermet. Comptes Rend. 1897, **124**, 621—624.

Carbonic oxide was found to decolorise weak potassium permanganate solution acidified with nitric acid. The decoloration was hastened by silver nitrate. For the detection of 0·02 per cent. and upwards of carbonic oxide in air, two solutions (A. and B.) were prepared. A. was made by dissolving 2—3 grms. of silver nitrate in a litre of distilled water; B. by boiling a litre of water with a few drops of pure nitric acid and adding permanganate until the colour persists—indicating the destruction of organic matter—and then, after cooling, adding 1 grm. of permanganate crystals and 50 c.c. of nitric acid. The reagent was prepared freshly, as required for use, by taking 20 c.c. of A., 1 c.c. of B., and 1 c.c. of nitric acid, and making up to 50 c.c. with water deprived of organic matter. 25 c.c. of this reagent was added to the suspected air collected by displacement of water in a stoppered flask; after standing for 24 hours screened from strong light, decolorisation was found to have ensued if carbonic oxide was present. A blank experiment was made simultaneously with good air. The decolorisation took place more rapidly the greater the amount of carbonic oxide. Air collected near an anthracite stove, and near a chimney subject to down draught, were found to be contaminated therewith.

Boussingault had previously found carbonic oxide accompanying the oxygen given off in daylight by plants. The author failed to detect it in the air of a greenhouse. It seemed possible that the quantities of carbonic oxide continually cast into the air might be partially oxidised to oxalic acid, and, to test this supposition, rain water was collected in an open field, concentrated, and examined. Traces of calcium oxalate were found. This matter is being further investigated.—J. A. B.

Telluride Gold Ores. R. Pearce. Eng. and Mining J., April 17, 1897, 376.

For the detection of tellurium, when present in an unoxidised form, as a telluride, in gold-bearing ores, a sample of the fine pulp is treated with strong nitric acid in a porcelain dish and heat applied until the material is perfectly oxidised. Strong sulphuric acid is then added, and heat applied until the excess of nitric acid is expelled, or until copious fumes of sulphuric acid are evolved. When cold, hydrochloric acid and water are added in about equal proportions, the solution is filtered and treated with stannous chloride precisely as in the case of oxidised tellurium ores, when a carmine tint is produced.

The production of the coloration by gently heating a minute fragment of the mineral with a drop of concentrated sulphuric acid is of little or no value, as the other minerals commonly associated with tellurium in gold-bearing ores, interfere with or conceal this reaction.—A. S.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Potash, Soluble; Estimation of. A. Mercier. Bull. Assoc. Belge des Chimistes **10**, [11], 403—405.

In place of the sodium formate employed in the old method of Correnwinder and Contamine, for the reduction of potassium platinum chloride, the author proposes to use mercurous chloride, with which the reduction commences at 60° C., and is complete at boiling temperature.

5 or 10 grms. of substance, finely ground under water, are digested cold for one hour in a 500 c.c. flask filled up to the mark with water, 25 c.c. of the clear liquid being then evaporated to dryness on the water-bath with 1 c.c. of hydrochloric acid and heated gently to drive off any ammonium salts and organic matter. The residue is taken up with water acidified with hydrochloric acid, filtered if necessary, and then evaporated to syrup with 10 c.c. of a 10 per cent. solution of $PtCl_4$. When cold the precipitate is extracted with 50 c.c. of 85° alcohol + 5 c.c. of ether and washed with the same liquid until all the $PtCl_4$ is removed, the remainder being dissolved in boiling water and the platinum precipitated as a heavy grey powder by the gradual addition of about 2 grms. of Hg_2Cl_2 at boiling temperature, 1 or 2 c.c. of hydrochloric acid being added after the precipitate is settled, and the whole again heated to boiling point. When somewhat cooled the platinum is filtered off, washed with boiling water until free from chlorine, and calcined without drying the filter, the weight of metal multiplied by 0·4835 giving the amount of potassium oxide present in the substance in the soluble form.—C. S.

Steel, Hardening-Carbon and Carbide-Carbon in; Determination of. H. Jüptner v. Jonstorff. Iron and Steel Inst. May 1897; Ironmonger 1897, **79**, 351—356.

Hardening carbon has not hitherto been determined directly, but has been estimated (when necessary) by

deducting graphitic and graphitic temper carbon, and the ordinary carbide carbon from the total carbon, thus accumulating all the errors of the whole process in this one determination. The author has now investigated the colorimetric and gravimetric tests for carbon in steel and publishes in detail the results of his experiments. To determine the hardening- and carbide-carbon contents in steel he proceeds as follows:—0·2 grm. of the steel is treated with 10 c.c. of dilute nitric acid in the cold (2 vols. of concentrated acid mixed with 3 vols. of water), the vessel being allowed to stand in cold water. The solution is well and repeatedly shaken, and when no further evolution of gas is observed, it is well mixed and filtered through a dry filter without subsequent washing. The filter should first be hardened with nitric acid. 2 c.c. of the filtrate are used for the determination of the hardening carbon by direct colorimetric comparison with the ordinary colour standards. In this way (working in the cold) the comparison is accurate, but when the hardening carbon is less than 0·15 per cent. the colour of the solution is somewhat greenish and the results appear to be a little too low. The error may be rectified by multiplying by 1·3 the results obtained, when the solution is at all greenish. Another 2 c.c. of the filtrate are set aside. Meanwhile 0·04 grm. of the steel is weighed, treated with the dilute nitric acid, and transferred to a water-bath at 80° C., simultaneously with the 2 c.c. of hardening carbon test that had been placed aside. When complete solution is effected, both series of solutions are compared with the same standard, and from the compared volume of the solution of the total carbon, is subtracted the compared volume of the hardening-carbon solution which had been heated to 80° C. The difference gives the volume corresponding to the carbide carbon, from which the percentage contents can be readily calculated. The author uses unalterable standards made by mixing slightly acidulated solutions of Fe_2Cl_6 and $CoCl_2$.

—W. G. M.

Copper, Determination of, as Sub-sulphide. R. Wegscheider. Monatsh. f. Chem., **18**, 44–47.

A reply to criticisms of Murmann's on a former paper of the author's (this Journal, 1894, 67, and 1897, 359). Murmann quotes the author as saying that the sub-sulphide method always gives figures too low by about 0·3 per cent. The author points out that his results were sometimes above, sometimes below the truth, and remarks that his work was performed on such small quantities that the manipulative losses, even with careful work, would always account for the low results. The same losses, on the amounts with which Murmann worked, would not have meant more than 0·1 per cent.

Murmann again quotes the author as directing that the crucible should be heated merely with the tip of the Bunsen flame; the real directions were to heat to dull redness, and the author pointed out that a rise of temperature above this would do no appreciable injury if not too long continued, for the reduction of the sub-sulphide by hydrogen is a very slow process. Murmann, lastly, makes the author say that the cause of too high figures when the ignition of the cupric sulphide is carried out in hydrogen sulphide, is *merely* that the cupric sulphide is then not completely decomposed, and points out that under these circumstances it is in part formed from the cuprous sulphide and hydrogen sulphide. The word "merely," which alters the sense of the statement, was not in the author's paper. The decomposition of cupric sulphide in a stream of hydrogen sulphide is so rapid that its incompleteness is only to be accounted for by the existence of an inverse action and the attainment of equilibrium between the two; but the author did not think it necessary in an analytical paper to draw attention to this, and contented himself with mentioning the ultimate result.—J. T. D.

Acetylene as Quantitative Reagent. [*Estimation and Separation of Copper.*] H. G. Söderbaum. Ber. **30**, 902–904.

It was shown recently (Ber. **30**, 760—814) that acetylene precipitates the metal completely from an ammoniacal copper solution as black cupric acetylide, and the mother-liquor, after acidulating, does not give the copper reaction with potassium ferrocyanide. For analysis, the salt is dissolved in 100 to 200 times its weight of water, a few c.c. of ammonia are added, the solution is then warmed on the water-bath, and acetylene is passed through until the solution is saturated. The estimation is carried out preferably in an Erlenmeyer flask, and, if closed, the precipitate may be left an indefinite time without undergoing any change. On standing in an open vessel, however, a part of the copper again passes into solution. After filtering and washing the acetylide, it is digested for half an hour with hot dilute nitric acid, filtered from a carbonaceous residue, the filtrate evaporated to dryness and converted by careful heating into cupric oxide, which is then weighed together with the ashed filter-paper. The method is an accurate one; 0·4775 grm. of $CuSO_4 5H_2O$ containing 0·1214 grm. = 25·42 per cent. of Cu, gave 0·1516 grm. of CuO = 0·1211 grm. = 25·35 per cent. of Cu The presence of alkali salts does not affect the precipitation, although an excess of ammonium salts does. The filtrate from the acetylide can be utilised for determining the acid, thus: 0·5105 grm. of $CuCl_2 2H_2O$ gave 0·8565 grm. of AgCl = 41·5 per cent. (calculated 41·6 per cent.). The method has also been employed for separating metals, and in the case of a mixture of copper and zinc, accurate results have been obtained, care being taken to have sulphurous acid present, when the copper is precipitated at first as a brownish-red precipitate, which turns black, and contains Cu : C = about 2:3. The zinc is determined in the filtrate either gravimetrically or volumetrically.—T. A. L.

ORGANIC CHEMISTRY.—QUALITATIVE.

Benzene, A Means of Distinguishing from Petroleum Spirit. A. Gawalowski. Chem. Zeit. Rep. 1897, **21**, 100.

Picric acid easily dissolves in benzene with an intense yellow colour, whilst in petroleum spirit it is only slightly soluble with a nearly colourless solution. If an equal volume of petroleum spirit be added to a concentrated solution of picric acid in benzene, a crystalline precipitate of picric acid separates out.—A. S.

Nitramines and Nitrosamines [*Liebermann's Reaction, &c.*], *Characterisation of Aromatic.* J. Pinnow. Ber. **30**, 833—843.

With a view of distinguishing between aromatic nitramines and nitrosamines, the author quotes certain reactions for this purpose, some of which can even be employed for separations, but no absolutely general method has been obtained. He employs principally Liebermann's nitroso reaction, for which purpose the substance is dissolved in phenol, two or three drops of concentrated sulphuric acid added, and the mixture is warmed for 5—10 seconds on the water-bath. Under these conditions, toluenediazo acid ($C_6H_4.CH_3.NHNO_2$), trinitrotolyl- and trinitrophenylmethylnitramine do not give the nitroso reaction, although the latter gives a fine red coloration, but this is probably due to the action of the potash. Tolylmethyl nitramine gives the nitroso reaction in the cold, but the coloration has a greenish shade and is weak. Under all conditions, *p*-nitrophenylnitramine and its methyl ether give bluish-green colorations, whilst the same is the case with o-nitrotolylmethylnitramine in the cold, and with *m*-nitrotolylnitramine and its methyl ether on warming. These two latter and *o*-nitrotolylnitramine give dirty green to olive-green colorations, whilst, on warming, *o*-nitrotolylnitramine and its methyl ether give greyish-blue, or, on longer heating, violet-blue shades. Tetranitrodimethyldinitramidodiphenylmethane requires to be warmed with phenol in order to dissolve it, and, according to the time of heating, gives bluish-green to pure blue colorations. The author concludes from these results that it would be possible in other cases to distinguish by means of this reaction between nitrosamines and purely aromatic nitramines, since the coloration, if any, obtained either in the cold or on heating, differs from the pure blue, given by the nitrosamines. It is further remarked that aromatic nitramines, under the action of mineral acids, are converted

into *p*- or *o*- nitrated anilines, whereas by the action of concentrated hydrochloric acid or of aniline hydrochloride on nitrosamines, the nitroso group is separated.—T. A. L.

[Brewing] Saccharification of the Mash, Testing the. J. Fuchs. Zeits. f. d. ges. Brauwesen, **20**, [15], 203—204.

To prevent the iodine reaction from being masked by the brown and red colorations produced by dextrin, albuminoids, and particles of grains present in the mash goods, the following method is adopted in the laboratory of the Munich Scientific Station for Brewing:—

Small dry plates of gypsum, 1 to 2 cm. wide, 10 to 12 cm. long, and 0·4 to 0·8 cm. thick, are used, on to which is allowed to fall one drop of the wort or mash to be tested. The liquid is rapidly absorbed, leaving the non-liquefied portions on the surface of the plate, so that if a little iodine solution be applied to the same spot, a blue coloration will be produced so long as any of the starch remains unconverted. The iodine seems to be decolorised on absorption by the gypsum, and thus the end point of conversion is more sharply defined than when the test is performed in tubes or on a porcelain plate. Moreover, the various tests for each mash can be made on the one slab, which, the intervals between each test being inscribed thereon, can be kept as a permanent record.—C. S.

Colouring of Wines, Distinguishing Coal-Tar Colours from Caramel. A. d'Aguiar and W. da Silva. Bull. Soc. Chim. 1897, **17**, 443—449.

It has been stated by Malaghaës (Comptes Rend. **123**, 896; this Journal, 1897, 156) that it is not possible, by the tests commonly in use, to distinguish caramel from certain coal-tar colours when used for the purpose of colouring white wines and brandies. The authors have therefore made experiments in this respect with a number of yellow and brown coal-tar colouring matters. On extraction with amyl alcohol, the extract is less coloured in the case of caramel than in that of any coal-tar colour, except Naphthol Yellow S, and the latter colour is readily distinguished from caramel by dyeing silk threads in the solutions. When the amyl alcohol extracts are evaporated to dryness and treated with sulphuric acid, caramel yields a brownish-black colour, differing from all the rest. With other tests the authors obtain equally distinctive indications, and they conclude that the present methods of testing are entirely satisfactory. —R. B. B.

Lavender Oil, Detection of Adulteration. Schimmel's Rep., April 1897, 24.

An ingenious fraud has lately been detected, demonstrating that certain unscrupulous dealers keep themselves well informed in the advance of analytical processes for the valuation of essential oils, and endeavour to meet the tests by more than ordinary methods. Two samples of lavender oil were sent for valuation, which gave the following data:—

No.	Specific Gravity.	Optical Rotation.	Per Cent. of Ester.
1	0·900	6°·53′	48·5
2	0·905	3°·50′	47·5

The facts that the specific gravity of these oils was abnormally high and that during the process of saponification a solid body, never before observed, separated from the alcoholic potash solution, aroused suspicion. A larger quantity was therefore saponified with aqueous potash solution, since ethyl or methyl esters would escape detection when alcoholic potash is used. On separating the alkaline solution and distilling, several c.c. of ethylic alcohol were obtained. In the residue, the presence of an organic acid was detected; this was isolated, purified, and proved to be succinic acid. Since pure lavender oil does not contain ethyl succinate, it is evident that this ester had been added for the purpose of giving a fictitious value to the oil, as shown by its ester number. In the present instance, about 8 per cent. of ethyl succinate had been added, which would require the same amount of potash for its saponification as about 18 per cent. of linalyl acetate. In order to rapidly detect the presence of the esters of succinic, oxalic, or corresponding acids in lavender oil, the following simple test is available:—About 2 grms. of the oil are saponified, and the portion insoluble in water washed out with ether, the aqueous residue is neutralised with acid and diluted up to 50 c.c.; to this, 10 c.c. of cold saturated barium chloride solution are added, the liquid warmed on the water-bath for two hours, and then cooled. The formation of any crystalline precipitate is indicative of adulteration, since the barium salts of acetic and butyric acids, the normal organic acids of lavender oil, are soluble.—J. O. B.

Star Anise Oil [Testing for Kerosene]. Schimmel's Rep., April 1897, 37.

Although much oil, which had an abnormally low congealing point, has been met with, the admixture of kerosene has not been discovered. To test the question, 30 kilos. of abnormal oil, congealing at 10°·25 and 10° C., were fractionated, but no kerosene was found in the first fraction of 300 c.c. It is possible that the abnormally low congealing point may be due to a large proportion of methylchavicol present. The experimental admixture of kerosene with star anise oil shows that its presence does not greatly modify the congealing point, but markedly lowers the specific gravity and affects the solubility in alcohol. The gravity of the pure oil should lie between 0·980 and 0·990 at 15° C., and it should give a perfectly clear solution with 3 parts of 90 per cent. alcohol.—J. O. B.

ORGANIC CHEMISTRY.—QUANTITATIVE.

Alizarin, Analytical Methods employed in the Manufacture of. A. G. Perkin. J. Soc. Dyers and Colourists, 1897, 81—87.

The Meister, Lucius, and Brüning modification of Luck's method, of 1876, is still universally employed. (See Lunge's Coal-Tar and Ammonia, 1887, 318—327.)

The nature of the impurities accompanying the anthraquinone, which require the use of fuming sulphuric acid for their removal, has not yet been properly explained. They are said to consist of quinones, which though unattacked by chromic acid like anthraquinone, differ therefrom in yielding sulphonic acids at 100°. Anthraquinone before treatment with sulphuric acid usually contains chromium compounds requiring the use of fuming sulphuric acid for their decomposition.

Bassett's addition to the present test, based upon a second oxidation with a mixture of chromic and nitric acids (this Journal, 1896, 385) should give a very pure product. By this means all methylanthraquinone and the chromium salts of impurities, are destroyed.

Commercial anthracenes are divided into two classes, known as A and B quality. The nature of the impurities present in the latter is not thoroughly understood, though of these, β-methylanthracene is frequently one. In the usual process this yields first its quinone and finally methylalizarins, which dye shades of a duller character and less fast than the true alizarins.

Paraffin, if present in anthracene, may be detected by heating the sample with sulphuric acid at 100°.

For the analysis of crude anthraquinone two distinct tests are necessary. (1.) Determination of the anthraquinone it is capable of yielding. (2.) Determination of the anthraquinone present in the crude product. The former operation is simply an anthracene test by Luck's method, whilst in carrying out the second test, 10 grms. of the finely powdered sample are treated with 10 to 15 grms. of sulphuric acid at 100° for one hour. The mass is then boiled out in water and the anthraquinone filtered, washed, dried, and weighed. To ascertain its purity, 1 grm. is treated with 10 grms. of sulphuric acid containing 10 per cent. of sulphuric anhydride at 100°, for one hour. It is then placed in a damp atmosphere for 12 hours and allowed to crystallise. After the addition of water the crystals are collected, washed with water, dilute alkali, again with water, dried and weighed. From the

difference between the result of this analysis and that of Luck's method, the percentage of unoxidised anthracene can be easily calculated.

The method employed for the analysis of purified anthraquinone, consists in heating 1 grm. with 10 grms. of sulphuric acid containing 10 per cent. of sulphuric anhydride, the course of procedure being identical with the second portion of the anthracene test. The grey or green tinge of anthraquinone purified with sulphuric acid, is due to the presence of a dark-coloured amorphous product, which is insoluble in the usual solvents and contains nitrogen, originating possibly from the carbazol or other nitrogenous impurities existing in crude anthracene. Methylanthraquinone being moderately soluble in boiling dilute sulphuric acid, especially if this contains other sulphonic acids in solution, a purer anthraquinone is obtained by filtering the purified product from the acid solution of sulphonated hydrocarbons at an elevated temperature.

For the final purification of anthraquinone it is either re-purified with sulphuric acid at 100°, or sublimed by means of superheated steam, or recrystallised from some suitable solvent. The estimation of pure anthraquinone is carried out in the same way as that employed with the purified product. Frequently, however, its valuation in this manner is accompanied by a second determination, in which the full anthracene test is used.

Sodium Anthraquinonemonosulphonate is produced on a large scale in an almost chemically pure condition, and no analysis is required beyond a determination of the moisture present. The filtrate and washings from this salt consist chiefly of a mixture of the two disulphonic acids. If neutralised and converted into colour, no further valuation is required, for knowing the weight of anthraquinone originally employed in the sulphonation, and also the anthraquinone value of the sodium mono salt and unattacked anthraquinone, this value can be at once determined by difference. The anthraquinone unattacked during the sulphonating operation, is mostly weighed and sampled in the wet condition, the amount of moisture being then determined. For its analysis 1 grm. of the dried sample is dissolved in glacial acetic acid and the solution treated with chromic acid (one-half the usual quantity) as in the first portion of the anthracene test.

Sodium Anthraquinone-disulphonates.—If the resulting solution of the α- and β-disulphonic acids be at once neutralised and converted into colour, no analysis is necessary, for as a rule the whole of the anthraquinone is sulphonated. If, however, a separation of the acids be desired, one of the resulting sulphonates must be examined. For its analysis, the amount of moisture is first determined and the dried residue subsequently incinerated with the aid of ammonium nitrate or sulphuric acid. The loss by ignition multiplied by $1 \cdot 526 \times 100$ and divided by the weight of dry substance, gives the percentage of the pure salt in the dry substance.

In the process of fusing the sodium salts of anthraquinonesulphonic acids, samples of the melt are drawn off at stated periods, weighed portions being dissolved in a measured quantity of boiling water and neutralised with acid. When considered essential, the precipitated colour can be roughly washed, dried, and weighed, for the weight of the main melt being approximately known, a fair indication of the progress of the reaction can be thus obtained. With experience, however, this can be judged from the appearance of the precipitate, and the colour of the filtrate in an acid or alkaline condition, taking into consideration also the length of time that the operation has been in progress. Special tests can be employed for the detection of metahydroxyanthraquinone and anthra- or iso-anthraflavic acids in the melts.

For the analysis of alizarin pastes as obtained from filter presses, the percentage of moisture is determined by evaporating a weighed sample in a flat platinum dish at 100°. After weighing, the inorganic matter present is determined by incineration. Usually the ash, which should not exceed 0·2 per cent. in a 20 per cent. paste, consists of either sodium or calcium salts, but sometimes lead and iron are also present, though contamination of alizarin with iron should be carefully avoided when dyeing bright shades. The tinctorial properties of the paste are then compared with those yielded by a standard sample of known strength and shade. In this operation a special five-striped cotton cloth is best employed, the mordants on which, consist of strong and weak aluminium and iron salts and a mixture of the two respectively. Of this, 10-in. lengths are most suitable. The dye vessels, preferably of glass, and of about 600 c.c. capacity, are arranged in rows in a long copper water-bath heated with gas. A portion (5 grms.) of the 20 per cent. paste under examination is suspended in a litre of water, and 50—70 c.c. of this, together with 4 c.c. of a 1 per cent. solution of calcium acetate are added to 500 c.c. of water, in the dye vessel. The temperature is gradually raised to 80° during one hour, the heat being maintained at that temperature for half an hour. After washing in cold water, the fents are well beaten with a wooden mallet and again rinsed, the treatment being continued until the loosely-adhering particles of alizarin are almost entirely removed. This is followed by two soaping operations, each lasting one hour, the first at 60° and the second at 80°, conducted in a large tinned copper vessel, which contains for each fent a solution of 0·5 grm. of curd soap in a litre of water. The patterns after washing are dried at the ordinary temperature. Swatches of Turkey-red cloth are very frequently dyed, and these, after the ordinary soaping, are heated under pressure with soap solution, to which a trace of stannous chloride has been added to confer on the tints a more brilliant appearance. The author has, in a previous communication (this Journal, 1894, 496), described a method depending on the fact, that the colours of the alkaline solutions of the alizarins are comparable to the dyed shades they produce.

m-Hydroxyanthraquinone may be detected, by treating a hot alkaline solution with baryta water and filtering the precipitate. A red coloured filtrate is obtained, which on acidification deposits a yellow precipitate.

Anthra- and Iso-anthraflavic Acids.—To a sample of the paste suspended in boiling water barium hydrate is added and the barium lake filtered off. A red coloured filtrate giving a yellow precipitate on neutralisation indicates the presence of these substances.—D. B.

Silk-Weighting, Estimation of. Leipziger Färber u. Zeugdrucker Zeit. **46**, [6], 259—260.

For the purpose of estimating the substances employed for the weighting of silk, either the foreign substances are first removed by means of solvents, or the amount of nitrogen is determined and that of silk present calculated therefrom. The latter method gives good results, but is not always easy of execution, as the presence of gelatin or Prussian blue would interfere with the results.

Black Silk is first dried at 110° C., and is then alternately boiled with sodium hydrate solution (2 parts of soda to 100 parts of water) and dilute hydrochloric acid (1 part of acid to 4 parts of water), the silk being well washed after each boiling, and dried at 110° C. The loss of weight gives the amount of weighting. Any residual metallic substances that may not have been removed by the foregoing process may be determined by incinerating the extracted silk and weighing the ash. The alkaline extract may be tested for Prussian blue with ferrous sulphate by first acidifying the solution. The acid solution would contain any iron or tin salts which are tested for by well-known methods.

The best method, however, is to ascertain the actual quantity of silk fibre by Kjeldahl's process, any gelatin or Prussian blue being first removed by boiling the sample first with ammonium carbonate, then in dilute acid, and finally washing well and drying. Pure silk contains 17·6 per cent. of nitrogen.

White or Light Coloured Silk.—The sample is boiled in water, and sugar is tested for by means of Fehling's solution. Magnesium salts or sodium sulphate are tested for in the same solution. Tin, if present, is best looked for in the ash; and for tannin, the silk is boiled in very weak hydrochloric acid and the solution tested with gelatin and ferrous sulphate. When the ash is not completely soluble in hydrochloric acid, barium sulphate or a silicate of the alkaline earths may be present. In dark-coloured silks, chromium may also be present, which is found by fusing the ash with sodium nitrate, and then testing for chromic acid.—I. S.

Fats, Note on Weighing out. C. E. Cassal. Analyst, 1897, **22**, 113.

In carrying out the Reichert process, it is essential that exactly 2·5 grms. of butter-fat should be operated upon to obtain comparative results. The author recommends the use of thin porcelain boats, open at one end, and having a small projecting handle at the other end (of shovel form). A number of these boats are ground to the same weight. When weighing out, one boat is counterpoised with another, and the fat may thus easily be weighed directly into the boat. The boat containing the fat is suspended in the neck of the distilling flask, and the fat washed down by heating the alcohol which has been placed in the flask for the process of saponification.—A. S.

Hübl's Iodine Solution, Waller's Method of Improving. R. Pelgry. Mitt. k. tech. Versuchsanst. zu Berlin, **14**, [586], 316—320.

The author confirms Waller's statement to the effect that the iodine numbers obtained by means of the modified solution of the latter (Waller adds 25 grms. of hydrochloric acid of sp. gr. 1·19 to every 500 c.c. of the iodine solution, Chem. Zeit. 1895, 1786, 1831) give results coinciding with the Hübl iodine values. The addition of hydrochloric acid as specified by Waller, renders the reagent more stable, so that even freshly prepared solutions may be used.—S. P. E.

Fat in Oilcakes, Estimation of, by Carbon Tetrachloride. A. Bilteryst. Bull. Assoc. Belge des Chimistes, **10**, [11], 406—410.

Ten grms. of substance are placed in the extractor, and heat applied for three hours, the solvent being then evaporated, and the fat weighed.

For the cold process, no extractor is required, 20 grms. of substance being shaken up in a flask with 100 c.c. of tetrachloride at intervals during 24 hours. 50 c.c. of the solution are then filtered off and evaporated, the residual fat representing the amount in 10 grms. of substance, except for the necessary correction due to the volume of the fat itself in the extract. This may be ascertained by redissolving the fat in 50 c.c. of tetrachloride and multiplying the weight of the fat by a fraction, the numerator of which is the new volume, and the denominator the 50 c.c. of solution evaporated.

The warm tetrachloride method is more expeditious than the ether method, and, at the same time, free from all risk of ignition of the solvent. Since the latter neither contains nor absorbs water, the oilcake need not be dried at 100° C. In the cold method, no fragile or expensive apparatus is required.—C. S.

Lard, Detection of Vegetable Fats in. A. Forster and R. Riechelmann. Zeits. für öffentl. Chem. 1897, 10; Pharm. Centralhalle, 1897, **38**, 151.

The authors detect the presence of vegetable fats in lard by identifying the crystalline form of phytosterin, which is a constant constituent of vegetable fats, but is never found in animal fats, nor in butter. The following method, which enables the examination to be completed in $2\frac{1}{2}$ hours, is given for the isolation of this body. 50 grms. of the lard are boiled with two successive 75 c.c. of 95—96 per cent. alcohol under a reflux condenser for about five minutes each, with thorough agitation. The mixture is thoroughly cooled, and the alcoholic solution filtered. The filtrate is heated on the water-bath with 15 c.c. of 50 per cent. soda solution, until about three-fourths of the alcohol has evaporated. The residue is evaporated nearly to dryness, transferred to a separator, and shaken out with ether. The ethereal extract is distilled to dryness, and the residue again taken up with a small portion of ether and filtered; on its evaporation, the residue is recrystallised from 95 per cent. alcohol, and is then in a state of sufficient purity for microscopical examination. Phytosterin is distinguished by the formation of acicular crystals in stellate or aggregate groups, readily distinguished from the rhomboid tablets of animal cholesterin.—J. O. B.

Cholesterin in Fats, Detection of. A. Forster and R. Riechelmann. Zeits. fur öffentl. Chem. **3**, 10; Analyst, 1897, **22**, 131.

Salkowsky's method (this Journal, 1888, 37) is said to require a considerable length of time and a comparatively large quantity of ether (500 c.c. for 10 grms. of fat); the separation of the fluids is said to be slow and often imperfect, and the saponification never to be complete. In the method of Kossel and Obermüller, the separation of the soap is, it is said, slow and incomplete, and the ethereal solution yields, on cooling, a crystallisable oil admitting of yet further saponification.

The process recommended is that given in the preceding abstract.—A. S.

Glycerin, Estimation of Small Quantities of. M. Nicloux. Bull. Soc. Chim. 1897, **17**, 455—458.

The author's process for the estimation of small quantities of alcohol by means of potassium bichromate in presence of sulphuric acid (this Journal, 1897, 473), has recently been proposed by Bordas and de Raczkowski for the estimation of glycerin (Comptes Rend. 1896, 1071; this Journal, 1897, 167). Several corrections should, however, be made in the statements of these authors. No formic acid is produced in the reaction, since this substance would be at once oxidised to carbon dioxide and water. The equation should be—

$$3(CH_2OH.CHOH.CH_2OH) + 7K_2Cr_2O_7 + 28H_2SO_4 = 7Cr_2(SO_4)_3 + 7K_2SO_4 + 9CO_2 + 40H_2O.$$

From this it will be found that 7·456 grms. of potassium bichromate correspond to 1 grm. of glycerin. A solution should be made containing 19 grms. of bichromate per litre, and 2 c.c. of this solution corresponds to 5 c.c. of glycerin (1 grm. per litre). Where the amount of glycerin is between 1 and 2 grms. per litre, the bichromate solution should be twice the above strength. The end reaction is indicated by a change of colour from bluish-green to yellowish-green with $\frac{1}{10}$th c.c. of the bichromate solution, solutions of the required tints, before and after excess of bichromate is added, being prepared beforehand for comparison.—R. B. B.

Glycerin, the Quantitative Estimation of. F. Bullnheimer. Forsch. Ber. ü. Lebnsm. u. ihre Bez. z. Hyg., &c. **4**, 12—21 and 31—45; Chem. Centr.-Bl. 1897, [1], 522—523 and 773—774.

The author gives the results of a series of experiments regarding the behaviour of glycerin towards metallic oxides, made with a view to quantitative estimation of the former.

Solvent Power.—Of the oxides that are known to be soluble in glycerin, those of lead, iron, and uranium were excluded on account of the instability of their solutions, thus leaving only the oxides of copper, bismuth, and antimony. When experimenting with alcoholic, alkaline copper oxide solution, the author met with a body which he considers is probably "copper-potassium glyceride," and this he is examining further. It forms minute, acicular crystals of a bright blue colour and silky lustre, gradually decomposed in the air and turning green; soluble in water and dilute alcohol, insoluble in absolute alcohol. Bismuth nitrate and antimony chloride behaved similarly towards glycerin. The solubility of basic bismuth hydrate (BiO_2H) depends on the degree of concentration of the alkaline glycerin solution, and is of no value for the quantitative estimation of glycerin.

Reducing Power.—Copper oxide and glycerin were heated together in a sealed tube to 150° C.; only a slight reduction took place. Ammoniacal silver oxide solution also gave unsatisfactory results, and it was necessary to use the oxide itself, to effect oxidation of the glycerin. Gold oxide in alkaline solution is reduced by heating with glycerin. A concentrated solution of glycerin, mixed with a few drops of such a liquid, gives, in the cold, a cherry-red colour, changing to violet on warming; on continued warming, the colour changes to a greyish-green, and metallic gold separates out. Freshly precipitated mercuric oxide undergoes no change on heating with glycerin alone, but in presence of strong bases, reduction to mercury

takes place. Mercuric iodide is reduced to metal more readily than mercuric chloride, by excess of glycerin in presence of caustic potash. Thallium trioxide is reduced in presence of alkali to monoxide, and the trichloride to monochloride; thallium monoxide is not altered. Lead dioxide has already been tried, without success, by Schaumann for estimating glycerin. Bismuth pentoxide is reduced, in presence of alkali, to basic hydrate (BiO_2H). Manganese dioxide and the basic oxides of manganese are not reduced in presence of alkali, but the acid-forming oxides give off oxygen. Ferric oxide, in presence of caustic potash, remains unchanged, but potassium ferricyanide is slowly reduced to ferrocyanide. Nickel sesquioxide is converted into the hydrate, $Ni(OH)_2$, but the reduction is incomplete. Neither cobalt sesquioxide nor the double cyanide of cobalt and potassium are reduced by glycerin; towards cobaltous chloride, glycerin behaves like the sugars. Metallic palladium is precipitated from palladium oxide by potash and glycerin; palladium chloride, in presence of potash and ammonia, is not altered. Platinum chloride is reduced by alkaline glycerin solution, but the reaction is not so sensitive as the palladium, silver, and gold reactions. Ruthenium potassium chloride is not altered by alkaline glycerin, but rhodium chloride is reduced. The brown solution of osmium tetrachloride in potash becomes nearly colourless on boiling with glycerin, but no reduction to metal takes place. Iridium chloride, on heating with potash and glycerin, undergoes no apparent alteration.

Six oxides—Ag_2O, Au_2O_3, HgO, RhO_2, PdO, and PtO_2—are reduced to metal by alkaline glycerin, the remainder only giving up either part of their oxygen or none at all.

Quantitative Estimation of Glycerin by the Reduction of Metallic Oxides.—Concordant results were obtained by the reduction of palladium oxide with alkaline glycerin, but as mono- and polyvalent alcohols, acetaldehyde, acetone, carbohydrates, formic acid, &c. also effect the reduction, the method is practically of no use. The reduction of mercuric iodide by alkaline glycerin gives valueless results. The amounts of metallic silver obtained by the reduction of silver oxide correspond fairly well when like quantities of glycerin are used, but with varying quantities of glycerin, the results are not strictly proportional. According to Kiliani (Annalen, **205**, 191), besides oxalic acid and carbon dioxide, formic acid and glycollic acid are obtained by treating glycerin with silver oxide, but the author states that as the oxidation products of glycerin are further oxidised by silver oxide, there can only result as final products of the reaction, formic acid, oxalic acid, and carbon dioxide. The silver oxide process is carried out in the following manner:—20 c.c. of glycerin solution, containing, at the most, 0·15 grm. of glycerin, are placed in an Erlenmeyer flask of 10 c.m. diameter at the bottom, with 4 grms. of $AgNO_3$, and after the latter has dissolved, 5 c.c. of 50 per cent. caustic soda are added, and the whole mixed by moderate shaking till a thick pulp forms. The flask is placed up to the neck in a vigorously-boiling water-bath for $3\frac{1}{2}$ hours, and then diluted with 100 c.c. of water. The liquid is cooled and decanted, the precipitate washed once with water, and then treated, on the water-bath, with a solution of sodium bisulphate (30 grms. of concentrated H_2SO_4 and 100 grms. of Na_2SO_4 to the litre), till it appears bright grey. The liquid is then decanted off as far as possible, the precipitate washed twice with 25 c.c. of 10 per cent. ammonia, dried, ignited, and weighed. One molecule of glycerin, when operating with 0·15 grm. of glycerin, corresponds to 1,060—1,070 parts of silver; in round numbers, 1 part by weight of glycerin = 11 parts by weight of silver.

After criticising the numerous methods for the estimation of glycerin in fermented liquors, the author considers that of the German Imperial Commission of 1884 to be the best, and he recommends the examination of the separated glycerin by his silver process. Thus, he found that the amount of real glycerin in that separated from different wines was as follows:—White wine, 80·7 to 97·5 per cent.; red wine, 86·9 to 100·1 per cent.; and sweet wine, 66·8 to 75·4 per cent.—A. S.

Chloroform, Analysis of. A. Béhal and M. François. J. Pharm. Chim. 1897, **5**, 417—424.

In certain respects commercial chloroform does not satisfy the tests of the Codex. It crystallises more or less on cooling to 20°—40° C., turns chromic acid green, and is coloured by crystallised magenta and dinitrosulphide of iron. The impurities to which these reactions are due, are water and alcohol, the former introduced with the alcohol added to render the chloroform less liable to decomposition.

The method adopted for the determination of the alcohol is a modification of that of Nicloux (this Journal, 1897, 473; see also Kuriloff, this Journal, 1897, 473). The bichromate solution contains 16·97 grms. per litre, 2 c.c. thus correspond to 0·01 c.c. of absolute alcohol. 5 c.c. of the dilute alcohol solution to be estimated are placed in a test tube surrounded by boiling water, 2 c.c. of sulphuric acid is added, and then the bichromate until the yellowish-green tint is reached. For the estimation of alcohol in chloroform, 10 c.c. of chloroform are shaken with 4 c.c. of sulphuric acid in a stoppered tube, provided with a tap at the lower end, the acid drawn off, and the process repeated with 4 c.c., and finally 2 c.c. of acid. The whole 10 c.c. of acid are placed in a small flask, 40 c.c. of water added, and 20 c.c. slowly distilled off, 5 c.c. of the distillate are now taken for the alcohol estimation. The test analyses quoted are satisfactory. Chloroform so deprived of alcohol and water should satisfy all the Codex tests.

The authors recommend that the Codex should recognise the addition of 5 or 10 c.c. of absolute alcohol to 1 litre of chloroform, and give the tests to which such a mixture should be subjected. The odour should be pleasant, as also that of the residue after evaporation, it should be neutral to litmus, should not precipitate or reduce silver nitrate, nor colour sulphuric acid or be coloured by boiling with solid potash. Also the chloroform deprived of alcohol by sulphuric acid, should entirely distil at 60·8° C., the distillate should have the density of 1·500 at 15° (1·523 at 0° C.), should not crystallise on cooling to 40°, and should not be coloured by contact with chromic acid or dinitrosulphide of iron. The magenta test is rejected as too delicate, the authors could not prepare chloroform which was not rapidly coloured in contact with air by this reagent.—A. C. W.

Ethyl Alcohol and Glycerin, Determination of. Nicloux. J. Pharm. Chim. 1897, **5**, 424—427.

Bordas and Raczkowski take a bichromate solution of 21 grms. in 1 litre, as compared with the 20 grms. given by Nicloux, 1 c.c. of each solution being said to correspond to 0·1 c.c. of absolute alcohol in 100 c.c. of the solution. The author now finds that, under the conditions he gave—addition of 4—5 c.c. of sulphuric acid, boiling for some seconds, end point a yellowish green—19 grms. per litre is more nearly correct. The method given by Béhal and François (see preceding abstract) gives accurate results under the conditions they observe.

In the determination of glycerin, solutions of 38 or 19 grms. per litre should be used, 1 c.c. corresponding to 5 c.c. of a solution of 1 or 0·5 grm. of glycerin per litre. 4 or 5 c.c. of strong sulphuric acid are used, the mixture is warmed for one minute, and after five minutes waiting the reaction is complete. The equation for the reaction is—

$$28H_2SO_4 + 7K_2Cr_2O_7 + 3C_3H_8O_3 = 7Cr_2(SO_4)_3 + 7K_2SO_4 + 9CO_2 + 40H_2O.$$

—A. C. W.

Caoutchouc, Valuation of Raw and Manufactured. R. Henriques. Zeits. für öffentl. Chem. **3**, 37; Analyst, 1897, **22**, 134.

Raw Caoutchouc.—This product contains water, inorganic substances (sand, stone, clay, &c.), organic impurities (leaves and twigs), added gum resins from other plants, and the so-called caoutchouc resin, distinguished from the true caoutchouc substance by its ready solubility in ethyl and methyl alcohol and in acetone. The method of analysis is as follows:—A weight of 50 to 100 grms. is macerated in water in a small roller apparatus—similar to the kneading and washing machine used in india-rubber works—by which

the mechanical impurities are removed. If a proper macerating apparatus is not available, the mechanical impurities may be eliminated by the aid of water and a pair of scissors. The resulting rubber is dried at 80° to 90° C., and weighed. The caoutchouc resin is then extracted by digesting 5 to 10 grms. of the washed and dried rubber for two to three hours with acetone, in a Soxhlet apparatus; the residue is quickly dried and weighed. The ash is also determined, and the percentage of true caoutchouc obtained by difference.

Manufactured Rubber.—The principal constituents are: 1. Added substances, such as lead oxide, heavy spar, gypsum, zinc oxide and carbonate, chalk, potash, magnesia and magnesium carbonate, golden antimony sulphide, ferric oxide, kieselguhr, asbestos, powdered glass, pumice stone, &c. 2. Caoutchouc resin, soluble in alkalis. 3. Caoutchouc substitute, asphalt, and gum resins. 4. Free sulphur. 5. Insoluble caoutchouc resin. 6. Combined sulphur, as vulcanite. 7. True caoutchouc substance. The rubber is analysed in the following manner:—5 grms. of the sample reduced to a fine powder by the aid of a sharp file, are boiled for three to four hours in a 3 to 4 per cent. alcoholic solution of caustic soda, which effects the solution of the free sulphur, resin, caoutchouc substitute, and the soluble caoutchouc resin, in addition to which, many organic constituents may be attacked and partly dissolved. The solution is filtered whilst still boiling, and the residue washed with boiling alcohol. If the presence of asphalt be suspected, the residue is treated with cold nitrobenzene; otherwise this operation may be omitted. The residue is then washed into a beaker, and boiled several times, first with water and then with hydrochloric acid, the washings being decanted through the same filter. The residue is then all brought on to the filter, washed with hot water, pressed, dried at 80° to 100° C., and weighed. The drying must not be continued longer than necessary, as the dry caoutchouc gradually takes up oxygen. The residue now contains the caoutchouc substance, the combined sulphur, insoluble inorganic substances, a very small quantity of the caoutchouc resin, and a little carbonaceous matter added for colouring purposes. If the ash and the sulphur be now determined, and their amounts deducted from the residue, the remainder will be the true caoutchouc substance.

—A. S.

Marmé's Reagent, Limit of Sensibility of, to certain Alkaloids. S. Verven. Ann. de Pharm. 1897, **13**, 145; Chem. Zeit. Rep. 1897, **21**, 116.

The reagent employed for the quantitative determination of its sensibility to the following alkaloids, consisted of 5 grms. of cadmium iodide and 10 grms. of potassium iodide in 100 c.c. of water. 5 c.c. of the aqueous solution of the alkaloid of known strength, rendered slightly acid with sulphuric acid, was added to 1 c.c. of the reagent and shaken; it produced a precipitate: the liquid was then diluted with water until the final dilution, with which only a slight precipitate was obtained, was in the following ratio for each alkaloid enumerated:—Atropine (Merck), 1:1600; cocaine hydrochloride (puriss cryst.), 1:16,900; veratrine (Merck, puriss cryst.), 1:5400; strychnine (cryst.), 1:19,200; brucine (cryst. puriss), 1:14,600; quinine (cryst.), 1:32,300; cinchonine (cryst.), 1:18,400; aconitine (Merck, purum cryst.), 1:1300. The following dilutions gave no precipitate, even on standing for a few minutes:—Atropine (Merck), 1:1900; cocaine hydrochloride (puriss cryst.), 1:20,800; veratrine (Merck, puriss cryst.), 1:8200; strychnine (cryst.), 1:32,000; brucine (cryst. puriss), 1:18,200; quinine (cryst.), 1:37,700; cinchonine (cryst.), 1:24,600; aconitine (Merck, purum cryst.), 1:27,400.—J. O. B.

Phosphoric Acid in Wine, Simple Method for the Estimation of; especially in Sweet Wine. W. Thörner and R. Uster. Forsch. Ber. ü. Lebensm u. ihre Bez. z. Hyg., &c., **4**, 55—58; Chem. Centr.-Bl. 1897, [1], 825—826.

25 c.c. of sweet wine (in the case of ordinary wine, 50—100 c.c. are taken and evaporated to 25 c.c.) are heated for some time with 10 c.c. of concentrated nitric acid (sp. gr. 1·35) in a covered beaker on the water-bath. A vigorous evolution of gas takes place, and the liquid becomes bright yellow. The reaction is complete in 20 to 30 minutes. Ammonia is added till the liquid distinctly smells ammoniacal, and then 25 c.c. of ammonium citrate solution. The liquid becomes darker, but remains clear. The phosphoric acid is finally precipitated, with stirring, by 15 to 20 c.c. of magnesia mixture. The results obtained by this method are always a few thousandths per cent. less than by the method in which the wine is first evaporated and the residue calcined.—A. S.

Chocolate, Detection of Earth Nuts [Arachis hypogæa] in. A. Bilteryst. Bull. Assoc. Belge des Chimistes, **10**, [12], 447—452.

The presence of arachis- or earth-nuts in chocolate may be detected microscopically by the large starch grains (with nucleus) visible in aqueous or glycerin preparations, and by the cells with decided internal markings discernible in chloral preparations.

In the chemical examination, 2 grms. of the chocolate are heated in a flask with 50 centigrms. of mercury, 20 c.c. of sulphuric acid, containing 15 per cent. of phosphorus pentoxide, and 10 grms. of acid potassium sulphate, slowly for 20 minutes, and then strongly until decolorisation occurs (in about half an hour). After cooling and taking up with 100 to 120 c.c. of water, the mixture is transferred to a 750—1,000 c.c. retort, where 100 c.c. of sodium hydroxide solution (500 grms. per litre), 20 c.c. of sodium sulphide solution (40 grms. per litre + 50 c.c. of above NaOH solution), and 2 grms. of zinc are added, and heated until 50 c.c. have distilled over. Ten minutes will suffice to drive off the ammonia, which is absorbed in 50 c.c. of decinormal sulphuric acid, the excess of which is then titrated with Congo red solution. To prevent the contents of the retort frothing over, 10 drops of paraffin oil are added before distilling.

The albuminoid matters determined by this process amounted to 9·1 per cent. in the case of pure chocolate; 28·18 per cent. in that of earth nuts; 17·57 per cent. in that of pure cocoa; 46·9 per cent. in that of earth-nut cake; 12·53 per cent. in that of chocolate + 10 per cent. of earth nuts, or 15·7 per cent. with 10 per cent. of earth-nut cake, and 21·18 in the case of cocoa + 10 per cent. of earth-nut cake. Foorster's modification of the Kjeldahl method may also be employed.

In estimating the fatty matter, the refractometer index may afford assistance when earth nuts have been employed to adulterate chocolate, the refractive index in the Amagat and Jean oleorefractometer being, for cocoa butter, − 19°, and for Arachis oil, + 3°, the presence of 5 per cent. of earth nuts altering the reading to − 18°, and 50 per cent. to − 7°. When earth-nut cake is used, this method is inapplicable.—C. S.

Caffeïne in Coffee and Tea, Estimation of. A. Hilger and A. Juckenack. Forsch. Ber. ü. Lebensm. u. ihre Bez. z. Hyg., &c., **4**, 49—50. Chem. Centr.-Bl. 1897, [1], 775.

20 grms. of finely ground coffee or powdered tea (raw coffee is dried as much as possible in the drying chamber before grinding) are soaked for some hours in 900 grms. of water at the ordinary temperature (3 hours for raw coffee and 1½ hours for roasted coffee and tea), and the mixture then raised to boiling, after adding water to compensate for evaporation. Cool to 60°—80° C., add 75 grms. of ammonium acetate liquor D. A. B., and whilst stirring, add 1·9 grm. of sodium bicarbonate; then boil for 5 minutes, and after cooling, bring the total weight up to 1,020 grms. with water. Filter, and evaporate on the water-bath, with stirring, 750 grms. of the perfectly clear filtrate (corresponding to 15 grms. of original substance) with 10 grms. of precipitated powdered aluminium hydrate, and some filter-paper shaken to a pulp with water; completely dry the residue, and exhaust for 8 hours with carbon tetrachloride in a Soxhlet's extractor. Distil off the carbon tetrachloride and dry and weigh the residue of perfectly white caffeïne. The nitrogen in the crude caffeïne can be estimated accurately by Kjeldahl's process and the amount of anhydrous caffeïne thus calculated. The numbers obtained by titration are on the average 2 to 4 mgrms. less than the gravimetric results.

—A. S.

Caffeïne, Estimation of. G. L. Spencer. J. Amer. Chem. Soc. 1897, **19**, 279—281.

The author found in comparative experiments with a number of methods for the determination of caffeïne in tea, the Gomberg method (this Journal, 1896, 384) gave the most satisfactory results, and admitted of a wider application than the gravimetric methods.—O. H.

Caffeïne in Tea, Estimation of. C. C. Keller. Ber. Pharm. Ges. 1897, **7**, 105; Chem. Zeit. Rep. 1897, **21**, 102.

The author recommends the following method:—6 grms. of dried tea leaves are added to 120 grms. of chloroform in a wide-necked separating funnel, and when the chloroform has come into thorough contact with the tea, 6 c.c. of 10 per cent. ammonia are added, the mixture being repeatedly and vigorously shaken for half an hour. The whole is then allowed to stand till the solution has become perfectly clear (in 3 to 6 hours) and the tea has completely absorbed the aqueous liquid. 100 grms. of the chloroform, corresponding to 5 grms. of tea, are now filtered through a small filter moistened with chloroform, into a small tared flask, and the chloroform distilled off on the water-bath. The residue is evaporated on the water-bath with 3 to 4 c.c. of absolute alcohol; the caffeïne separates out in white crusts, whilst the chlorophyll deposits on the bottom. A mixture of 7 c.c. of water and 3 c.c. of alcohol is added, and the flask heated on the water-bath. The caffeïne is thus dissolved, and the solution is filtered through a small filter moistened with water. The flask and filter are washed with 10 c.c. of water, the filtrate evaporated in a small, tared glass basin and the residue of pure caffeïne weighed.—A. S.

Butter Analysis, Methods of. L. Drumel. Bull. Assoc. Belge des Chimistes, **10**, [11], 411.

Natural butters, whether artificially coloured or not, are all decolorised by heat—boiling in a test tube for a few seconds — whereas the ordinary commercial margarines retain their colour under these conditions, and mixtures of the two exhibit partial decolorisation, the extent of which depends on the relative proportions of the constituents. The only exception observed by the author is in the case of margarines containing neutral lard, these behaving like natural butter; they are, however, rarely met with in commerce.—C. S.

Filicic Acid, Various Methods of Determining, in Extract of Male Fern. J. Pharm. Chim. 1897, **5**, [6], 443.

Kraft (Schweiz. Woch. für Chem. und Pharm. (35), 1896) recommends the following process:—5 grms. of the extract are agitated for 15 minutes with a solution of 2 grms. of potassium carbonate in 4 grms. of water and 60 grms. of 95 per cent. alcohol. 83 grms. of liquid are then filtered off into a separator, 9 grms. of 25 per cent. hydrochloric acid are added, followed by 50 grms. of ether and 35 grms. of water. The mixture is well agitated, the aqueous portion rejected, and the ethereal layer washed with another 35 grms. of water. The ether solution is then run into a tared flask, the solvent distilled off, and the residue evaporated to 2 grms. This is dissolved in 1·5 grm. of boiling amylic alcohol, 5 grms. of methyl alcohol are added, and then the filicic acid is precipitated by the further gradual addition of 25 grms. of methyl alcohol. The flask is then allowed to stand and well corked for 12 hours, when the precipitate is collected on a tared filter, washed with 10 c.c. of methylic alcohol, dried between 60° and 70° C. and weighed. The resulting weight of filicic acid represents the amount present in 4 grms. of original extract. The amount of filicic acid obtained from different specimens of commercial extract varies from between 0·4 and 10 per cent.

Bocchi's Method.—This method (Boll. Chim. Farm. 1896, 449) is based on the fact that filicate of calcium is soluble in cold water, forming a solution from which it may be liberated by an acid. One or two grms. of the extract are dissolved in ether, and the solution washed with lime water until the latter is no longer coloured and gives no precipitation on acidulating a portion. The alkaline aqueous extracts are mixed and filtered into a separator and acidulated with acetic or hydrochloric acid; the filicic acid thus liberated is washed out with carbon bisulphide, the solution filtered, evaporated to dryness on the water-bath, and weighed as filicic acid.

Fromme's methods (Pharm. Zeit. 1896, **41**, 607, and *ibid.* 1897, **42**, 44) closely resemble the preceding. From 1·5 to 2 grms. of the extract are weighed out into a small capsule, 2 grms. of ether added and then 3 grms. of magnesia are carefully triturated with the mass. When the ether has evaporated, water is gradually added to form a fluid paste. After standing for some time, the aqueous portion is thrown on a filter and the residue again treated with water and again filtered, the process being repeated until no precipitate is obtained on acidulating a portion with hydrochloric acid. The volume of the filtrate will then be from 200 to 250 c.c. This is acidulated with hydrochloric acid, and extracted in a separator with carbon bisulphide, in four successive washings. This extract is evaporated to dryness on the water-bath in a small tared flask. The dried residue is redissolved in 10 drops of amylic alcohol, and the filicic acid precipitated with methyl alcohol, and dried precisely as in Kraft's method. *Fromme's second method* consists of shaking together 5 grms. of extract, 30 grms. of ether, and 100 grms. of 2 per cent. barium hydrate solution. After agitation for five minutes, 86 grms. of the aqueous solution, equivalent to 4 grms. of extract, are run off; this weight is saturated with a slight excess of hydrochloric acid and extracted with four successive washings with ether. The ethereal extracts are evaporated to dryness in a tared flask, the residue redissolved in amyl alcohol, and precipitated with methyl alcohol, and finally weighed, as in Kraft's process, except that the drying is commenced at 40° C. The first step in the process should be rapidly conducted, or the barium hydrate will decompose a portion of the filicic acid. —J. O. B.

Thymol, Determination of, in Thyme Oil. Kremers. Pharm. Rundschau. 1896, 221; Schimmel's Rep., April 1897, 41.

Five c.c. of the oil are weighed and introduced into a glass stoppered burette graduated in $\frac{1}{10}$ c.c.; an equal volume of petroleum ether is then added, followed by a 5 per cent. solution of potassium hydrate; after well shaking together, the liquid is allowed to stand until separation is complete. The alkaline solution is then run off into a 100 c.c. flask, the residual oily liquid being again washed with alkali until no further decrease in the volume of the oil follows. The mixed washings are then made up to 100 or 200 c.c. with a 5 per cent. soda solution. To 10 c.c. of this solution in a graduated 500 c.c. flask $\frac{1}{10}$ N. iodine solution in slight excess is added: it precipitates the thymol. This excess of the precipitant may be determined by the addition of a few drops of dilute HCl to a small portion of the liquid, if any thymol remains uncombined with the iodine, a turbidity results, whereas if iodine be in excess a clear brown solution is obtained. When this point is reached, the original liquid is rendered slightly acid with HCl and diluted to 500 c.c. From this 100 c.c. are filtered off, and the excess of iodine is determined by titration with $\frac{1}{10}$ N. thiosulphate solution. The number of c.c. required, multiplied by 5 and deducted from the number of c.c. of iodine solution added, gives the number of c.c. of iodine solution used, each c.c. of which is equivalent to 0·003741 grm. of thymol.—J. O. B.

Carvacrol, Determination of. E. Kremers. Pharm. Rundschau. 1896, 221; Schimmel's Rep., April 1897, 41.

The method of the determination of carvacrol is precisely similar to that given for thymol, with this slight modification—that after the iodine solution has been added, the mixture is vigorously shaken to cause the cohesion of the precipitate, and is then filtered. The filtrate is then acidulated with hydrochloric acid and the process continued precisely as described for thymol.—J. O. B.

Sandalwood Oil, East Indian; Analytical Data of. Schimmel's Rep., April 1897, 36.

A genuine sandalwood oil should have the following characters—specific gravity not below 0·975 at 15° C., optical rotation, 17°—19°. It should be soluble in 5 parts of 70 per cent. alcohol at 20° C., and should contain at least 90 per cent. of santalol. The latter constituent may

be determined as follows:—About 20 grms. of the oil are mixed with an equal volume of glacial acetic acid (or acetic anhydride, *see* Schimmel's Rep., Oct. 1895, 13), a little anhydrous sodium acetate is added and the whole gently boiled for an hour and a half. After cooling, the acetylised oil is washed, first with water, then with very dilute soda solution to remove free acid, and it is then dried by shaking with anhydrous sodium sulphate. From 2 to 5 grms. of this dry acetylised oil is weighed off and sapoitied by boiling with an excess of normal alcoholic potash solution under a reflux condenser. The amount of potash used up is then determined by titrating back with normal acid. The percentage of santalol may be found from the following formula—

$$P = \frac{a \times 22 \cdot 2}{S - (a \times 0 \cdot 042)}$$

where—

P = percentage of santalol.
a = the number of c.c. of potash used up.
S = quantity of acetylised oil weighed off.

—J. O. B.

Acetone, Impt. of Squibbs' Volumetric Method for Estimating. L. F. Kebler. J. Amer. Chem. Soc. 1897, **19**, 316—320.

The author has studied and worked with Squibbs' modification of Robineau and Rollin's method (this Journal, 1897, 168), and finding that a difficulty was experienced in obtaining pure acetone, and the drop final reaction being lengthy and tedious, has modified the method so that the use of pure acetone and the drop final reaction are dispensed with.

The solutions required are as follow:

1. A 6 per cent. solution of hydrochloric acid.
2. A decinormal solution of sodium thiosulphate.
3. Alkaline potassium iodide solution prepared by dissolving 250 grms. of potassium iodide in water, made up to a litre; dissolving 257 grms. of sodium hydroxide (by alcohol) in water, likewise made up to a litre. After allowing the latter to stand, 800 c.c. of the clear solution are added to the litre of potassium iodide.
4. Sodium hypochlorite solution: 100 grms. of bleaching powder (35 per cent.) are mixed with 400 c.c. of water; to this is added a hot solution of 120 grms. of crystallised sodium carbonate in 400 c.c. of water. After cooling, the clear liquid is decanted, the remainder filtered, and the filtrate made up to a litre; to each litre is added 25 c.c. of sodium hydroxide solution (sp. gr. 1·29).
5. An aqueous acetone solution containing 1 or 2 per cent. of acetone.
6. Bicarbonated starch solution, prepared by treating 0·125 grms. of starch with 5 c.c. of cold water, then adding 20 c.c. of boiling water, boiling a few minutes, cooling, and adding 2 grms. of sodium bicarbonate.

To 20 c.c. of the potassium iodide solution are added 10 c.c. of the diluted aqueous acetone, an excess of the sodium hypochlorite solution is then run in from a burette and well shaken for a minute. The mixture is then acidified with the hydrochloric acid solution, and while agitated, an excess of sodium thiosulphate solution is added, the mixture being afterwards allowed to stand a few minutes. The starch indicator is then added and the excess of thiosulphate re-titrated. The relation of the sodium hypochlorite solution to the sodium thiosulphate being known, the percentage of acetone can be readily calculated.—O. H.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Chlorine Water, Preparation of. G. Griggi. Bolletino Farmac. Chim. 1896, 680; Chem. Centr.-Bl. 1897, [1], 873.

Pure chlorine water may be obtained from lead dioxide (or red lead), oxalic acid, and calcium chloride, without use of apparatus, by the following formula—

$$2(C_2H_2O_4 + 2H_2O) + PbO_2 + (CaCl_2 + 6H_2O) = CaC_2O_4 + PbC_2O_4 + 12H_2O + Cl_2.$$

—A. S.

Strontium Chromate, Action of, on Mercuric Chloride. G. Belugou. Bull. Soc. Chim. 1897, **17**, 473—474.

On digesting strontium chromate with a saturated solution of mercuric chloride on the water-bath, filtering, and cooling, fine orange crystals are deposited.

Analysis leads to the formula, $2(3HgCl_2.SrCrO_4).HCl$.

Basic mercuric chromate probably remains in the insoluble portion.—A. C. W.

Strontium Chromate, Action of, on Mercuric Chloride. H. Imbert and G. Belugou. Bull. Soc. Chim. 1897, **17**, 471—473.

The double salt, $SrCrO_4.2HgCl_2.HCl$, is obtained by dissolving strontium chromate ($\frac{1}{2}$ mol.) in concentrated hydrochloric acid, diluting and dissolving in the liquid mercuric chloride (1 mol.). After digesting on the water-bath and filtering, a salt of the above composition crystallises out in orange-red crystals. The compound is not decomposed by water, and recrystallises unchanged. In its solutions ammonia produces a canary-yellow precipitate, which appears to be the body $2(HgCl.NH_2).HgCrO_4$.—A. C. W.

Magnesium Methylate. E. Szarvasy. Ber. **30**, [7], 806—809.

The author had previously shown (Ber. **30**, 305) that the chief product of the action of methyl alcohol on magnesium nitride was magnesium hydroxymethylate, $Mg(OH)OCH_3$. Magnesium and methyl alcohol react together according to the equation—

$$Mg + 2CH_3.OH = Mg(OCH_3)_2 + H_2.$$

If the reaction be carried out at a high temperature, magnesium methylate is left as a white powder readily decomposed by moisture.

At the summer temperature, methyl alcohol dissolves clean magnesium, converting it into a gelatinous precipitate; from a solution in the proper proportions, crystals, of the composition $Mg(OCH_3)_2 + 3CH_4O$, separate. This crystallised methylate is soluble in methyl and ethyl alcohols and in benzene, the amorphous compound is, however, almost insoluble; thus a solution of the crystalline compound on heating, deposits the amorphous substance.

The main reaction of bromine on the methylate takes place as follows:—

$$Mg(OCH_3)_2 + 4Br_2 = MgBr_2 + 2CHBr_3 + 2H_2O.$$

—A. C. W.

Urea, Yield in the Transformation of Ammonium Carbonate into. L. Bourgeois. Bull. Soc. Chim. 1897, **17**, 474—477.

Ammonium sesquicarbonate was heated for six hours in a sealed tube at 130°. After cooling, the carbon dioxide was liberated, the tube again sealed, and heated once more for six hours. The whole process being several times repeated; finally on evaporating the contents to dryness on the water-bath, urea was left, mixed only with small quantities of its decomposition products (biuret, &c.). The yield varied from 3·2 to 9·52 per cent.; ammonium bicarbonate, treated in the same way gave 2·5 to 2·9 per cent., and ammonium carbonate 2·6 to 3·7 per cent.

In an experiment in a steel autoclave with the sesquicarbonate, 7·55 per cent. of urea was obtained; the autoclave was seriously attacked by the carbonic acid, a considerable quantity of ferrous carbonate being found in the liquid. In the absence of air, copper is unattacked under the same conditions.—A. C. W.

Acetylene, Action upon Cupric Salts. H. G. Söderbaum. Ber. **30**, [7], 814—815.

Acetylene gives, in a cold ammoniacal solution of a cupric salt, a black precipitate, which, dried *in vacuo*, appears to have the formula $12C_2Cu + H_2O$. The precipitation takes place more slowly in a cold solution, but is quantitative. The composition of the acetylide is considerably influenced by the temperature of precipitation. With rapid heating the acetylide explodes at 50° C., but with a slow rise of temperature at 70°, or even 80° after long desiccation.

—A. C. W.

Acetone, Condensation of [to Terpene-like Hydrocarbons], by boiling with Zinc Chloride. P. N. Raikow. Ber. **30**, 905—906.

Freshly distilled acetone when heated under a condenser with zinc chloride on a sand bath, becomes yellow and finally dark brown. After boiling for about 20 hours a mobile yellowish oil separates, which on adding more zinc chloride and continuing the boiling, becomes more and more viscid, darker, and begins to exhibit an olive-green fluorescence. For the purposes of the investigation an oil was taken resulting from boiling the mixture 30 hours. After separation from the lower layer, it was fractionated, a small portion passing over at 120°—150° C., the thermometer then rising continuously to 390° C. when the distillation was stopped. Two fractions boiling at 162° C. and at 252° C. were analysed and gave numbers which corresponded to terpenes rather than to mesitylene. A condensation of acetone with separation of water should only yield polymeric hydrocarbons of the general formula $(C_3H_4)_n$, whilst the formation of hydrocarbons of which the percentage numbers agree with those of the terpenes can only be explained on the assumption that a decomposition of the carbon chain in acetone, or in its condensation product, has taken place.—T. A. L.

Arrow Poison from the Larva of a Beetle. (Diamphidia locusta.) R. Boehm. Chem. Zeit. Rep. 1897, 79; Pharm. Centralh. **38**, 277.

The tribe of bushmen of that part of South Africa known as Kalahari use the juice of the leaf beetle *Diamphidia locusta* and its larva for poisoning their arrowheads. Lewin (Pharm. Centralh. **36**, 599) found in its body besides inert fatty acids a toxalbumin which caused paralysis and finally, death.

To obtain a solution of the poison, Boehm recommends the maceration of the whole larvæ in distilled water for some hours, when they swell up, and the liquid, which now possesses poisonous properties, is light yellow in colour, and acid in reaction. This acidity is not removed by shaking out with ether. It gives the usual reactions for a toxalbumin, and can be precipitated from its aqueous solution with ammonium sulphate.—J. O. B.

Diastase, Action of Light on, and its Biological Significance. J. R. Green. Proc. Royal Soc. 1897, **61**, 25.

See under XVII., *page* 549.

International Congress on Technical Education. Chem. and Druggist, June 19, 1897, 946.

This Congress was opened at the Society of Arts, Adelphi, on June 15th, the Duke of Devonshire presiding, after M. Léo Saignat, the retired president, formally gave up the office. The Duke of Devonshire explained that the meeting of the Congress in London was due to the Society of Arts and to the City companies, which had guaranteed the needful expenditure. He remarked on the beneficial effect of public conferences on educational questions, and thought the present time well chosen for an international congress on technical instruction, for in all countries there were signs of increasing interest in foreign methods of education. English education, he said, had been materially affected during the last 60 years by foreign influences. There was one point at least in which continental critics were now paying Great Britain the compliment of careful study and even of admiration. We had been recently working out an interesting and fruitful experiment in combining financial aid from the central exchequer with great liberty of action on the part of county and county borough councils. The policy of permitting local authorities to expend their share of the residue of the excise duties on technical education had probably done more than any previous act of the State to stimulate national interest in technical instruction. The local authorities, with few exceptions, had risen to their new duties with commendable alacrity and enterprise.

"The Teaching of Pure Chemistry and Applied Chemistry" was the first paper read, Prof. Otto N. Witt, of Berlin, being the author. In it he advocated a thorough grounding in pure chemistry as the best training for future work, and deprecated specialism in the early part of the young chemist's career; that should come later. Prof. H. E. Armstrong read a paper on an allied subject, and Dr. J. H. Gladstone followed with one on teaching chemistry in continuation evening schools, stating that in Manchester there are arrangements for 12,451 pupils at evening schools, and for 6,619 in higher-grade day schools, science being provided for all, if they want it. At present 2,738 students are attending the practical chemistry laboratories. These papers opened the discussion on technical education, in the course of which Sir H. Roscoe stated that he recently visited some large colour works near Frankfurt, where 100 men were employed, including many highly trained scientific chemists who had devoted years to original research with a view to making new discoveries. One employé, who received 1,000*l.* a year, worked for several years without producing any results. But eventually he made a discovery which repaid the firm ten times over, and placed an entirely new branch of manufacture in their hands. That showed what could be done by the application of science.

An excellent paper on "The Teaching of Chemistry," by Prof. G. Lunge, was read by Sir H. Trueman Wood. Prof. Lunge held that, to raise English chemical industry to the foremost rank, it was necessary that the technical management of chemical factories should not be left in the hands of "rule-of-thumb" men, but should be entrusted to real chemists. These men should have a much fuller education than the majority of chemists seem to obtain in Great Britain at present. His views as to the kind of education necessary were the same as Prof. Witt's. He did not even think that, apart from isolated exceptions, high-class knowledge was much good to foremen, whose duty it was to carry out their instructions and to see that the men did their work as prescribed by the staff, but who were not to meddle with the chemical process itself. It seemed a great waste of time and means to give some superficial chemical teaching to tens of thousands of workmen on the remote chance that one of them might have some real benefit of it, while at the same time many hundreds of educated men were receiving a really efficient training in the same direction, many of whom could not find properly remunerated places owing to a great extent to the cheap labour of "bottle-washers."

On Wednesday, June 16th, the work was divided into two sections, one meeting in the Society of Arts' house and the other in the neighbouring premises of the London School of Economics. The proceedings in both sections consisted chiefly of papers and discussions by science teachers upon the methods of teaching and the people taught. Comparatively little was said about the teachers. Foreign delegates took part in the discussions, and some of the best papers were by them.

New Books.

Analyse der Fette und Wachsarten. Von Dr. Rudolf Benedikt. Dritte erweiterte Auflage, herausgegeben von Ferdinand Ulzer (Vienna). Verlag von Julius Springer, Berlin. 1897. Price M. 12. H. Grevel and Co., 33, King Street, Covent Garden, London.

8vo volume, commencing with a frontispiece of Dr. Benedikt, and followed by a literary memoir, and a list of Benedikt's scientific contributions. After this follows Benedikt's preface to his second edition of this work, published in 1891. The present volume contains 654 pages of subject-matter, illustrated with 48 woodcuts, and followed by an alphabetical index.

The plan of the work is as follows:—I. Constituents of Fats and Waxes. a. Acids. b. Alcohols. II. Physical and Chemical Properties of the Fats and Waxes. III. Estimation of Non-fatty Constituents and Preparation of the Substance for Analysis. IV. Method of Determining the Physical Properties of the Fats. V. Elementary Analysis of Fats. VI. Qualitative Examination of a Fat of known Origin. VII. General Methods for Determining the Quantitative Composition of Fats and Fatty Mixtures. a. Quantitative Reactions. b. Quantitative Estimation of Single

Constituents of the Fats. VIII. Identification and Quantitative Determination of such Foreign Constituents as are either dissolved in the Fatty Substance or melted up with it. A. Estimation of the Unsaponified Matter present. B. Identification of Small Quantities of Fats in Mineral Oils. C. Investigation of the Unsaponifiable Constituents. D. Identification and Estimation of Pure Resin and Colophony in Fat. IX. Investigation of Materials and Products of the Fat Industry. A. Candles. B. Soaps. C. Turkey Red Oil. D. Lubricating Oils. E. Degras. F. Wool-dressing Oils ("Wollspickmittel"). G. Edible Fats. H. Oil Seeds and Oil Cakes. J. Glycerin. K. Acetic. L. ("Faktis") India-rubber Substitutes. M. Blown Oils. X. Investigation of Liquid Fats. A. Physical Methods. B. Different Solubility of Oils as a Means of Identification. C. Chemical Methods. XI. Investigation of Solid Fats and Waxes. XII. Description of the several Fats and Waxes. A. Liquid Fats. B. Solid Fats. XIII. Examples.

Jahresbericht über die Leistungen der Chemischen Technologie, mit besonderer Berücksichtigung der Elektrochemie und Gewerbestatistik für das Jahr 1896. Von Dr. Ferdinand Fischer. Mit 262 Abbildungen. Verlag von Otto Wigand, Leipsic, Germany. 1897. H. Grevel and Co., 33, King Street, Covent Garden, London. Price 24s.

In this annual report, the progress and position of chemical industry up to the end of the year 1896, are treated of. Trade statistics, with more special reference to the German Empire, are also given. The last Jahresbericht contained 1140 pages of subject-matter. The present one (1896) contains 1183. Alphabetical indexes covering 53 pages (names and subject-matter) are given. An index of German patents follows those of names and subject-matter.

The text is illustrated with 262 engravings referring to plant and apparatus. The following is a synopsis of the subjects treated of in the volume, the extent of each group and its treatment being indicated by the number of pages filled :—

Group I. Chemical Technology of Fuel (pages 5 to 102). II. Chemical Metallurgy (pages 103 to 252). III. Electrochemistry (pages 253 to 354). IV. Chemical Manufactures, Inorganic (pages 355 to 478). V. Chemical Manufactures, Organic (pages 479 to 686). VI. Glass, Pottery, Cements, Artificial Stone (pages 687 to 766). VII. Foodstuffs (pages 767 to 974). VIII. Chemical Technology of Fibres (pages 975 to 1039). IX. Other Industries connected with Organic Chemistry (pages 1040 to 1134). X. Apparatus (pages 1135 to 1177). It was remarked in the notice of the Jahresbericht for 1894 that the subject of Electrochemistry filled 85 pages; for 1895 it filled 106 pages; and in the present (1896) volume the text on this subject fills 103 pages.

Chemisch - Technisches Repertorium. Uebersichtlicher Bericht über die neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der technischen und industriellen Chemie, mit Hinweis auf Maschinen, Apparate und Literatur. Herausgegeben von Dr. Emil Jacobsen. 35, Jahrgang. 1896. Zweites Halbjahr. Zweite Hälfte. R. Gaertner's Verlagsbuchhandlung, Herman Heyfelder, Schönebergerstrasse 26, Berlin, S.W. 1897. H. Grevel and Co., 33, King Street, Covent Garden, London.

The second issue for the second half-year of 1896 of Jacobsen's Repertory of Chemical Technology, containing reports on the progress of the following branches of chemical industry :—

I. Foods. II. Paper. III. Photography. IV. By-products, Residues, Manures, Sanitation, Disinfectants, &c. V. Soaps. VI. Explosives, Matches, &c. VII. Preparation and Purification of Chemicals. VIII. Chemical Analysis. IX. Apparatus, Machinery, Electro-technology, Thermo-technology, Appendix, and New Books.

Handbuch der Chemischen Technologie (Bolley's Technologie, 56. (Bd. I. 3, 3) Nachträge und Register). In Verbindung mit mehreren Gelehrten und Technikern bearbeitet, und herausgegeben von Dr. P. A. Bolley und Dr. K. Birnbaum. Fortgesetzt von Dr. C. Engler. Ersten Bandes dritte Gruppe.

Die Chemische Technologie der Brennstoffe. Von Prof. Dr. Ferdinand Fischer (Göttingen). Restlieferung zum dritten Heft, enthaltend Nachträge und Register. Friedrich Vieweg und Sohn, Braunschweig. 1897. H. Grevel and Co., 33, King Street, Covent Garden, London.

This forms the appendix and conclusion of the work of Fischer on the Chemical Technology of Fuel. The volume contains matter from pages 593 to 636 on the Measurements of Heat and Light, illustrated with numerous wood engravings. From pages 637 to 647 there follows the alphabetical index of the entire work.

Anleitung zur Chemisch-technischen Analyse für den Gebrauch an Unterrichts-Laboratorien. Bearbeitet von Prof. F. Ulzer und Dr. A. Fraenkel (Vienna). Verlag von Julius Springer, Berlin. 1897. Price M. 5. H. Grevel and Co., 33, King Street, Covent Garden, London.

This analytical work contains preface, table of contents, and subject-matter covering 188 pages, followed by an alphabetical index. The text is illustrated with 12 engravings of special excellence. The subjects of technical-analytical interest treated of, are as follows :—I. Alkali Manufacturing Industry, &c. II. Mortars, Cements, and Clays. III. Metallurgy (Iron, Zinc Blende, Zinc Dust, Ordinary and Refined Copper). IV. Alloys. V. Manures. VI. Sugar Industry. VII. Fermentation Industries. VIII. Fats, Waxes, and Mineral Oils. IX. Mordants and Tanning Matters. X. Textile Industry and Dyeing. XI. Coal-Tar Products.

Anleitung zur Quantitativen Bestimmung der Organischen Atomgruppen. Von Dr. Hans Meyer (Vienna). Verlag von Julius Springer, Berlin. 1897. Price M. 3. H. Grevel and Co., 33, King Street, Covent Garden, London.

8vo volume, containing preface, table of contents, index of abbreviations, introduction, and subject-matter filling 112 pages. An alphabetical index concludes the work. There are 12 illustrations.

The work is subdivided into the following sections :—

I. Determination of the Hydroxyl Group. II. Of the Carboxyl Group. III. Of the Methoxyl Group. IV. Of the Ethoxyl Group. V. Of the Carbonyl Group. VI. Of the Methylimido Group. VII. Of the Ethylimido Group. VIII. Of the Nitrile Group. IX. Of the Amido Group. X. Of the Amine Group. XI. Of the Imido Group. XII. Of the Diazo Group. XIII. Of the Hydrazine Group. XIV. Of the Nitro Group. XV. Of the Iodo Group and the Iodoso Group. XVI. Of the Peroxide Group.

Appendix.—The iodine number, &c.

Anleitung zu Analytisch - Chemischen Uebungsarbeiten auf Pharmaceutischem und Toxicologischem Gebiete. Zugleich als II. Auflage von Prof. Dr. A. Meyer's "Handbuch der Qualitativen Chemischen Analyse." Bearbeitet zum Gebrauche in Pharmaceutisch-chemischen Laboratorien von Dr. Ed. Schaer und Dr. Paul Zenetti (Strassburg). Hermann Heyfelder, R. Gaertner's Verlagsbuchhandlung, Berlin, S.W. 1897. Price M. 5. H. Grevel and Co., 33, King Street, Covent Garden, London.

This little work contains preface, table of contents, and text covering 175 pages, followed by the alphabetical index. There are eight illustrative woodcuts. The work is subdivided as follows :—I. Chief Reactions of the more important Acids and Organic Preparations of Pharmaceutical Interest. II. Identification or Detection of Inorganic and Organic Poisons in Mixtures of Animal and Vegetable Substances. III. Volumetric Analysis of Preparations of Pharmaceutical Interest. IV. Some more Important Exercises in Chemical Physiology. V. Quantitative Estimation of Alkaloids and other Active Principles in Pharmaceutical Drugs and Galenical Preparations. Processes of Separation. Appendix : Composition of Reagents. Atomic and Molecular Weights, &c.

Prizes.

PRIZES FOR TECHNICAL ESSAYS.

Eng. and Mining J., May 22, 1897, 509.

The Industrial Society of Rouen, France, offers gold medals for a portable pyrometer indicating with accuracy temperatures above 300° C., for a new method of utilising the solid products of the distillation of petroleum, for the best lamp for burning acetylene, for the application of an industrial process for the extraction of sulphur from iron and copper pyrites, and for a rapid and accurate method of determining cobalt and nickel in the New Caledonia ores. The essays must be forwarded to the President of the Society before September 30. They must be written in French. The competition is open to members and non-members of the Society.

Trade Report.

TARIFF CHANGES AND CUSTOMS REGULATIONS.

NEW FRENCH SUGAR LAW.

Bd. of Trade J., June, 1897, 663.

This is a translation of the text of the new French sugar law, which was published in the *Journal Officiel* of the 8th April last.

CUSTOMS DUTIES ON SUGAR IN FOREIGN COUNTRIES.

Bd. of Trade J., June 1897, 679.

A statement showing the rates of Customs duties leviable on unrefined and refined sugar, and on molasses imported into the principal European countries and the United States, according to the latest information in the possession of the Board of Trade.

NEW CUSTOMS TARIFF OF CANADA.

Bd. of Trade J., June 1897, 684.

The following are extracts from the resolutions proposed in the Committee of Ways and Means of the Canadian House of Commons relative to duties of Customs, excise, &c. in Canada, to take effect from the 23rd April last:—

Resolved,—That, unless the context otherwise requires,—

(*a.*) The initials "n.e.s." represent and have the meaning of the words "not elsewhere specified";

(*b.*) The initials "n.o.p." represent and have the meaning of the words "not otherwise provided for";

(*c.*) The expression "gallon" means an imperial gallon;

(*d.*) The expression "ton" means 2,000 pounds avoirdupois;

(*e.*) The expression "proof" or "proof spirits," when applied to wines or spirits of any kind, means spirits of a strength equal to that of pure ethyl alcohol compounded with distilled water in such proportions that the resultant mixture shall, at a temperature of 60° F., have a specific gravity of 0·9198 as compared with that of distilled water at the same temperature.

The regulations respecting the manner in which molasses and syrups shall be sampled and tested for the purpose of determining the classes to which they belong with reference to the duty chargeable thereon, shall be made by the Controller of Customs; and the instruments and appliances necessary for such determination shall be designated by him and supplied to such officers as are by him charged with the duty of sampling and testing such molasses and syrups; and the decision of any officer (to whom is assigned the testing of such articles) as to the duties to which they are subject under the tariff shall be final and conclusive, unless, upon appeal to the Commissioner of Customs within 30 days from the rendering of such decision, such decision is, with the approval of the Controller, changed; and the decision of the Commissioner with such approval shall be final.

That, in the case of all wines, spirits, or alcoholic liquors subject to duty according to their relative strength of proof, such strength shall be ascertained either by means of Sykes' hydrometer or of the specific gravity bottle, as the Controller of Customs directs; and in case such relative strength cannot be correctly ascertained by the direct use of the hydrometer or gravity bottle, it shall be ascertained by the distillation of a sample, and the subsequent test in like manner of the distillate.

That all medicinal or toilet preparations imported for completing the manufacture thereof or for the manufacture of any other article by the addition of any ingredient or ingredients, or by mixing such preparations, or by putting up or labelling the same, alone or with other articles or compounds, under any proprietary or trade name, shall be, irrespective of cost, valued for duty, and duty shall be paid thereon at the ordinary market value in the country whence imported of the completed preparation when put up and labelled under such proprietary or trade name, less the actual cost of labour and material used or expended in Canada in completing the manufacture thereof or putting up or labelling the same.

That all medicinal preparations, whether chemical or other, usually imported with the name of the manufacturer, shall have the true name of such manufacturer and the place where they are prepared permanently and legibly affixed to each parcel by stamp, label, or otherwise; and all medicinal preparations imported without such names so affixed shall be forfeited.

That packages, when imported, shall be subject to the payment of the following duties, viz.:—

(*a.*) All bottles, flasks, jars, demijohns, carboys, casks, hogsheads, pipes, barrels, and all other vessels or packages manufactured of tin, iron, lead, zinc, glass, or any other material capable of holding liquids, and all packages in which goods are commonly placed for home consumption, including cases, not otherwise provided for, in which bottled spirits, wines, or malt liquors, or other liquids are contained, and every package being the first receptacle or covering enclosing goods for purpose of sale, shall in all cases, not otherwise provided for, in which they contain goods subject to an *ad valorem* duty or a specific and *ad valorem* duty, be charged with the same rate of *ad valorem* duty as is to be levied and collected on the goods they contain, and the value of the packages may be included in the value of such goods.

(*b.*) Provided that all such packages as aforesaid containing goods subject to a specific duty only, and not otherwise provided for, shall be charged with a duty of 20 per cent. *ad valorem.*

(*c.*) That packages not herein-before specified, and not herein specially charged with or declared liable to duty, and being the usual and ordinary packages in which goods are packed for exportation according to the general usage and custom of trade, shall be free of duty.

(*d.*) Provided further, that all such special packages or coverings as are of use or apparently designed for use other than in the importation of the goods they contain, shall be subject to the same rate of duty as would thereon be levied if imported empty or separate from their contents.

That when the Customs tariff of any country admits the products of Canada on terms which on the whole are as favourable to Canada as the terms of the reciprocal tariff herein referred to, are to the countries to which it may apply, articles which are the growth, produce, or manufacture of such country, when imported direct therefrom may then be imported direct into Canada, or taken out of warehouse for consumption therein at the reduced rates of duty provided in the reciprocal tariff set forth in Schedule D.

(*a.*) That any question that may arise as to the countries entitled to the benefits of the reciprocal tariff, shall be decided by the Controller of Customs subject to the authority of the Governor in Council.

(*b.*) That the Controller of Customs may make such regulations as are necessary for carrying out the intention of the two preceding sections.

Inland Revenue.

6. *Resolved*,—That it is expedient to amend section 130 of chapter 34 of the Act 49 Victoria (the Inland Revenue Act), as amended by section 1 of chapter 25 of the Act 58–59 Victoria, by repealing such section and substituting, in lieu thereof, as follows:—

There shall be imposed, levied, and collected on all spirits distilled the following duties of excise, which shall be paid to the collector of inland revenue as herein provided, that is to say:—

(*a*.) When the material used in the manufacture thereof consists of not less than 90 per cent., by weight, of raw or unmalted grain, on every gallon of the strength of proof by Sykes' hydrometer, and so in proportion for any greater or less strength than the strength of proof, and for any less quantity than a gallon, 1 dol. 90 cts.

(*b*.) When manufactured exclusively from malted barley, taken to the distillery in bond, and on which no duty of Customs or excise has been paid, or when manufactured from raw or unmalted grain, used in combinations, in such proportions as the Department of Inland Revenue prescribe, with malted barley, taken to the distillery in bond, and on which no duty of Customs or of excise has been paid, on every gallon of the strength of proof by Sykes' hydrometer, and so in proportion for any greater or less strength, and for any less quantity than a gallon, 1 dol. 92 cts.

(*c*.) When manufactured exclusively from molasses, syrup, sugar, or other saccharine matter, taken to the distillery in bond, and on which no duty of Customs has been paid, on every gallon of the strength of proof by Sykes' hydrometer, and so in proportion for any greater or less strength, and for any less quantity than a gallon, 1 dol. 93 cts.

Also to repeal so much of the Inland Revenue Act and amending Acts as determine the excise duty on vinegar, and to provide that the excise duties thereon and upon acetic acid shall be as follows:—

Vinegar manufactured in whole or in part from spirits in bond, 4 cents per proof gallon.

Acetic acid, produced by the destructive distillation of wood, 4 cents per proof gallon.

Provided, that the Governor-General in Council may establish regulations exempting acetic acid from excise duty in whole or in part, when used in the mechanical arts.

7. *Resolved*,—That it is expedient that a license fee of 50 dols. be collected in each fiscal year from every manufacturer of acetic acid.

9. *Resolved*,—The excise duties hereby fixed and determined shall come into force and effect on and after the 23rd day of April 1897.

[Following these resolutions are Schedules A., B., and C., which contain the duties leviable on all articles imported into Canada, a list of articles admitted free of duty, and of prohibited goods. These schedules it is proposed to publish in a future number of the *Board of Trade Journal*.]

Schedule D. reads as follows:—

"On all the products of countries entitled to the benefits of this reciprocal tariff, the duties mentioned in Schedule 'A' shall be reduced as follows:—

"On and after the 23rd day of April 1897 until the 30th day of June 1898 inclusive, the reduction shall in every case be one-eighth of the duty mentioned in Schedule 'A,' and the duty to be levied, collected, and paid shall be seven-eighths of the duty mentioned in Schedule 'A.'

"On and after the 1st day of July 1898, the reduction shall in every case be one-fourth of the duty mentioned in Schedule 'A.,' and the duty to be levied, collected, and paid shall be three-fourths of the duty mentioned in Schedule 'A.'

"Provided, however, that these reductions shall not apply to any of the following articles, but such articles shall in all cases be subject to the duties mentioned in Schedule 'A,' viz.:—Ales, beers, wines, and liquors; sugars, molasses, and syrups of all kinds, the product of the sugar cane or beet-root; tobacco, cigars, and cigarettes."

Coal in the Pas-de-Calais.

Bd. of Trade J., June 1897, 710.

Her Majesty's Consul at Calais, in a report to the Foreign Office, states that in the department of the Pas-de-Calais the output was, in 1895, 11,110,470 tons, and 11,870,517 tons in 1896. In the Nord the output was, in 1895, 5,059,871 tons, and 5,226,754 tons in 1896. The increase, however, was not sufficient to compensate for the fall in prices from 10 fr. 79 c. to 10 fr. 38 c. per ton. The labour employed in the Pas-de-Calais district was 35,907 hands working in the pits, and 10,176 above ground.

For some years past several companies have been formed to search for coal in the neighbourhood of Calais, but with little or no success till quite recently, when one of these companies found a bed of coal near a small hamlet, called Estrouannes, situated between Wissant and Blanc-Nez, some 12 kiloms. from Calais. The demand for further concessions continues to increase, and boring is actively carried on, and should the efforts be crowned with success, there is no doubt but that Calais would benefit considerably as a port of export.—(*Foregn Office Annual Series, No.* 1899.)

A New Sugar-Cane in the French West Indies.

Bd. of Trade J., June 1897, 711.

In a report to the Foreign Office, Mr. Gustave Borde, Her Majesty's Acting Consul at Martinique, states that the disappointments that the planters were wont to experience in regard to the quantity of sugar-cane reaped, have greatly diminished since the introduction on the sugar plantations of a species of sugar-cane, known locally as the "Cristalline," which is to be met with in Saint Lucia as the "Caledonian Queen." It is generally believed that this new plant is a valuable acquisition, and will be most useful to agriculture. When young it grows very fast, being the reverse of the sugar-cane plants now under cultivation, the growth of which is slow. By its rapid growth the new plant covers ground quickly, and checks the development of the grass that would otherwise impede the spreading of its leaves. It resists more easily the irregularities of the seasons, and is able to sustain very long droughts. The "shot borer" seems to shun its company, or rather, if a few plants are attacked, their vegetation is not in any way impeded. Judging from the experiments tried during the past year particularly, this species of sugar-cane is as as rich in saccharine matter as all the others that have been hitherto cultivated. Hence, wherever the ordinary plant dies out, the planters endeavour to introduce the "Cristalline," the good qualities of which were conspicuously manifested in many localities very seriously affected by the fungus or other cane disease. For many years chemists have been endeavouring to find out the cause of the disease, and have tried various methods of fighting it, but invariably failed.—(*Foreign Office Annual Series, No.* 1897.)

Reduction of Royalties on Phosphates in South Carolina.

Bd. of Trade J., June 1897, 716.

A report from H.M. Acting Consul at Charleston, states that, in accordance with a decision of the South Carolina State Phosphate Commission, the royalty on all phosphate rock mined in that State on and after April 1st, 1897, will be 25 cents currency, equal to about 1*s.* sterling per ton, instead of 50 cents per ton previously charged.

This reduction, however, under the final action of the Commission, just published, will not apply to rock on hand April 1st last, but previously mined, which will be required to pay the former royalty of 50 cents per ton. The reduction in the Carolina royalty has been brought about by the decline in prices and the keen competition in the business resulting from the growth of phosphate mining in Florida, Tennessee, and elsewhere during the past two or three years.

The Mining and Metallurgical Industries of Germany.

Bd. of Trade J., June 1897, 703.

A despatch from the Commercial Attaché at Berlin, encloses memorandum, based on tables in the official *Reichsanzeiger*, relating to the yield of mines and the products of metal works in the German empire.

The following table shows the quantity and value of the output of the various minerals in Germany and Luxemburg in the years 1895 and 1896:—

—	Quantity.		Value.		Average Price per Ton.	
	1896.	1895.	1896.	1895.	1896.	1895.
	1,000 tons.	1,000 tons.	£ 1,000	£ 1,000	Marks.	Marks.
Coal	85,639	79,169	29,652	26,944	6·92	6·81
Lignite	26,767	24,788	3,046	2,900	2·27	2·34
Iron ores	14,162	12,349	2,569	2,053	3·63	3·33
Zinc ores	729	706	851	528	23·32	14·97
Lead ores	154	161	618	646	80·00	80·06
Copper ores	717	633	847	768	23·64	24·28
Silver and gold ores	18	10	119	85	129·09	157·51
Salt (rough)	541	522	731	712	26·88	27·28

The immense increase in coal, amounting to 6,470,000 tons, and valued at 2,708,000*l*., from 1895 to 1896 deserves great attention; and besides this there is for lignite an improvement of 1,979,000 tons, worth 146,000*l*. The increase in iron ores is also large, being 1,813,000 tons, valued at 516,000*l*.

The following table shows the output of iron and other metal works:—

—	Quantity.		Value.		Average Price per Ton.	
	1896.	1895.	1896.	1895.	1896.	1895.
	1,000 tons.	1,000 tons.	£ 1,000	£ 1,000	Marks.	Marks.
Raw iron	6,295	5,417	14,786	11,733	46·98	43·32
Zinc (block)	153	150	2,355	2,081	307·70	277·05
Lead	113	111	1,251	1,113	219·98	200·60
Copper	29	25	1,458	1,163	995·03	902·96

English Cement in the Transvaal.

Bd. of Trade J., June 1897, 718.

H.M. Consul at Lorenzo Marques states that English cement, at one time a promising article of import to the Transvaal, has of late years, it is said, been almost entirely superseded at that port by German cement. This is partly attributed to the fact that the latter is packed in iron drums, which withstand the rough handling and exposure often injurious to the contents of the frailer barrels in which English cement continues to be exported.

Cement, however, is likely to form in the future an ever diminishing article of import into the Transvaal, since a factory, enjoying Government support and a protective tariff, has now been established in that country.

Portland cement might still recover the market were it not for this native product and the high charges levied in its interest by taxation, and an almost prohibitive railway charge.

A New Crisis in German Sugar Production.

U.S. Consular Reps. 53, [197], 167—170.

According to the law passed by the German Parliament on 15th May 1896, the limit of production was raised from 1,400,000 tons, the amount stated in the original bill, to 1,700,000 tons per annum (raw sugar equivalent)—an amount that had been reached only once, and that in the campaign of 1894—95, which led to the crisis of the year following. Over-production—the principal danger to be feared, and, if possible, averted—was thus established by the new law as a permanent possibility. Furthermore, the contingent production authorised was to be double the increased home consumption, and this was distributed among the factories, not on the basis of their average annual production for five years previous, but on the basis of the highest figures reached during two of the three preceding years. As a result, this has stimulated the factories in 1896 to obtain the highest possible output, so as to entitle themselves to the largest share of the bounty. The effect of this has been to give important advantages to the large and wealthy factories over the smaller ones owned by the farmers and capitalists of small means. The new law has also fixed the internal revenue tax on manufacture so low as to have but little value to the State, the revenue yielded being wholly out of proportion to the heavy export bounties to be paid, which have become, more than ever before, a burden to the treasury.

The effects of the new statute have now become apparent. The foreign sugar market responded to the increased export bounty by a proportionate decline in price, so that the German exporter now receives only the same price for his sugar as before, and of this price the German Government pays more and the foreign consumer less than hitherto, so that the net result has been, not to encourage the German sugar industry, but to supply Great Britain, the United States, and other importing countries, with cheaper sugar, and thus further demoralise and weaken the general market.

The outlook is so menacing that the Reichstag will be petitioned to make certain amendments in the existing statute, with the object of revising the method of "Contingentirung" (or additional quota distributed among the factories on the basis of the average annual production of each), so as to restrict instead of stimulating production, and to invite international negotiations for the gradual abolition of all export bounties, which have been proven to be simply a burden on the treasury which pays them for the benefit of non-producing foreign countries.

In conclusion, it is pointed out that if this attempt at revision fails, there will then remain, in the opinion of practical men, but one way out of the dilemma—the organisation of the whole German sugar-producing interest into a syndicate, an organisation which shall, by its own regulations, carefully restrain over-production and be content with what can be done to control prices of sugar by commercial action, and by regulating the supply in home and foreign markets.—J. L. B.

German Beet-Sugar Returns for 1895—1896.

U.S. Consular Reps. 53, [197], 170—172.

The following data are taken from the publications of the Imperial Statistical Bureau and Licht's reports:—

The campaign of 1895–96 proved more favourable than the preceding one. Although the area under cultivation

had been reduced and the yield per hectare diminished, the saccharine contents of the beet were higher, and the average price of raw sugar better than in 1891–95. The value of the total sugar production of 1,615,111 tons in 1895–96 was 363,400,000 marks, against 351,719,000 marks in 1894–95. The total area under cultivation being 376,669 hectares, each hectare yielded, therefore, a gross profit of 965·04 marks, as compared with 796·75 marks in 1894–95. After deducting the working expenses per hectare, the net profit comes out 237·04 marks, against 53·55 marks in 1894–95.

The area under cultivation was reduced from 441,141 hectares in 1894–95 to 376,669 hectares, or about 14⅝ per cent. The yield per hectare was 31 tons, against 32·9 tons in 1894–95; 42·94 per cent. were grown by the factories and 56·06 per cent. bought, as compared with 41·64 per cent. grown and 58·36 per cent. bought in 1894–95.

The number of factories in operation was reduced from 405 in 1894–95 to 397—three new factories having been established and 11 closed, nine permanently and two temporarily.

The beetroot crop was 11,672,816 tons, against 14,521,029 tons in 1894–95, or a decrease of 19·61 per cent.

The number of steam engines and horse-power used in the factories was 5,320 and 97,977 respectively, against 5,324 and 94,952 in 1894–95. The average time required to manufacture the beetroots into raw sugar was 75 days, as compared with 99 days in 1894–95. The saccharine contents were 13·19 per cent.; in 1894–95 they were 12·2 per cent. To manufacture 1 cwt. of raw sugar, 7·58 cwts. of beetroot were required, against 8·2 cwt. in 1894–95. The export decreased from 1,073,590 tons in 1894–95, to 964,963 tons. The home consumption increased from 623,874 tons in 1894–95 to 730,784 tons. This increase is only apparent, and was largely caused by the stipulations of the new sugar law (see preceding report), which increased the home-consumption tax from 18 to 20 marks per double cwt., and thereby caused an extensive withdrawal of sugar from the bonded stores prior to its going into effect.

The average consumption of sugar per head of population was 12·72 kilos., against 10·7 kilos. in 1894–95, which increase, however, for the reason just stated, should be considerably discounted.

The tax on sugar, less export bounties, yielded to the Government 103,701,000 marks, against 85,714,000 marks in 1894–95, which equals a tax of 1·97 marks per head of population, against 1·65 marks in 1894–95.

The total raw sugar production in Europe was as follows:—

Countries.	1895–96.	1894–95.
Germany	1,615,111	1,841,461
Austria-Hungary	791,405	1,055,821
France	667,853	792,511
Russia	783,189	615,058
Belgium	260,050	243,957
Holland	106,829	84,597
Denmark, Sweden, Italy, Roumania, Spain.	168,800	156,000
Total	4,393,537	4,789,405

—J. L. B.

GENERAL TRADE NOTES.

Unification of the Methods of Testing.

A circular, signed by Professor Hermann Wedding, of the Berlin School of Mines, has been issued respecting the proposed establishment at Zürich of an International Society for the Unification of the Methods of Testing Materials of Construction. It is proposed to establish a central laboratory there, in charge of Her von Juptner, and an appeal is made to the members of the Iron and Steel Institute to contribute towards the expenses of maintenance. Herr Krupp, Essen, has subscribed 50*l.* per annum, and the Austrian ironmasters have offered 350*l.* per annum. About 2,000*l.* a year will be needed. Subscriptions may be sent through Mr. Brough, Secretary of the Iron and Steel Institute.

Chemical Imports into France.

Chem. and Druggist, May 29, 1897, 838.

According to the excellent little monthly circular published by the British Chamber of Commerce in Paris, the value of the imports of chemical products into France during the first quarter of 1897 showed an increase upon that of the first quarter of 1896, the figures being respectively 9,117,000 frs. and 8,778,000 frs. The circular in question will be found very useful by firms dealing with France. Specimen copies of it may be had upon application to the Secretary of the Chamber, 25, Bd. des Italiens, Paris.

Phenacetine in U.S.A.

Chem. and Druggist, June 5, 1897, 871.

Judge Lacombe, of the United States Circuit Court, has decreed that Bayer's patents for the manufacture of sulphonal of October 30, 1888, and also for the manufacture of phenacetine of March 26, 1889, are good and valid, and has restrained a traveller named Henry Gill from infringing the patents by selling products which are not made by Bayer.

Disinfectants for Russia.

Chem. and Druggist, May 29, 1897, 858.

The Imperial Russian *Official Gazette* contains a notification to the effect that all disinfectants purchased abroad by Russian imperial or civic authorities will be admitted into Russia free of Customs duty.

The Increasing Demand for Wood Pulp.

Scient. American, **76**, [23], 358.

In the year 1880, which marks the real beginning of the wood-pulp industry, there were, in the State of Maine, seven pulp mills, with capital invested of 440,000 dols., an annual production of 300,000 dols., and 12 paper mills, with a capital of 2,000,000 dols. and a production somewhat in excess of that sum. In 1890 there were 11 pulp mills, with capital invested of 2,695,611 dols., and a production value of 1,518,611 dols. At the present time there are over 13,000,000 dols. invested in pulp and paper mills, giving employment, directly or indirectly, to over 5,000 men. New York state has a daily production of 1,800,000 lb. of wood pulp for books and newspapers; Wisconsin, a daily production of 670,000 lb.; Maine, 665,000 lb.; Massachusetts, 614,000 lb.; Pennsylvania, 403,000 lb.; and New Hampshire, Michigan, Ohio, and Vermont, a somewhat smaller amount. It is estimated that 1,000,000,000 feet of spruce logs are required to supply the mills with raw material. Spruce wood has invariably been employed for making the best white paper, though attempts have been made in Europe to utilise the pine for this purpose. Both the pine and the poplar are more easily worked into ground pulp than spruce, but the woods lack the strength needed for large newspaper sheets, and there is also a difficulty in disposing of the pitch and resin in pine. Hemlock is not suitable, as its fibre is more brashy and has less strength than spruce. The spruce trees are unfit for ordinary paper purposes until they have attained the age of 100 to 150 years.

With regard to the systematic culture and development of spruce trees, it is stated that the German pulp manufacturers, with an area smaller than that covered with spruce trees in New York state, have supplied all the raw material needed for their paper mills, and have annually exported over half a million dollars' worth. The forests are merely thinned out, the old trees being cut down as soon as they reach the proper age; and the results are so satisfactory that the supply of raw material is assured as long as the present system of forestry is continued.—A. S.

BOARD OF TRADE RETURNS.

Summary of Imports.

Articles.	Month ending 31st May	
	1896.	1897.
	£	£
Metals	1,767,629	1,683,518
Chemicals and dyestuffs	461,232	481,832
Oils	581,425	574,728
Raw materials for non-textile industries.	3,483,085	4,143,172
Total value of all imports	33,349,988	36,336,348

Summary of Exports.

Articles.	Month ending 31st May	
	1896.	1897.
	£	£
Metals (other than machinery)	2,951,548	2,954,991
Chemicals and medicines	707,812	743,510
Miscellaneous articles	2,804,901	2,888,967
Total value of all exports	18,835,243	19,322,146

Imports of Metals for Month ending 31st May.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Copper:—				
Ore ... Tons	2,596	1,863	26,190	16,057
Regulus ... „	6,953	7,340	177,108	161,345
Unwrought ... „	4,930	6,383	223,705	316,355
Iron:—				
Ore ... „	464,785	495,831	329,031	366,848
Bolt, bar, &c. ... „	8,386	6,229	65,936	48,138
Steel, unwrought ... „	1,684	3,483	12,955	25,586
Lead, pig and sheet „	13,009	13,216	140,681	156,700
Pyrites ... „	49,343	52,423	79,731	80,902
Quicksilver ... Lb.	1,661,648	58,678	149,594	5,598
Silver ore ... Value £	..	..	90,303	102,513
Tin ... Cwt.	62,871	34,654	189,316	104,891
Zinc ... Tons	6,341	6,296	133,905	105,633
Other articles ... Value £	..	..	179,174	192,952
Total value of metals	..	..	1,767,629	1,683,518

Imports of Chemicals and Dyestuffs for Month ending 31st May.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Alkali ... Cwt.	18,134	25,343	8,410	9,888
Bark (tanners', &c.) „	25,225	30,687	9,734	11,281
Brimstone ... „	77,185	40,590	14,739	9,374
Chemicals ... Value £	..	..	110,407	132,208
Cochineal ... Cwt.	203	286	1,258	1,863
Cutch and gambier Tons	1,208	1,241	29,595	23,830
Dyes:—				
Alizarin ... Value £	..	..	17,933	18,558
Anilin and other „	..	..	41,477	39,034
Indigo ... Cwt.	2,579	2,503	30,784	36,986
Nitrate of potash . „	41,254	38,672	30,264	29,088
Valonia ... Tons	2,871	4,510	29,584	45,326
Other articles ... Value £	..	..	137,047	124,396
Total value of chemicals	..	..	461,232	481,832

Imports of Oils for Month ending 31st May.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Cocoa-nut ... Cwt.	10,156	19,570	11,560	22,604
Olive ... Tuns	2,095	2,145	67,851	70,001
Palm ... Cwt.	109,936	88,842	110,922	89,814
Petroleum ... Gall.	11,467,310	13,414,437	245,882	244,726
Seed ... Tons	2,318	2,428	43,197	48,984
Train, &c. ... Tuns	870	1,246	16,497	17,999
Turpentine ... Cwt.	11,764	1,526	10,966	1,573
Other articles .. Value £	..	..	74,550	79,027
Total value of oils...	..	..	581,425	574,728

Imports of Raw Material for Non-Textile Industries for Month ending 31st May.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Bark, Peruvian .. Cwt.	5,305	3,742	8,804	11,465
Bristles ... Lb.	229,742	359,597	34,330	49,605
Caoutchouc ... Cwt.	29,875	30,191	283,799	330,506
Gum:—				
Arabic ... „	14,672	5,659	39,237	10,056
Lac, &c. ... „	14,964	17,597	79,225	73,890
Gutta-percha ... „	2,919	3,615	22,506	41,207
Hides, raw:—				
Dry ... „	30,820	54,384	76,208	135,219
Wet ... „	37,023	43,113	83,003	94,731
Ivory ... „	624	629	25,491	21,916
Manure:—				
Guano ... Tons	590	2,112	2,344	13,589
Bones ... „	4,727	3,899	18,187	14,460
Nitrate of soda... „	8,750	19,393	67,842	145,209
Phosphate of lime „	21,840	26,947	37,329	39,476
Paraffin ... Cwt.	38,984	49,071	39,671	45,167
Linen rags ... Tons	1,893	2,912	19,950	26,232
Esparto ... „	9,625	19,356	40,939	75,914
Pulp of wood ... „	29,304	34,318	150,700	181,689
Rosin ... Cwt.	62,202	74,516	16,589	17,904
Tallow and stearin „	171,902	175,281	175,895	163,502
Tar ... Barrels	700	5,635	413	2,696
Wood:—				
Hewn ... Loads	190,291	260,395	377,526	519,725
Sawn ... „	471,922	441,780	1,036,972	1,109,236
Staves ... „	12,713	7,053	60,623	38,815
Mahogany ... Tons	3,052	4,681	27,272	43,055
Other articles ... Value £	..	..	758,230	937,908
Total value ...	..	..	3,483,085	4,143,172

Besides the above, drugs to the value of 67,787*l*. were imported, as against 100,120*l*. in May 1896.

Exports of Metals (other than Machinery) for Month ending 31st May.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Brass ... Cwt.	9,769	8,280	38,757	37,952
Copper:—				
Unwrought ... „	50,779	39,303	121,148	101,651
Wrought ... „	25,915	25,568	73,699	77,171
Mixed metal ... „	15,213	15,624	34,297	37,481
Hardware ... Value £	..	..	172,493	179,593
Implements ... „	..	..	122,165	110,599
Iron and steel ... Tons	304,018	343,515	2,051,275	2,125,265
Lead ... „	5,969	6,597	72,839	82,487
Plated wares ... Value £	..	..	24,276	27,125
Telegraph wires . „	..	..	112,215	61,226
Tin ... Cwt.	12,597	11,405	40,803	36,685
Zinc ... „	16,124	11,723	12,223	9,223
Other articles .. Value £	..	..	75,358	68,533
Total value ...	..	..	2,951,548	2,954,991

Exports of Drugs and Chemicals for Month ending 31st May.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Alkali............ Cwt.	374,564	501,695	101,185	128,264
Bleaching materials „	91,723	91,246	31,151	29,463
Chemical manures Tons	21,261	23,670	129,577	134,562
Medicines....... Value £	...	..	95,226	92,710
Other articles... „	..	..	350,673	358,711
Total value	..	..	707,812	743,510

Exports of Miscellaneous Articles for Month ending 31st May.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Gunpowder...... Lb.	1,025,500	512,800	17,288	12,650
Military stores.. Value £	..	..	167,813	127,081
Candles.......... Lb.	2,210,100	2,036,100	32,787	28,196
Caoutchouc ..... Value £	..	..	97,587	94,255
Cement.......... Tons	34,527	41,038	55,993	67,380
Products of coal Value £	..	..	150,445	163,465
Earthenware ... „	..	..	154,729	167,298
Stoneware ...... „	..	..	20,121	14,700
Glass:—				
Plate........... Sq. Ft.	164,844	146,513	10,792	7,588
Flint........... Cwt.	7,121	6,851	17,880	15,238
Bottles......... „	70,332	69,711	33,526	32,233
Other kinds.... „	23,238	20,628	18,980	15,247
Leather:—				
Unwrought .... „	13,168	12,468	118,080	114,567
Wrought ..... Value £	..	..	22,007	31,058
Seed oil.......... Tons	5,349	5,490	102,299	91,261
Floorcloth ..... Sq. Yds.	2,050,100	2,376,800	80,000	96,404
Painters' materials Val. £	..	..	145,668	146,875
Paper .... Cwt.	92,512	89,424	137,530	130,383
Rags............ Tons	4,943	4,451	20,085	26,589
Soap............. Cwt.	68,600	65,487	67,969	70,563
Total value	..	..	2,804,901	2,888,967

Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—PLANT, APPARATUS, and MACHINERY.

Applications.

12,133. E. Robinson. Cooling by evaporation. May 17.

12,224. D. N. Fleming. Improvements in appliances for storing and diffusing compositions for preventing internal crustation or corrosion of boilers and the like. May 18.

12,752. G. Watson. Improvements in furnaces for burning towns' and other refuse; applicable also to furnaces for general purposes. May 24.

12,768. E. Paul. Improvements in, and in apparatus for, the manufacture of salt from brine. May 24.

13,353. W. N. Twelvetrees. Improvements in cleansing, drying, and disinfecting apparatus. May 31.

13,379. J. H. Rosenthal. Improved salinometer. May 31.

13,754. H. Tomkins. Improvements in means and appliances for charging open-hearth furnaces. June 4.

13,868. T. Melvin. Melvin's improved method of evaporating moisture. June 5.

13,908. C. E. Challis. Improved filters and attachments. June 5.

13,893. W. Schmidt. Improved construction of tubes for cooling and heating purposes; specially adapted for brewers, dyers, dairies, and other trade uses; also for condensing steam and vapours. June 8.

14,226. L. Lewis. An improved composition for preventing incrustation in steam boilers. Complete Specification. June 11.

Complete Specifications Accepted.*

1896.

16,306. W. Oliphant. Apparatus for drying solid materials. June 16.

16,552. R. H. F. Finlay. Evaporating apparatus. June 9.

16,659. J. Foster. Apparatus for condensing steam vapour or gases, applicable also as a feed-water heater. June 9.

17,053. J. Foster. Evaporating apparatus. June 2.

17,105. F. D. Cummer. Separation and collection of solid matters held in suspension in aeriform fluids. June 9.

17,862. C. A. Kunzel, jun. Filters. June 16.

17,980. W. L. Wise.—From Solvay and Co. Decanting apparatus. June 16.

18,259. H. A. Jones. Deoxidising furnaces. June 9.

21,203. J. H. W. Ortmann and C. W. C. Herbst. New or improved apparatus for pasteurising liquids. June 9.

28,712. H. B. Wright. Mechanical water purifiers. June 9.

1897.

154. S. Sbail. Muffle furnace. June 2.

7108. W. P. Thompson.—From A. Testelin. Process and apparatus for spraying, distilling, evaporating, or concentrating liquids. June 2.

7534. H. A. Wheeler. A smokeless furnace. May 26.

8482. W. Tattersall. Heating or cooling apparatus. June 9.

9042. T. Fletcher and Fletcher, Russell, and Co., Ltd. Furnaces for use with light hydrocarbons as fuel. June 2.

9941. J. Klein. Apparatus for cooling, concentrating, evaporating, and graduating liquids. May 26.

II.—FUEL, GAS, and LIGHT.

Applications.

12,263. A. Preston. A new or improved apparatus for the manufacture of acetylene gas. May 18.

12,275. W. B. Hartridge. Improvements in the manufacture of artificial fuel blocks, and apparatus for that purpose. May 18.

12,422. A. Apps. Improvements in or relating to the storage of ozone. May 19.

12,440. E. Fessoirat. Improvements in the manufacture of incandescence bodies for illuminating purposes. May 19.

12,556. R. Quatannens-Moens and E. Carreer-Dilger. A new or improved automatic apparatus for the production of acetylene gas. Complete Specification. May 20.

12,591. T. F. Ennis and F. S. Green. Improvements in the utilisation of the refuse obtained from gas works. May 21.

12,619. C. Jorisset and H. N. Ward. Improvements in and relating to apparatus for the generation and burning of acetylene gas. May 21.

12,660. V. von Pindter. Improvements in and connected with apparatus for consuming smoke. May 22.

12,930. Count A. J. De Montby. Improvements in the manufacture of artificial fuel. May 25.

13,081. F. Dresser. Improvements in apparatus for generating acetylene gas. May 27.

13,082. F. Dresser. Improvements in apparatus for generating acetylene gas. May 27.

13,103. F. H. Smith. Improvements in the manufacture of calcium carbide and the like, and in the method of using the same in lamps or generators. May 27.

13,191. F. G. Bartlett. Improvements relating to burners employed in incandescent gas-lighting. May 28.

13,496. F. A. Mitchell. Improvements in acetylene gas generators. Complete Specification. June 1.

13,667. D. Whalley, J. Hacking, and The Ideal Gas Co., Ltd. Improvements in acetylene gas plant. June 3.

13,671. F. H. Smith. An acetylene gas generator. June 3.

13,798. S. S. Bromhead.—From J. Peyré. Instruments in generators for acetylene gas. June 4.

13,899. R. Laughans. Improvements in the manufacture of incandescing media for lighting purposes. June 5.

13,905. E. Soxhlet, F. V. Bergh, A. Borremans, and V. Borremans. Improvements in the process of obtaining lighting, heating, and motive power, and in apparatus therefor. Filed June 5. Complete Specification. Date applied for Jan. 22, 1897, being date of application in Belgium.

14,015. F. Trendel. Improvements in apparatus for producing acetylene gas. June 8.

14,090. E. Jimeno. Improvements in apparatus for the production of acetylene. Complete Specification. June 9.

14,091. E. Jimeno. Improvements in or connected with gas holders for acetylene and other lighting gases. Complete Specification. June 9.

14,208. F. H. Haviland and W. H. Murch. Improvements in apparatus for automatically generating, storing, and supplying acetylene gas. June 11.

14,259. H. Kayser. Improvements in the manufacture of incandescent bodies for gas and other lighting. June 11.

14,260. O. R. Rangabe. Improvements in apparatus for generating acetylene gas. June 11.

14,264. F. de Bal. Improvements in incandescence hoods or mantles for illuminating purposes. June 11.

14,274. H. H. Leigh.—From F. Saldana. Improvements in apparatus for the production of acetylene gas. June 11.

14,275. H. H. Leigh.—From F. Saldana. Improvements in the construction of lamps for generating and burning acetylene gas. June 11.

14,313. G. W. Gaskill and R. F. Reeve. Improvements in apparatus for generating gas by the addition of a solid to a liquid. June 12.

14,339. C. Tellier. Improvements in tubes and other bodies for absorbing or radiating heat. June 12.

Complete Specifications Accepted.

1896.

9047. T. D. Farrall. Method of and apparatus for heating by electricity. June 9.

11,581. G. Mereau and A. Prilhies. Gas lighting. May 26.

12,771. C. Whitfield. Process and apparatus for manufacturing producer and water-gas. June 9.

13,147. J. E. Atkinson and J. M. Labouchere. Automatic apparatus for the manufacture of acetylene gas. May 26.

15,061. G. F. Redfern.—From La Comp. Continentale d'éclairage par le gaz acètylene et ses applications industrielles. Apparatus for the production of acetylene. June 16.

15,962. F. S. Thorn and C. Hoddle. Apparatus for generating, storing, and cooling acetylene gas. June 9.

16,692. W. L. Wise.—From E. Thomson. Manufacture of gas and apparatus therefor. June 9.

16,966. W. Nicholls. Manufacture of incandescence bodies for illuminating purposes. June 9.

16,984. H. Strache. Method or process of and apparatus for removing gaseous iron compounds from water-gas. June 16.

17,228. A. Verley. Apparatus for the production of ozone. June 9.

1897.

5236. C. B. v. Franco. Means for generating, holding, and burning acetylene gas. May 26.

5913. C. E. Alexandre. Apparatus for the manufacture of acetylene gas. May 26.

7921. J. B. de Lery. Burners for incandescence gas lighting. May 26.

7991. A. A. Stephenson. Composition and apparatus for enriching gas. June 2.

8552. H. Cousin. Apparatus for manufacturing acetylene. May 26.

10,186. D. C. Morency. Apparatus for generating acetylene gas. June 2.

10,199. W. P. Thompson.—From The Deutsche Acetylen Gas Gesellschaft mit beschränkter Haftung. Apparatus for developing acetylene gas. June 2.

10,249. W. P. Thompson.—From The Deutsche Acetylen Gas Gesellschaft mit beschränkter Haftung. Apparatus for the production and consumption of acetylene gas. June 16.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Application.

10,665. W. Young and J. Fyfe. Improvements in and relating to retorts for the destructive distillation of shale. June 3.

IV.—COLOURING MATTERS and DYES.

Applications.

12,179. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of ethers of *m*-oxy-*p*-amidobenzoic acid. May 17.

12,180. H. Imray.—From The Chemical Works, Bindschedler. Process for obtaining new dyestuffs of the phthaleine series. Complete Specification. May 17.

12,181. H. Imray.—From The Basle Chemical Works, Bindschedler. Process for obtaining new dyestuffs of the phthaleine series. Complete Specification. May 17.

12,963. L. Lederer. A process for the production of indifferent (neutral) compounds of alkoxylated phenols. Complete Specification. May 26.

12,964. L. Lederer. Process for the extraction of the hydroxylated ethers of phenol from mixtures. Complete Specification. May 26.

13,104. O. Imray. From The Farbwerke vormals Meister, Lucius, and Brüning. Process for the manufacture of brown to black dyestuffs. May 27.

13,216. J. Grossmann. A new or improved non-fermenting indigo vat to produce effects similar to wood and other fermentation vats. May 28.

13,420. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation. Manufacture of a new red basic colouring matter. May 31.

13,709. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Improvements in the production of aromatic aldehydes. June 3.

13,999. R. Holliday and Sons, Ltd., J. Turner, and H. Dean. Improvements in the manufacture of naphthylamine derivatives and colouring matters therefrom. June 8.

14,043. J. E. Bedford and C. S. Bedford. Improvements in the treatment of logwood colouring matter. June 9.

14,132. H. R. Vidal. Improvements in the manufacture of colouring matters. June 10.

Complete Specifications Accepted.

1896.

14,955. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. The manufacture and production of new products and colouring matters from aromatic nitro derivatives. May 26.

16,449. R. Vidal. Colouring matters. June 9.

16,582. T. R. Shillito.—From J. R. Geigy and Co. Production of azo colours. June 16.

17,064. I. Levinstein and Levinstein, Ltd. Production or manufacture of new colouring matters. June 9.

17,065. I. Levinstein and Levinstein, Ltd. Manufacture or production of new colouring matters. June 16.

17,207. O. Imray.—From G. H. Weiss. Manufacture of amino-oxycarbonilcarbonic acid and azo dyestuffs therefrom fixed on chrome mordants. June 2.

17,226. E. H. Temple. Improved ink. June 2.

17,590. S. Pitt.—From L. Cassella and Co. Manufacture of primary disazo dyestuffs. June 16.

17,591. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Production of dyestuffs on fibre. June 16.

18,720. T. R. Shillito.—From J. R. Geigy and Co. Production of red colouring matters. June 16.

V.—TEXTILES, COTTON, WOOL, SILK, Etc.

Applications.

12,511. C. L. Bachelerie. Improvements in the treatment of textile plants to remove the fibre therefrom. Complete Specification. May 20.

13,256. L. Johnstone and D. Scott. Improvements in or relating to treating, preparing, or applying cellulose for dressing or finishing textile fabric, or for other purposes. May 28.

13,274. E. Neuweiler. Improvements in the manufacture of yarns. May 28.

Complete Specifications Accepted.

1896.

13,273. A. S. Oetzmann and S. J. Narracott. Manufacture of inlaid fabrics for covering floors and other surfaces, and apparatus therefor. June 16.

13,815. W. Warburton. Extracting vegetable fibres. June 2.

19,951. J. McCreath. Wool drying. June 9.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

Applications.

12,940. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Improvements in dyeing vegetable fibre with indigo. May 25.

13,088. H. Zublin and A. Zuigg. An improvement or improvements in white and colour discharge on enlevage printing of dyed para-nitraniline red and related substances. Complete Specification. May 27.

13,138. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. An improved process for producing fast shades on cotton. May 27.

13,175. J. Grossmann. A new or improved method of producing indigo resist and similar styles. June 1.

13,591. J. Holden and S. Woods. Improvements in or relating to the manufacture of embossed or raised paper for covering walls, and for other purposes. June 2.

13,916. B. Willcox.—From S. Wallach and Co. and C. Scheen. Improvements in fixing insoluble colouring matters on fibres or fabrics. June 5.

14,319. V. Floquet and L. Bonnet. Improvements in bleaching and disinfecting fibrous materials, and apparatus for that purpose. June 12.

VII.—ACIDS, ALKALIS, and SALTS.

Applications.

12,266. The Compagnie Generale l'Aluminc Soc. Anonyme. A new process of manufacture of double chloride of aluminium and soda. Complete Specification. Filed May 18. Date applied for Nov. 19, 1896, being date of application in Belgium.

12,274. S. Rosenblum, S. Rideal, and The Commercial Ozone Syndicate, Ltd. Improvements in the manufacture of hydrogen peroxide and in apparatus therefor. May 18.

12,387. A. J. Bambridge and G. Weddell. Improvements in the manufacture of bicarbonate of ammonia; also other compounds of carbon dioxide and ammonia and bicarbonate of soda. May 19.

12,534. A. R. Davis. Improvements in the manufacture of sodium chlorate. May 20.

12,768. E. Paul. Improvements in and in apparatus for the manufacture of salt from brine. May 24.

12,817. F. Blum. Process for the production of divisional products of halogen-albumen by means of alkalis. May 24.

12,952. E. G. Scott. Improvements in the manufacture of acetic acid. May 25.

12,982. W. Mills. Improvements in manufacturing the silico fluoride of ammonium. May 27.

13,280. H. Imray.—From L. Buchner. Process for producing alkali phosphates. May 28.

13,519. W. Feld. Process and apparatus for obtaining barium oxide and barium hydroxide from barium carbonate. June 1.

13,550. A. Royers. Process for the extraction of chlorine from halogen salts in solution in water, and the transformation of the light or heavy corresponding metal either into oxidised salts or oxidised salts of chlorine, and for the production with alkaline sulphates of the corresponding hydroxide salts. June 1.

13,551. W. Majert. A new process for the production of ammonium bichromate. Complete Specification. June 1.

13,825. H. Steinem and K. S. Murray. Improvements in and apparatus for the manufacture or production of carbonic acid gas. June 4.

Complete Specifications Accepted.

1896.

16,255. L. M. Bullier. Manufacture of carbides of the earth metals and alkali-earth metals. May 26.

1897.

2308. G. F. Zacher. Process for producing oxalic acid. June 9.

7192. A. Schmidt. Process and apparatus for the production of the acetates of lead, copper, and the like. June 9.

10,501. A. Feldmann. A process for removing carbonic acid and hydrogen sulphide from ammoniacal liquor. June 9.

VIII.—GLASS, POTTERY, and ENAMELS.

Application.

12,759. W. Wachter. Method of transferring metachromo types upon ceramic objects before glazing the latter. May 24.

Complete Specifications Accepted.

1896.

14,901. K. W. R. Goerisch. Manufacture of sulphate glass. June 16.

14,953. H. Propfe. Manufacture and production of water-glass with valuable by-products of the said manufacture. May 26.

1897.

6184. E. Walsh, jun. Plate-glass annealing kilns. June 2.

10,130. H. de Wit. Manufacture of enamelled ceramics with a slaggy biscuit covered over with coloured enamels. June 16.

11,735. G. Tasche and F. Sporer. Glass cooling or annealing ovens. June 16.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, and CEMENTS.

Applications.

13,026. W. Chrometzka. Improvements in fireproof ceilings. Complete Specification. May 27.

13,821. G. F. Lebioda. Improvements in apparatus for drying and hardening wood. Complete Specification. Filed June 4. Date applied for Nov. 14, 1896, being date of application in France.

14,068. J. A. Fisher. Improved fireproof ceilings and partitions. Complete Specification. June 9.

14,295. A. Patrick. Improvements in the manufacture of Portland or similar cement. June 12.

14,299. A. D. Elbers. Process of treating blast-furnace slag for use in cement, mortar, or the like. June 12. Date applied for Nov. 25, 1896, being date of application in United States.

Complete Specifications Accepted.

1896.

15,316. S. P. Davies.—From G. Frederick. Composition for use in streets, roads, pavements, and the like, and on the surfaces of wood and metal. June 2.

1897.

9315. C. Richardson. Asphaltic pavements and the like. June 2.

X.—METALLURGY, MINING, Etc.

Applications.

12,325. J. C. Bull. Improvements in alloys. May 18.

12,442. C. D. Jenkins. Improvements in the production of aluminium and in apparatus therefor. May 20.

12,691. G. Weil, E. Quintaine, and C. Lepsch. Nickelling, silvering, gilding, &c. of aluminium. May 22.

12,796. F. W. Dupré. Improvements in or relating to the extraction of gold and silver from ores. May 24.

12,832. J. Golding. Improvements in furnaces specially applicable for the manufacture of lead. May 24.

13,302. C. P. Shrewsbury and H. R. Gregory. Improvements in means or apparatus for extracting gold and other metals from solutions containing such metals. May 29.

13,308. J. Parry and G. H. Llewellyn. Improvements in the manufacture of steel. May 29.

13,672. W. E. Harris. Method of treating and annealing sheet metal. Complete Specification. June 3.

13,739. J. P. van der Ploeg. Improvements in or relating to the treatment of antimonial and arsenical ores. June 4.

14,007. B. K. Jamison. Improvements in apparatus employed in the face-hardening of armour-plates or other articles of steel, or of steel alloyed with other metals. June 8.

14,325. C. C. E. Bohne. Improvements in the treatment of slag resulting from the smelting of tin ore. June 12.

Complete Specifications Accepted.

1896.

6654. A. Karyscheff and S. Demmenic. A new pyrochemical process and apparatus for the direct production of iron and other metals from their ores. June 2.

11,245. Davies Bros. and Co., Ltd., E. A. Davies, and S. T. Thomas. Preparing metal sheets for purpose of galvanising them or otherwise coating them with metal or metallic alloy. May 26.

11,839. A. S. R. Robinson. Method and machinery or apparatus for extracting gold from crushed ore or material containing it. June 2.

12,056. M. L. Ross.—From E. Oberle. Manufacture of refractory metallic threads. June 9.

12,396. E. A. G. Street. Fusing chromium and similar metals, and manufacture of alloys of such metals. May 26.

12,467. E. A. G. Street. Manufacture of alloys. May 26.

13,169. B. Mohr. Treatment of sulphide ores for the separation of zinc from the lead and other metals. June 16.

15,041. L. Doig.—From The Rossel and Erwin Manufacturing Co. Annealing metals. May 26.

15,699. G. Duncan. New metallic alloy. May 26.

15,749. O. Imray.—From E. F. Turner. Improved regenerative process for the treatment of sulphide ores. June 16.

16,312. A. M. Clark.—From E. A. Ashcroft. Treatment of solutions of ores containing zinc, for the recovery of zinc as oxide. June 2.

16,685. C. Vautin. Production of metals and alloys. June 16.

17,493. C. W. H. Gopner and H. L. Diehl. Recovery of gold and silver from their solutions. June 16.

1897.

6151. F. A. Gooch. Processes for reducing aluminium. June 9.

11,602. E. E. von der Linde. Treatment of tin oxide in the form of powder, sludge, or paste, for the smelting thereof. June 16.

XI.—ELECTRO-CHEMISTRY and ELECTRO-METALLURGY.

Applications.

12,544. H. P. M. Brunel. Improvements in the process and apparatus for the electrolysis of alkaline solutions and the like. May 20.

12,549. W. Majert. Improvements in plates for accumulators. Complete Specification. May 20.

12,665. H. Tee. The combined generation and application of electricity supply and the manufacture of salt. May 22.

12,675. R. Krayn and C. Koenig. Improvements in galvanic batteries. Complete Specification. May 22.

12,827. V. Jeanty. Improvements in electric primary and secondary batteries. Filed May 24. Date applied for Nov. 19, 1896, being date of application in France.

13,100. C. N. Gauzentés. Improvements in or connected with primary or galvanic electric batteries. May 27.

13,363. H. Nehmer and F. Nehmer. Improvements in apparatus for distributing gas, liquids, solids, or particles of solids by electricity. May 31.

13,689. E. A. Jahncke. Improvements in or relating to electric batteries. June 3.

13,735. W. Rowbotham. Improvements in primary batteries. June 3.

13,992. W. Mather. Improvements in anodes for electrolytic cells. June 8.

14,022. R. Memmo. New electric furnace for the manufacture of carbide of calcium, with continuous operation, and with water covering for the gas. June 8.

Complete Specifications Accepted.

1896.

11,133. G. Bell and G. W. Bell. Apparatus for electrolytic decomposition of liquids. May 26.

13,646. W. H. Smith and W. Willis. Electric accumulators or storage batteries and manufacture of plates or electrodes therefor. June 9.

14,393. P. Jensen.—From H. C. F. Stormer. Anodes for electrolytic purposes. June 9.

16,270. M. O. A. Garreau. Secondary electric batteries. May 26.

16,516. J. Vaughan-Sherrin and H. H. Sherrin. Accumulator batteries. June 2.

17,161. S. Hammacher. The manufacture of an improved active material for the plates or electrodes of electric accumulators. June 9.

17,879. V. M. Cornely and L. C. H. Dantel. Electric batteries. June 16.

1897.

9563. H. T. Cheswright. Plates for secondary batteries or accumulators. May 26.

9913. E. Marckwald. A manufacture of electrodes for electric accumulators. May 26.

9914. E. Marckwald. A manufacture of electrodes for electric accumulator plates. May 26.

10,254. W. A. Boese. Process for the manufacture of accumulator plates. June 2.

XII.—FATS, OILS, AND SOAP.

Applications.

12,456. R. T. Cardell. Improvements in soaps and all toilet preparations. May 20.

13,807. I. Kohn and M. Kohn. Improvements in or relating to transparent soap blocks or tablets containing inscriptions. Complete Specification. June 4.

14,238. J. Davidson and C. G. Hepburn. Improvements in apparatus for refining fats and oils. Complete Specification. June 11.

14,241. L. Grote. Bleaching soap. June 11.

Complete Specifications Accepted.

1896.

16,129. C. H. Simpson and F. H. T. Allan. Process for the purification of spent lyes. June 2.

24,090. T. H. Bateman. New or improved liquid soap. June 9.

1897.

9311. F. Rapp-Rosenthal. Process for the production of oil from peat. June 9.

10,526. J. R. Whiting and W. A. Laurence. Process and apparatus for deodorising oil. June 2.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

13,829. H. C. P. Graves. A new or improved compound for use as a substitute for india-rubber, leather, or other substances, or for any other purposes to which it may be applicable. May 24.

13,534. E. F. von Wilmowsky. Improvements in purifying gutta-percha and similar matters or gums. Complete Specification. Filed June 1. Date applied for Nov. 4, 1896, being date of application in United States.

14,105. F. Boyling, C. L. Garland, and A. Ogden. An improved enamel paint and method of applying the same. Complete Specification. June 9.

14,262. H. E. Serullas. An improved process for obtaining gutta-percha. June 11.

14,272. P. H. Hansen and J. K. Westengaard. Improvements in the manufacture of casein compounds for electric insulating and other purposes. Complete Specification. June 11.

Complete Specification Accepted.

1896.

17,031. A. M. Clark.—From W. Hampe and C. Schnabel. Manufacture of zinc pigments. June.

XIV.—TANNING, LEATHER, GLUE, and SIZE.

Applications.

12,326. S. Herzberg. A new or improved vegetable glue and a process for manufacturing the same. Complete Specification. May 18.

12,449. P. G. Sanford. Improvements in the treatment of blood and other albuminous matter, both animal and vegetable, for use in manufactures and the arts. Complete Specification. May 20.

12,450. P. G. Sanford. Improvements in the treatment of tanning liquids for clarifying, bleaching, or decolorising them. May 20.

12,712. H. Bremer. An improved process for the solution of organic albumenous substances and for the production of soluble preparations of albumen. May 22.

13,310. J. B. Scammell. Improvements in artificial leather compounds. May 29.

13,459. A. Hollings and J. Young. Improvements in apparatus for tanning or treating hides or skins and leather. June 1.

13,778. W. Sagar, sen., A. P. Sagar, and W. Sagar, jun. Improvements in the method of and apparatus for removing grease or fatty matter from sheep skins and other leather and the like. Complete Specification. June 4.

13,997. W. Turner. Improvements in means or apparatus for working or treating skins, hides, or leather. June 8.

14,052. J. E. Chevallot. The manufacture of new iron compounds and their application to the preparation of leather. Filed June 8. Date applied for Dec. 8, 1896, being date of application in France.

14,076. C. J. Dunn. Improvements in or relating to the tanning of leather. June 9.

Complete Specifications Accepted.

1896.

13,096. T. H. Cobley. Treatment of waste substance for the production of leather board and material suitable for being pressed into form for decorative and other purposes, or for use in substitution for leather floorcloth and the like. June 16.

1897.

11,111. E. O. Daniel. Preparation for and tanning of hides and skins. June 9.

XV.—AGRICULTURE and MANURES, Etc.

Application.

13,811. I. Kovacs. An improved process for making a consistent friable artificial manure. Complete Specification. June 4.

Complete Specifications Accepted.

1896.

16,861. R. Tuggener. Apparatus for turning fæcal matter into manure and for disinfecting water-closets. May 26.

1897.

9704. C. H. Thompson. Manufacture of improved fertilised and fertilising materials. June 2.

XVI.—SUGARS, STARCHES, GUMS, Etc.

Applications.

13,357. W. Powell. Improvements in or connected with the manufacture of sugar. May 31.

13,358. W. Powell. Improvements in or connected with the manufacture of sugar. May 31.

13,962. M. von Schmidt. A process to dissolve gum which is otherwise indissoluble. June 8.

Complete Specifications Accepted.

1896.

16,009. W. Field. Extracting sugar from molasses by means of barium hydroxy-sulphide, and recovering the same from the resulting by-products. June 16.

16,441. W. Feld. Extracting sugar from molasses by means of barium hydroxy-sulphide, and recovering the latter. June 9.

17,536. E. J. Mills. Formation and preparation of soluble colloids of the gelatine class. June 9.

1897.

9108. A. J. Boult.—From C. M. Higgins. Adhesive compounds. May 26.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

12,958. H. Krausz and L. Vass. Process for utilising the waste of hops and malt of breweries. Complete Specification. May 25.

13,312. P. F. Van der Gucht and A. Kreps. New or improved malting apparatus. May 29.

13,435. A. J. Boult.—From G. R. Besser. Improvements in apparatus for treating or maturing fluids containing alcohol. May 31.

13,722. J. Goodfellow. Manufacture of nutritious products from yeast. June 3.

14,106. N. Bendixen. Improvements in propagating apparatus for developing pure culture of yeast and bacteria. Complete Specification. June 9.

Complete Specifications Accepted.

1896.

16,671. J. Dunn. Treating barley for alimentary purposes, including brewing and distilling. June 2.

1897.

5582. L. Prochazka and E. Frischauer. Process and apparatus for mashing and brewing. May 26.

10,180. M. van Look. Process for the preparation of mead. June 2.

XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

Applications.

A.—Foods.

12,731. R. G. Nash. Improvements in the aeration and preservation of milk, and apparatus therefor. May 22.

13,141. H. Higgins. Improvements in or relating to the treatment of separated milk for alimentary purposes. May 27.

13,142. H. Higgins. Improvements in or relating to the treatment of separated milk for alimentary purposes. May 27.

13,143. H. Higgins. Improvements in or relating to the treatment of separated milk for alimentary purposes. May 27.

13,985. E. B. Watson. Improvements in enriching vegetable and other foods. June 8.

B.—Sanitation.

12,984. F. P. Candy. Improvements in the connection with the purification of sewage and polluted water. May 27.

13,400. W. D. Scott-Moncrieff. Improvements in or relating to the purification of sewage, and apparatus therefor. May 31.

13,429. H. McPhail. Improvements in the purification of liquids. May 31.

14,058. W. J. Lomax and T. Aspinall. Improvements in or relating to the treatment and purification of sewage and other polluted waters and liquids. June 9.

Complete Specifications Accepted.

A.—Foods.

1896.

11,637. A. J. Vause. An improved process for preserving milk and certain other classes of fresh foods by sterilisation. May 26.

17,720. H. M. Amos. Fruit evaporators. June 16.

1897.

6065. H. Humbser. Process for drying substances. June 2.

B.—Sanitation.

1896.

15,456. E. Sergeant. Combined apparatus for burning refuse and disinfecting. May 26.

XIX.—PAPER, PASTEBOARD, Etc.

Application.

12,208. W. A. Hall. Improvements in the manufacture or treatment of paper. May 17.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

Applications.

12,399. S. Smithson. Improvements in or relating to machinery or apparatus for treating myrobolams for obtaining extracts therefrom. Complete Specification. May 19.

13,055. W. Simpson. New or improved method of preparing fruit or other essences and extracts in tablet form. May 27.

13,214. J. B. Cohen. Improvements in the method of preparing aldehydes and ketones from alcohols. May 28.

13,917. B. Willcox.—From C. Goldschmidt. The manufacture or production of new pharmaceutical products. June 5.

13,993. H. Imray.—From F. Hoffmann, La Roche, and Co. Manufacture of bismuthoxyiodide pyrogallate. June 8.

14,305. M. Otto. Improvements in apparatus for extracting the essential matters contained in aromatic plants, and analogous purposes. June 12.

14,328. J. E. T. Woods. Further improvements in the manufacture of substitutes for camphor, called camphorete, and products made therefrom. Complete Specification. June 12.

Complete Specifications Accepted.

1896.

14,928. H. Imray.—From Harmann and Reimer. Process for the manufacture of vanilline from iso-eugenol and eugenol. June 2.

17,177. J. J. A. Trillat. Production of vapours of formic aldehyde and apparatus therefor. June 16.

XXI.—PHOTOGRAPHY.

Applications.

12,922. T. C. Porter. A new or improved sensitive plate or film, and a method of producing, developing, fixing, and toning the same. May 25.

XXII.—EXPLOSIVES, MATCHES, Etc.

Applications.

12,701. F. Render. Improvements in explosive cartridges. May 22.

13,064. S. Rolfe. Improved method of detonating explosive compounds. May 27.

13,552. W. Majert. A new explosive compound. Complete Specification. June 1.

13,611. G. de Wolf. An improved method of manufacturing gunpowder. June 2.

13,624. F. Render. Improvements in apparatus for the manufacture of cartridges of explosive substances for blasting and similar purposes. June 2.

13,724. E. A. G. Street. Improvements in or relating to the manufacture of explosives and explosive substances. June 3.

13,822. S. Fulop and J. Leckovic. An improved blasting powder. June 4.

14,196. F. Volpert. Improvements in the manufacture of explosives. Complete Specification. June 10.

14,271. F. J. Maubeuge. Improvements in combined time and percussion fuses. June 11.

Complete Specifications Accepted.

1896.

14,320. C. Laun.—From A. Yernander. Safety fuse or quick-match. June 2.

1897.

9396. L. Gathmann. Safety fuses for high explosive shells. June 16.

Printed and Published by Eyre and Spottiswoode, East Harding Street, London, E.C., for the Society of Chemical Industry.

THE JOURNAL

OF THE

Society of Chemical Industry.

A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

No. 7.—Vol. XVI.] JULY 31, 1897. [Non-Members 30/- per annum; Members 21/- per Set of extra or back numbers; Single Copies (Members only) 2/6.

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NOTICES.

The Society's Office will be closed from August 30th to September 14th next, and the General Secretary will feel obliged if members who have changes of address to communicate will send them in before that date.

Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, part of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows:—

To Members (see Rules 25 and 27) and Past Members (see Rule 30); Libraries, Corporations, and Exchanges on the Society's List	Each copy 10*s.*
To Subscribers	„ 12*s.* 6*d.*
To others	„ 15*s.*

B

Jubilee Honour List.

Her Majesty has been pleased to confer the honour of knighthood on George J. Smith, Esq., of Tuckingmill, Cornwall.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. Eyre and Spottiswoode, the Society's printers and publishers, to whom all communications respecting them should be addressed. The circulation of the Journal is now more than 3,000 per month.

LIST OF MEMBERS ELECTED 14th JULY 1897.

Ablett, A. W., Rand Central Ore Reduction Co., Box 1891, Johannesburg, S.A.R., Cyanide Works Manager.
Ackermann, Eugene, 132, Alderney Street, London, S.W., Analytical Chemist and Civil Engineer.
Beaver, Chas. J., c/o W. T. Glover and Co., Salford, Chemist.
Davis, Wm. A., Merton House, Braintree, Essex, Chemist.
Douglas, Geo., Bowling Dyeworks, Bradford, Yorks, Dyer.
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Pollak, Dr. Fritz, 12, Princess Road, Crumpsall, Manchester.
Smith, James, 14, Mersey Road, Aigburth, Liverpool, Analytical Chemist.
Turney, Fred. N., Whitemoor Leather Works, Nottingham, Leather Dresser.
Wilson, E. Siddons, Arthog, Goddington Road, Strood, Kent, Chemist.

LIST OF MEMBERS ELECTED 26th JULY 1897.

Berg, Julius, Elsaesischer Petroleum Gesellschaft, Godramstein, Pfalz, Germany, Oil Refinery Manager.
Burland, Lt.-Col. Jeffrey H., 824, Sherbrooke Street, Montreal, Canada, Paper and Card Manufacturer.
Dancer, Wm., 466, Chester Road, Old Trafford, Manchester, Analytical Chemist.
Duckham, Alex., 40, Crooms Hill, Greenwich, S.E., Works Chemist.
Eckford, Geo. S., Dacre House, Arundel Street, Strand, W.C., Scientific Secretary and Chemist.
Hunt, Edmund, 121, West George Street, Glasgow, Patent Agent.
Skvortzoff, Basil N., Imperial Technical School, Moscow, Russia; and 33, Guildford Street, Russell Square, W.C.; Chemical Technologist.
Wardle, Sir Thos., Leek, Staffordshire, Silk Dyer.

CHANGES OF ADDRESS.

Bower, Frank, l/o Marylebone Road; Truman's Brewery, Spitalfields, E.
Buck, C. A.; Journals to 521, Locust Street, South Bethlehem, Pa., U.S.A.
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Farrell, Frank, l/o Altrincham; 37, Crown Road, Great Yarmouth.
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Hogg, A. F., l/o Cliffe Terrace; 13, Victoria Road, Darlington; and Journals, until further notice, to 134, Birchanger Road, South Norwood, S.E.
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Rae, Roderic H.; Journals to 113, Sotheby Road, Highbury Park, N.
Read, E. J., l/o Bedford; 49, Aston Street, Iffley Road, Oxford.
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Waterhouse, Col. J., l/o Calcutta; Oak Lodge, Court Road, Eltham, Kent.
Williamson, J. A., l/o Pitsea; 81, Cheverton Road, Upper Holloway, N.
Willson, Thos. L., l/o New York; St. Catherine's, Ontario, Canada.
Winser, P. J., l/o Greendale; The Acres, Bebington, near Birkenhead.
Winstanley, Henry, l/o Hayfield; Chadswell, near Clitheroe, Lancashire.

CHANGES OF ADDRESS REQUIRED.

Fogg, J., l/o 11, Claude Villas, Grove Vale, East Dulwich.
Wilkinson, Dr. S., l/o The Grove Co., Ltd., Marsh Gate Lane, Stratford.

Deaths.

Hills, Henry, Chemical Works, Deptford, S.E. June 29.
Hodgkinson, J. H., c/o E. Potter and Co., Dinting Vale, Glossop. June 21.

PROCEEDINGS

OF THE

SIXTEENTH ANNUAL MEETING.

President: Dr. EDWARD SCHUNCK, F.R.S.

The Annual Meeting of the Society of Chemical Industry was held in Manchester on Wednesday, July 14, in the Chemical Theatre of the Owens College. Dr. Edward Schunck, F.R.S., President of the Society, was in the chair, and the attendance, representative of all parts of the country, was large. At the opening of the proceedings the Secretary (Mr. C. G. Cresswell) announced that apologies for non-attendance had been received from Mr. E. Rider Cook (Hon. Treasurer), Sir Robert Pullar, Sir Henry Roscoe, Mr. David Howard, Mr. R. Forbes Carpenter, Mr. G. N. Stoker, and Dr. Messel.

The minutes of the previous meeting, held in Carpenters' Hall, London, on July 15, 1896, were read, confirmed, and signed by the President.

Appointment of Scrutators.

On the motion of Dr. Benj. H. Paul, seconded by Dr. J. Lewkowitsch, Dr. J. H. Bowman and Mr. H. C. Clanahan were appointed scrutators.

The President then declared the ballot closed.

Report of Council.

The Secretary read the report of the Council, which was as follows:—

We have the honour to report that the number of members on the register is 3,037, as compared with 2,971 at the last Annual Meeting. During the year, 226 new members have been elected, as compared with 237 last year; while the loss has been 162, as compared with 161 last year. Among the latter are 23 deaths:—J. M. T. Anderson, J. Ascough, R. W. Peregrine Birch, Jos. Cunliffe, H. E. Dee, L. Demuth, R. Fulton, A. Goskirk, Josiah Hardman, Dr. Sydney J. Harris, Peter Hart, Dr. H. Kunheim, W. Martyn, A. H. Mason, Frank Mead, Jno. Molineux, W. A. Rowell, Fred. Scott, T. Shimidzu, A. J. Tyrer, G. M. P. Vary, Alex. Walker, and Jos. D. Weeks.

During the past session, 101 papers have appeared in the Journal, as compared with 92 last year. The excess of revenue over expenditure, as may be seen from the Treasurer's statement in the last number of the Journal, amounted last year to 1,011*l.* 3*s.* 11*d.*, as against 162*l.* 3*s.* the year before.

This large difference is accounted for by the fact that various economies mentioned in the last Report have been effected during the year. There has been a reduction in most of the items of expenditure, the principal saving being in Journal expenses, both publishing and editorial. The increase in the receipts is, however, largely due to the amounts received as subscriptions to the Collective Index, which will be required when the index is published; so that there will not be such a large balance of revenue over expenditure at the end of the present year.

The production of the Collective Index has involved more labour than was anticipated. Experience gained during the progress of the work has necessitated partial recasting, to the great improvement of the index as an aid to research. The Council feels confident that on completion the delay in the production of the work will be justified.

The following chairmen of Sections retire this year:—Dr. John Clark (Scotland) and Mr. F. J. R. Carulla (Nottingham), and are succeeded by Mr. George Beilby and Mr. Jas. O'Sullivan. Mr. H. R. Procter retires from the secretaryship of the Yorkshire Section. His successor is not yet appointed. The Council acknowledges the debt of the Society to these gentlemen for their efforts on its behalf.

In the session of 1896 a committee of Parliament reported in favour of some amendment of the law in relation to the adulteration of food and drugs. In view of promised legislation, the Council appointed a committee to take such action at the proper time as might be found necessary to secure the Society's representation upon the Board of Reference proposed to be created by Parliament.

In October last a special committee was appointed to co-operate with the London Chamber of Commerce and other bodies in making enquiries as to what further concessions by the authorities in regard to the use of spirit were required in the interests of British manufactures. Several meetings of the joint committee were held, and eventually a conference took place with the officials of the Excise Department of the Inland Revenue, which it is hoped will have satisfactory results.

Dr. F. Hurter proposed and Dr. G. Schack-Sommer seconded the adoption of the report.

Dr. Stevenson Macadam said the report referred to the saving in expense in connection with the publication of the Transactions. He found that ten years ago the cost came to 13*s.* per page, but the figure had increased from that to 15*s.*, then to 21*s.*, and now it was 25*s.* per page of printed matter in the Transactions. There was a great increase in the expenses connected with the editorial department. As to the publication department, he did not find such an anomaly. From first to last the expense had been almost exactly 30*s.* per page. He desired at the same time to draw attention to the anomalous position in which the Publication Committee stood towards the Society. He was quite aware that he could not take a formal motion on this matter, because it was not till the 1st of July that he received a definite answer from the Council to the communication he submitted to them. The question he now raised had especially to do with the nomination, appointment, and retirement of members of the Publication Committee. He could not directly call the Publication Committee in question, because they were simply a branch of the Council, but he would say that the Committee should come more directly under the command of the Society itself. He wished to ask why such a rule as applied to the Council did not apply to the Committee. Of the members of the Publication Committee now acting he found that nine were members 15 years ago; of the Committee of 10 years ago there remained 14; and of six years ago, 23. No doubt they had rendered good service, but the effect was that the Committee was a standing committee and, practically, did not retire. Consequently the Committee was not properly responsible to the Society. He moved, as a recommendation, that the Council should devise rules whereby the nomination, appointment, and retirement of the Committee should be carried on on the same lines as the appointment, nomination, and retirement of members of the Council. This would enable them to get fresh blood on the Committee.

Mr. A. Ernest Powell seconded the motion.

Mr. Thos. Tyrer said he thought Dr. Macadam and Mr. Powell were under a misapprehension. It was true that they had members of the Publication Committee who were old members. But he believed that in every Society provision was made for continuity. While, undoubtedly, the Committee should change, yet the change should be slow and steady. Committees were appointed annually by the Council, and the Council was responsible to the Society for their doings. Any matter of controversy was referred to the Council. The Publication Committee retired every year, but the attendances were called out before the re-election. Correspondence as to slip proof by a member of Publication Committee counted as an attendance. It would be monstrous to expect a man living in Glasgow always to come down to London. It was from communications of this character that very valuable amendments were often made. The point which Dr. Macadam had now so courteously raised was not that which he referred to the Council. With regard to the analysis of expenses, he was quite sure that a careful comparison of all the facts and figures would show that real economies had been made and that Dr. Macadam's criticism would not be up held. They could not expect remuneration to remain stationary when the staff served them well and loyally. It was an invidious task to cut contributions down, but it had to be, and it was done most carefully. If, year by year, there was to be a competition for places on the Publication Committee, the work would probably not be as well done as it was now. Meanwhile, the Council would undoubtedly give consideration to what Dr. Macadam had said on both points.

Mr. F. J. R. Carulla pointed out that the pages of the Journal contained nearly double the matter of the first six volumes.

Dr. S. Macadam said he would be satisfied if the Committee were elected in the same manner as the Council, but he was in the meantime content to accept the assurance of Mr. Tyrer. All he asked was that his recommendation should appear in the Journal, and on that understanding he withdrew it.

The report was then adopted.

Treasurer's Report.

In the absence of the Honorary Treasurer the report was read by the Secretary. It has already appeared in these columns. On the motion of Mr. T. Fairley, seconded by Mr. H. Brunner, the report was adopted.

The President then delivered the following address :—

President's Address.

Some years ago a meeting was held within the walls of the building in which we are now assembled for the purpose of forming a society for the promotion of technical as distinguished from scientific chemistry. The Society was then founded and its general scope and object were defined ; the name it now bears, that of the Society of Chemical Industry, being at the same time given it. I was present at that meeting, and made a little speech in favour of its object. I may therefore be considered in a sense one of the founders of the Society. In subsequent years I was unfortunately not in a position to attend the meetings of the Society or its sections, nor to assist at its deliberations, but I have always followed the record of its proceedings and its progress with interest. The history of this Society has been in all respects one of continuous success, as may be seen by anyone looking over the 15 volumes of the Journal in which our achievements are recorded.

In taking such a retrospect, one thing, I think, must strike everyone ; that is, the high tone assumed by the Society and reflected in its Journal, the entire devotion to the objects for which it was established, the total absence of laudatory or depreciatory criticism as regards new inventions, the intention so conspicuously shown of only imparting useful information on chemico-technological subjects. These qualities have given our Society, in my opinion, a distinguished position in the technological world. That this position may for many long years be retained is my earnest wish.

In looking at the list of past Presidents of the Society, as given in our Journal, it will be seen how many eminent men have held the post, and on examining the reports of our annual meetings how valuable and interesting were the addresses delivered by them. When therefore I was informed of my nomination as President for the past year I was taken by surprise and felt something like dismay, as being very conscious of my inability, should I be elected, to follow the example of my predecessors in performing the duties attached to the office, and more especially in adding another to the able addresses which adorn our Journal. It would have been useless to refer to the fact that I am no longer in touch with the advanced chemical technology of the day ; the reply might have been : Do your best, which is indeed the utmost any man can do under any circumstances. A more valid reason for refusal might have been found in my advanced age, which was likely to prove, and I am afraid has proved, a serious drawback to my activity. It would, perhaps, have been to the advantage of the Society, and certainly so to mine, had I been appointed to the high office I now hold 10 or more years ago, when my faculties were I will not say at their prime, but certainly brighter and more fitted for hard work than they are at present. We read of the Psalmist's three score years and ten as the limit beyond which the bodily and mental powers of man cease to show any vigour. The Government of this country discharges its servants at the age of 65, at which age they are supposed to be superannuated. And yet a scientific society does not hesitate to bestow an office involving some mental and bodily exertion on a man of more than 75. I do not complain of this, being too sensible of the great honour that has been conferred on me. I mention the circumstance merely as bespeaking your indulgence for any mistakes and shortcomings I may have been guilty of during my term of office.

The delivery of an address by the President on this occasion being a *sine quâ non*, the question arose with me as to what the subject should be. I might imitate the example of some of my most distinguished predecessors, such as Sir H. Roscoe, Sir F. Abel, Mr. Weldon, Dr. Mond, Sir Lowthian Bell, and last but not least, Mr. Tyrer, my distinguished predecessor, who have all given us in the form of addresses valuable and exhaustive treatises on the subjects with which they were specially familiar. The question may be asked, Why not go and do likewise ? I would willingly have done so, but, alas, the speciality which I may or might have called my own—the chemistry of organic colouring matters—has sailed away far out of my reach and ken, and would not, I fear, have been overtaken except after arduous labour, and then what I might have to say on the subject would probably have done little to extend it.

Instead, therefore, of giving you an elaborate disquisition on any particular subject, I shall rather endeavour to divert you by referring in the first place to some forgotten or half forgotten facts connected with the history of chemical technology in the Manchester district during the first half of this century, more especially in connection with the personalities of the men to whom we owe so much in this branch of industry. I shall, in the next place, refer shortly to a few organic colouring matters about which I think I know as much as most men ; on which will follow a few remarks on the theory of dyeing as applied to cotton, and some general observations on the dyestuffs of modern times as compared with those formerly employed ; and lastly, some views and opinions on some subjects on which I have thought, but which I do not profess to have studied in a manner such as their importance would demand.

Facts and Reminiscences relating to the History of Chemical Industry in Manchester during the First Half of the Century.

I think it may be of interest to the members if I were to recall some facts, partly known to myself, partly due to information derived from others, regarding the history and achievements of some persons living and flourishing during the first half of the century in our town and neighbourhood to whom chemical industry is more or less indebted. I begin with John Dalton. I suppose there is no one here present but myself, and very few persons still living, who were personally acquainted with this great man. On my mind his image is so indelibly impressed that, were I a portrait painter, I think I could trace out his lineaments to the minutest feature ; though this might not be very difficult, since the expression of his face, though fine, was remarkably passionless.

Dalton lived with the master of the school that I attended, and I had frequent opportunities of seeing and wondering at his ways. How well I remember his peering every morning out of his bedroom window, which opened on to the school yard, and looking at his thermometer outside, the bulb of which was shaded by a card—a circumstance which puzzled me very much at the time. Very soon after he would appear at the front door of the house arrayed in his snuff-coloured Quaker dress, with knee breeches and broad-brimmed hat, stooping, as most thoughtful men do, with a large lantern in his hand, with which he proceeded across the street to his laboratory in the building belonging to the Literary and Philosophical Society. His object in carrying the lantern was to light the fire in his laboratory— matches, except of the rudest description, being then unknown. His laboratory was a room the dimensions of which did not exceed 20 ft. by 15 ; here all his great discoveries were made, and here I suspect all his works were written, there being no room in the house where he lived in which he could well be alone to compose his writings. I was often puzzled in after years to know how, in the contracted room he used for his laboratory, and which still exists, though used for other purposes, he could make those world-wide discoveries on which his fame depends. The answer to the puzzle was, however, simple enough : it was the mind that made the discoveries, not the place. I mention Dalton first and foremost, not so much on account of his great chemical discoveries, but because he was, in his day, the universal referee in all matters connected with technical chemistry, such as the purity of waters used for dyeing purposes, the genuineness of drugs, &c. He was on intimate terms of friendship with Thomas Hoyle, the eminent calico-printer of this town, also a Quaker—a friendship of which religious sympathy and association probably formed an element ; and it is just possible that Dalton may have had some share in the development of the process whereby the production of the so-called Hoyle's purples on calico was brought to the high state of perfection such as we know it, but there is no evidence I know of in support of this view.

A very eminent family connected with chemical industry flourished in Manchester at the same time as Dalton. I allude to the Henry family. Of this family three gene-

rations successively distinguished themselves. The first member of any note was Thomas Henry, a gentleman, it is understood, of Irish extraction. He was the first to prepare the so-called "Henry's magnesia"—a compound of the virtues of which the discoverer, or rather the preparer, entertained somewhat exaggerated conceptions, going a little beyond what its merit as a useful medicine justified. The distinctive merit of Henry's magnesia, I have always understood, consisted in this, that when stirred up in water it formed a soft, milk-like emulsion in which no grittiness was perceptible, and which gave no deposit, even on long standing; and this property, I have been told, was due to precipitation with potassium carbonate in the place of sodium carbonate, and to using the solution of magnesia in a very concentrated state—a proof, if correct, of how often success in manufacturing manipulation depends on minute details. A favourite practical joke in Mr. Henry's works was to ask a lady visitor to lift up a huge block of magnesia, weighing apparently a hundredweight; she, exerting herself to her utmost, would be surprised to find that it could be lifted like a bundle of feathers. In addition to magnesia, Mr. Henry manufactured aromatic vinegar, an article without which no lady's toilet table was thought to be complete in those days. By the manufacture of these two articles it is supposed that in the course of years a large fortune was amassed. That Thomas Henry was a man of some culture may be seen by his paper "On the advantages of Literature and Philosophy in general, and especially on the consistency of Literary and Philosophical with Commercial Pursuits," published in the first volume of the memoirs of the Manchester Literary and Philosophical Society. I cannot help thinking that the class of men to which Mr. Henry belonged —men who combined an assiduous attention to professional duties with a considerable amount of general culture—is gradually becoming less numerous. If this be indeed the case, we can only look back and regret what is past and gone. No doubt the present has, and the future will have, much to compensate us for what we have lost—still, what is lost is gone, and can never return. Thomas Henry was succeeded by his son William Henry, who had enjoyed the advantages of a university education, but continued nevertheless to carry on the business of a manufacturing chemist. William Henry took a great interest in theoretical chemistry, and was the author of a work entitled "The Elements of Experimental Chemistry," which ran through several editions and had much vogue in its day. In one of the early editions of this work the author says, referring to the plan of a chemical laboratory: "It is scarcely possible to offer a plan of a laboratory which will be suitable to every person and to all situations, or to suggest anything more than a few rules that should be generally observed. Different apartments are required for the various classes of chemical operations. The principal one may be on the ground-floor, 25 ft. long, 14 or 16 wide, and open to the roof, in which there should be contrivances for the occasional escape of suffocating vapours. This will be destined chiefly for containing furnaces, both fixed and portable. It should be amply furnished with shelves and drawers, and with a large table in the centre. Another apartment may be appropriated to the minuter operations of chemistry, such as those of precipitation on a small scale, the processes that require merely the heat of a lamp, and experiments on the gases. In a third, of smaller size, may be deposited accurate balances and other instruments of considerable nicety, which would be injured by the acid fumes that are constantly spread through a laboratory." I quote this passage in order to show the wide difference there was at that time between the well-appointed laboratory of the private amateur and the humble apartment in which Dalton's great discoveries were made, and in which the atomic theory first saw the light. In one of the earlier editions of his "Elements of Chemistry" Dr. Henry gives a list, with plates, of all the various apparatus necessary for carrying on chemical operations. The list is very extensive, but singular to say does not include one of the most ordinary pieces of apparatus—one with which every beginner is familiar—I mean a stand of test tubes. To what this is to be attributed, whether to oversight, or to the fact that in those days the attention of chemists was directed more to experiments with gases and the results of igneous fusion than to reactions in the wet way, it is hard to say. To Dr. William Henry is sometimes given the credit of the invention of aerated waters. There is some doubt about this, but if correct, he must be numbered among the benefactors of mankind, as having added largely to its innocent gustatory pleasures. A third generation of this remarkable and highly-gifted family was represented by Dr. William Charles, son of Dr. William Henry. Dr. William Charles Henry was a tall, dignified person of polished—one might say aristocratic—manners and bearing. He had passed through the regular medical course of the Edinburgh University, and practised as a physician in Manchester. He gave up his practice, however, and devoted himself to commercial pursuits in connection with the works belonging to his family. I have good reason to remember Dr. Charles Henry, for, he hearing that I was learning chemistry entirely from books, very kindly offered to give me some practical instruction in science, and I was accordingly introduced into the laboratory attached to his works—a somewhat dark and dingy apartment, but a paradise to me who had never seen its like before—and taught the rudiments of analysis. Dr. Charles Henry was a favourite pupil of Dr. Dalton, who bequeathed to him the whole of his scientific apparatus. This was afterwards given to the Literary and Philosophical Society, in whose rooms it is still to be seen. He also wrote a life of Dalton, a classic production forming one of the most important volumes of the Cavendish Society's series.

An eminent contemporary of the Henrys and belonging to the same class, *i.e.*, of men distinguished not only for skill as manufacturers, but also for general culture and taste, was James Thomson of Primrose, near Clitheroe, head of the firm of calico-printers called after him. The history of calico-printing in this country has still to be written, but when it is written the name of James Thomson will occupy a conspicuous position in it. He was a man of wide culture and had an extensive library; a man of science also, with a large, well-furnished laboratory. In Thomson's time very little had been done to elucidate the theory of dyeing. Dyeing and calico-printing were in this country considered mere arts to be classed with wall painting and decorating. In France, however, much had been done through the labours of Berthollet, but more especially of Chevreul, to place the art of dyeing on a scientific basis, and establish the principles on which it depends. Thomson was, no doubt, acquainted with their works, and, carrying out their ideas, was able to effect great improvements in the art which he cultivated. He will always be remembered as the inventor of the process for discharging Turkey-red by printing tartaric acid on the cloth and then passing it through a solution of bleaching powder—a process which, I believe, is still in operation. This process was a great improvement upon the pressplate printing as carried out in Scotland and brought to perfection by Monteith, of Glasgow, in 1818. Thomson's large-mindedness was shown by his calling to his aid men of scientific distinction, such as the present Lord (then Dr.) Playfair, an eminent pupil of Liebig. Mr. Steiner, who afterwards distinguished himself by his improvements in Turkey-red dyeing, was also with him for some time. We, therefore, can hardly feel surprised when we are told that the Primrose Printworks were everywhere celebrated for their productions, though I am not aware that they were noted for any speciality with the exception of the process just mentioned. Mr. Thomson was the first to give a drawing of the cotton fibre as it appears under the microscope in his memoir on the "Mummy Cloth of Egypt," in which he arrived at the conclusion that the cloth consists entirely of linen, and that cotton was probably unknown to the Egyptians. Most ancient fabrics consisted, I imagine, of wool only; this was the case with some coloured fabrics belonging to quite a late period of Egyptian history, which I examined some time ago. Thomson's social position enabled him to be of assistance in passing measures of public utility. He was of great service in helping to pass the Copyright of Designs Bill, and he was a great supporter of the Manchester School of Design.

The name I have now to mention is that of a man of a type differing widely from that of James Thomson and the

Henrys—it is that of John Mercer, probably the greatest genius who ever applied himself to the improvement of calico-printing. Born in the humblest circumstances and without even the rudiments of learning, he taught himself all he required, from reading to music, mathematics, and chemistry, and by his industry and ingenuity raised himself gradually from the position of a handloom weaver to that of a partner in an important calico printing firm. Mercer's inventions in connection with calico-printing are characterised by great originality and by the ease with which they could, it seems, be carried out at once in practice. Coming as they did from one who was entirely selftaught and had read but few books on chemistry they are indeed marvellous. To name them all would take more time and space than I have at my disposal. They are described at length in Parnell's "Life and Labours of John Mercer," an excellent work which is unfortunately out of print. Among the inventions due to Mercer the following may be held to be the most important: The application of the orange-coloured sulphide of antimony and of the brown oxide of manganese in calico-printing, his improvements in Turkey-red dyeing, the successful attempts to replace the cowdung previously used in calico-printing by substitutes such as sodium phosphate or arseniate, and last though not least, the invention of a process, now called "mercerising," whereby the properties of cotton fibre, both chemical and physical, are modified in a remarkable manner. This process of mercerising, which consisted in passing calico or any other cotton fabric through a strong solution of caustic soda, showed, like everything that Mercer did, much ingenuity and originality, but it did not come into such general use in Mercer's time as he had expected, partly, I understand, because the dyes of his day were not of a kind to withstand the energetic action of the alkali. Latterly, however, it has again come into use as a means of producing a peculiar corrugated or crinkled effect on calico, and its employment for this purpose has assumed, I am told, quite extraordinary dimensions. Its use as a means of imparting to cotton a silk-like lustre has been described in a paper read before the Yorkshire section during last session. Mercer, with his manifold mental activity, did not confine himself to subjects connected with the arts of dyeing and printing, he also speculated and always with ingenuity on subjects belonging to pure chemistry. He paid attention to so-called catalysis, to the composition of bleaching powder, and to the relations between the atomic weights of the elements. On the last-named subject he was probably the first to put forth more or less speculative opinions, such as were subsequently developed by Newlands, Mendeleef, and others. Whatever he said, whatever he wrote, was characterised by ingenuity and originality, truly one of nature's own original geniuses. I knew John Mercer. I always associate him in my memory with Sam Bamford, the Radical, whom I also knew, both Lancashire men of the best type, of robust bodily frame, thoroughly honest and outspoken, without guile or deceit, gentle and devoid of suspicion. May the type never die out!

The next name I have to mention is that of Mr. John Dale. His name brings us down to comparatively recent times, when new substances and new methods were being introduced into manufacturing chemistry. In the transition period through which chemical technology then passed Mr. Dale bore a conspicuous part. Mr. Dale was a native of Birmingham, but he came to Manchester early and spent here the greater part of his life. Arrived in Manchester he became a pupil of John Dalton, but in his case, as in that of most of Dalton's pupils, no characteristic anecdote or trait of the master seems to have come to us through him. After passing through various employments, that of taking charge of some calico-printworks being one, he settled down at last as partner of the noted firm of Roberts, Dale, and Co., of this city. It was in connection with this firm that the greater part of his work was done. The manufacture of oxalic acid by fusion of sawdust with caustic alkali in accordance with Gay-Lussac's invention was one of the first processes carried out practically by Dale. Then followed improvements in the manufacture of mauve, the preparation of Manchester Brown, and Manchester Yellow, after which came the manufacture of aurin, the theory of which was worked out by the late lamented Professor Schorlemmer in conjunction with Mr. Dale's son. Mr. Dale is the author of what is probably the best method invented so far of estimating the quantity of colouring matter contained in commercial indigo. I knew John Dale well and was always impressed when conversing with him, with the originality of his views and the simple unaffected manner of his expressing them.

In this place I might mention the name of Mr. Peter Hart—a valued member of our Society quite recently deceased. He was for many years connected with the firm of Tennant, Clow, and Co. They manufactured salts of tin, sulphates of copper and zinc, nitrates of lead and iron, sodium arsenate, and sulphate of indigo.* Dr. James Young, afterwards celebrated for his inventions in connection with the mineral oil industry, was for nine years with Messrs. Tennant, Clow, and Co., and introduced the manufacture of red prussiate of potash. The chief part of Mr. Young's career was, however, spent in Scotland.

I now propose to transgress somewhat the limits I had set myself in bringing before you this subject—the early history of technical chemistry in this district by referring to men who are still amongst us and to processes still in operation. Many years ago Mr. E. Sonstadt, who was associated with the late Walter Weldon in scientific and literary undertakings, was struck by a remark of Bunsen in one of his memoirs, that if the metal magnesium could be produced on anything like a considerable scale it might be found possible to obtain photographs in dark and obscure places. Mr. Sonstadt, struck with this suggestion, resolved to try to produce this metal. In due time his experiments were successful, he having devised the well-known process of obtaining magnesium by the decomposition of the double chloride of magnesium and potassium by means of sodium. It was found at first that much of the magnesium so obtained was in tiny globules interspersed among the mass. In order to obtain it in a more concrete form he devised an ingenious process of distilling the metal, which was found to work satisfactorily. Almost the first specimen of magnesium produced was shown to Michael Faraday, who was highly pleased with it, exclaiming, "This is a triumph, this is a triumph!" It was suggested that he should accept the specimen for his own collection, but he with characteristic modesty declined, saying, "If you will allow me I would like to present it to the Royal Institution in your name." In due course works were erected in Salford and the manufacture of magnesium commenced on a large scale, conducted under the care of Mr. Samuel Mellor, who has been associated with them ever since. The manipulation of magnesium was attended by many difficulties, as it would neither roll into sheets, draw into wire, nor be beaten by the hammer on the anvil into shape. After many trials and experiments, however, Mr. W. Mather of the Salford Iron Works, devised a highly ingenious machine for forcing it into wire of any thickness or fineness. This machine has never been superseded and remains to this day a specimen of the inventor's great mechanical ability. The suggestion that magnesium could be utilised for taking photographs in dark and foggy weather was very soon carried out, Mr. A. Brothers, of this town, being the first to make use of the metal for photographic purposes. For signalling purposes, too, it was found that magnesium could be utilised. In the Ashantee War, Captains Bolton and Colomb devised a clever method of signalling, words being spelt out by lights of longer or shorter duration, in which magnesium was used in the form of dust or powder mixed with kauri gum or lycopodium. For fireworks and signal rockets also, magnesium came to be extensively used.

After Messrs. Bell Bros., of Newcastle, had abandoned the manufacture of aluminium, all the sodium consumed in England and almost all over the world was produced in this district, and so arose another local industry. Potassium was produced at the same time. A curious incident, which occurred in the early period of the manufacture of these metals, may here be recorded. A rather large quantity of

* Garancine—an article no longer in use, made by the action of sulphuric acid on madder—was also manufactured by this firm.

potassium and sodium having to be sent to a German professor, the two metals were put into the same vessel to save room. When the parcel arrived at its destination, singular to say the metals were no longer separate and solid, but had combined to a liquid alloy like mercury. This formation of a liquid alloy of potassium and sodium explains the fact that potassium obtained from so-called pure salts, but still not quite free from sodium, is more or less liquid, causing great annoyance to the manufacturer. It is not likely that this curious compound will ever be examined, as it has extremely explosive properties.

The history of the discovery of vanadium in this district, is one of peculiar interest. Some 25 years ago in the neighbourhood of Mottram and Alderley, Cheshire, some mineral deposits were found—I presume in the red sandstone—containing copper, cobalt, and other metals; and works were erected there for obtaining these metals, under the care of Dr. Gerland, an esteemed member of our Society. While working with this material, Dr. Gerland, obtained a blue liquid, from which no copper was precipitated by zinc. On examination, the blue colour was found to be due to some compound of vanadium. In consequence of this interesting discovery, a quantity of raw material was collected at the original site and at another site near Shrewsbury, and with the assistance of Mr. Mellor, of the Magnesium Metal Co., this was worked up and brought into a state of comparative purity. With this material to work on, Sir Henry Roscoe was enabled to carry out his classic researches on vanadium and its compounds. It also proved of great use in the hands of Mr. John Lightfoot who was then engaged in an investigation of aniline black, and who found vanadic acid to be far the best agent to use for the production of that dye. The material thus obtained was sufficient to supply all the vanadium salts used in commerce for a number of years.

I come now to the second division of my address, referring to—

Organic Colouring Matters.

Of these it is principally the natural colouring matters, *i.e.*, those occurring in nature, that I shall allude to; and of these, too, I shall refer to only a few—such, namely, as I have specially studied and am familiar with. I will begin with chlorophyll. This substance, to which the green colour of leaves and other parts of plants is due, is certainly, in some way not yet understood, involved in one of the most important functions of vegetable life—that of the conversion of the CO_2, NH_3, and H_2O of the atmosphere and soil into organic matter. Chlorophyll is one of the most fugitive of substances. Under the action of air and light, it is rapidly destroyed—this seems, indeed, to be always its fate, not only outside, but also within the vegetable organism. When, however, it is submitted to the influence of strong chemical agents, it yields, singular to say, products of remarkable stability, a fact of which our present vegetable chemistry fails to give a satisfactory explanation. Whether mineral acids or whether alkaline lyes are employed, the result, so far as stability is concerned, is the same. When submitted to the action of strong acids, chlorophyll undergoes complete decomposition, yielding definite products, to one of which, and the most important, the name of phyllocyanin has been applied. This substance, when in alcoholic solution, resists the action of air and direct sunlight for weeks, whereas an alcoholic solution of chlorophyll would, under the same circumstances, very soon lose its colour and be decomposed. Phyllocyanin yields compounds with metallic oxides of a brilliant green colour, which are at the same time remarkably stable. Of these compounds the most remarkable, so far as regards brilliancy of colour, is that with copper. This compound might, I think, on account of its great stability, be used with advantage, when properly prepared, in painting, more especially water-colour painting. When employed in the representation of foliage, for which it would seem well adapted, it would require the admixture of a yellow pigment, since the natural colour of leaves is due to the mixture of a green with a yellow colouring matter, the varying proportion between the two giving rise to the variety of tints we see in spring and summer foliage. The brilliant autumnal coloration of some leaves is due to quite a different class of substances. Chlorophyll, so called, is used, I believe, as a means of imparting a green colour to a particular class of soaps, but why a green colour should be required in this case, and why any other green pigment should not answer as well, I cannot understand. It occurs in commerce as a dark green syrupy mass under the designation "chlorophyll purissimum." It is, however, neither purum nor purissimum, nor even chlorophyll at all, being a mixture of fat with some derivative of chlorophyll, such as cupric phyllocyanate or the product of the action of alkalis on chlorophyll, which, like phyllocyanin, is a very stable body, and has a fine green colour. How these substances are applied in soap-making, I have never been able to ascertain. It may be asked whether chlorophyll, communicating as it does such a brilliant green colour to vegetable tissues, might not be employed for dyeing fabrics the same colour. I have myself made some experiments in this direction, but without any conspicuous success. The insolubility of chlorophyll in water and its great instability present obstacles to its employment in this way, which are almost insuperable. In my experiments I used the cupric phyllocyanate, which is prepared by decomposing chlorophyll with hydrochloric acid, collecting the resulting product, dissolving it in boiling glacial acetic acid, and adding cupric oxide, when a liquid of a splendid bluish-green colour is obtained, which, on cooling, deposits crystals of a fine cuprous lustre by reflected light. This compound, of which acetic acid is in reality an essential constituent, was dissolved in acetic acid, and bits of wool, silk, and cotton, sometimes prepared previously with albumin, were dipped in the solution and left to lie some time, then taken out, exposed to the air, and washed. The result was the production on the tissues of various shades of green according to the fabric used, but the tone was anything but brilliant, and in these days when brilliancy rather than solidity is sought for, such a colour would not be of much value. Some doubt might arise as to whether this imparting of green colour to fabrics is in reality a dyeing process in the true sense, or whether it is not rather a depositing of a pigment on the fibre in consequence of the evaporation of its solvent. It would resemble the green colour formerly much used in calico-printing, which was made by dissolving chromium arseniate, which is a compound of a pale sea-green colour, in hydrochloric acid, then printing on the calico and exposing to air; the solvent escaping, the compound was left fixed on the fibre. However, this is a matter of academic interest only.

This brings me to the subject of pickles, comparatively a not very dignified subject, but still not unimportant as everything is that concerns articles of food in daily use. Much discussion has of late arisen as regards the real or supposed injurious consequences of imparting to pickles and other articles of food the much admired vivid green colour so often seen in them. Since the points to be considered are connected more or less with the chemistry of chlorophyll it can hardly be expected that the public in general, perhaps not even the majority of medical men, whether analysts or not, can arrive at correct conclusions on the matter. The opponents of the so-called adulteration says this: the bright green colour so frequently seen in pickles is communicated by the addition of some salt or compound of copper, now copper compounds are poisonous, therefore the colouring or dyeing of vegetables by means of copper in any shape must be injurious. To this it may be replied that copper salts taken internally are no doubt injurious, even poisonous, though probably far less so than compounds of some other metals. The practical question to be determined however is whether the quantity of copper actually present in the coloured articles is really as injurious as has been represented. The green colour seen, say in a pickled gherkin, after the addition of some compound of copper is that of a derivative of chlorophyll, it is that of cupric phyllocyanate, the compound already referred to, which has a considerable tinctorial power. If we consider how little chlorophyll most vegetables contain, probably not 1 per cent. that chlorophyll yields by decomposition a not very large amount of phyllocyanin and that the cupric phyllocyanate contains according to my determination only 9 per cent. of copper, and when we consider moreover that the copper is contained in

the cupric phyllocyanate in a form in which it cannot be detected by ordinary reagents, then I think we may arrive at the conclusion that the small quantity of copper contained in a green pickle or vegetable cannot be very injurious; it probably does not exceed the amount we are supposed to swallow daily with our food. The mischief, however, consists in this, that the pickling vinegar containing usually an excess of copper salt is allowed to stand in contact with the pickles and may be consumed to some extent with the latter. If the vinegar is poured off and the vegetable washed with water, which is also poured off before adding fresh vinegar, then I feel convinced any quantity of the product may be eaten with impunity. An eminent physiological chemist, Prof. Tschirch of Berne, who has undertaken an exhaustive examination of the subject, has arrived at a similar conclusion. From his experiments, which agree with those of others, it follows: 1. That copper, or rather the salts of copper, are by no means innocuous as has sometimes been asserted, but that a tolerably large dose is required in order to produce injurious effects. 2. That for a human being weighing 60 kilos., the quantity of copper that may with safety be taken per day amounts to 0·1 grm. 3. That as regards the artificial green coloration of vegetables with compounds of copper 50 mgrms. of copper may be allowed to 1 kilo. of vegetable without fear of any injurious consequences.

These are precise and definite conclusions derived from accurate experiments, and I cannot conceive what objection there can be to establishing in this case the exact percentage beyond which the adulteration, if it may be so called, shall go, and so avoiding the unseemly disputes which sometimes take place in courts of justice between those who maintain that cupric compounds being poisonous should on no account be permitted in the preparation of food, and those who assert that the use of such compounds may, to a certain extent, be allowed. All that need be ascertained in any doubtful case would be whether the limit in regard to the amount of copper is transgressed or not. For the information of those to whom this view of the matter is not acceptable, and who still feel anxious about copper in food, I may say that all pickles—that is, pickled vegetables—that show a grass-green colour, however slight, certainly contain copper. I lately bought a bottle of gherkins of a pale grass-green, prepared according to the label by a first-class firm in London, and "warranted free from copper," but notwithstanding the assurance given they did contain copper. Pickles prepared with vinegar only are always and without exception olive-green. For my part I quite fail to understand the pleasure and satisfaction derived from the eating of a bright green article of food. An olive-coloured gerkin, if well prepared, is quite as toothsome as a bright green one; I am inclined to think more so. Should a substitute be required for the copper salts hitherto employed in cooking, which shall be both bright and innocuous, I think it might easily be found among the numerous green colouring matters of artificial origin now occurring in commerce. So much for the industrial applications of chlorophyll.

Before leaving the subject of chlorophyll, I should like to say a few words relating to an important discovery connected with the chemistry of chlorophyll. This discovery was made in my laboratory, but the merit of it is chiefly due to my esteemed friend and former collaborator, Dr. Marchlewski. The discovery is not of importance from an industrial point of view, though one can never tell what the future may bring forth, the mere scientific curiosity of one age sometimes becoming the basis of an important industry in the next. From another point of view the discovery is both interesting and important, and I will therefore shortly describe it. By the action of strong acids on chlorophyll a certain product is obtained, and the action of alkali on this product, in sealed tubes, leads to the formation of phylloporphyrin, a substance crystallising in lustrous red needles which has remarkable properties. Treated in a similar manner, hæmoglobin, the colouring matter of blood, yields an analogous substance—hæmatoporphyrin. Now these two substances, phylloporphyrin and hæmatoporphyrin, resemble one another in several respects in a most remarkable manner. Both are red in colour and give red solutions; both act the part of weak bases towards strong acids; both when heated give off fumes of pyrrol; the ethereal solutions of both show absorption spectra of seven bands, the intensity and relative position of which are in both cases absolutely the same, the only difference being that in the case of hæmatoporphyrin the bands are slightly nearer the red end of the spectrum (see page 595). As to composition too, the two substances approach one another, that of phylloporphyrin being expressed by the formula $C_{16}H_{18}N_2O$, that of hæmatoporphyrin, according to Nencki, by $C_{16}H_{18}N_2O_3$; they differ therefore in the amount of oxygen they contain, phylloporphyrin, as might be expected, having the least.

I might speculate at some length on the resemblance thus shown to exist between a derivative of chlorophyll, that most important substance in the vegetable economy, and an analogous derivative of hæmaglobin, which is a substance equally important in the animal system, but I refrain. Prof. Nencki, an acknowledged authority on the subject of blood colouring matters, referring to the discovery I have just shortly described, says it may throw some light on the process of evolution which the chemical constitution of organised structures has undergone in former ages, the development of the material of which these structures consisted having gone on *pari passu* with that of their form and structure, but I do not venture to follow him on this domain.

There is another subject connected with the chemistry of plants with which I am tolerably familiar, and on which I wish to say a few words, seeing that it is not, generally speaking, well understood; it is the nature of the process whereby the well-known blue colouring matter, indigo, is formed in and obtained from the tissues of certain plants. Many years ago I made a series of experiments which led to the conclusion that the organs, particularly the leaves, of indigoferous plants contain a peculiar substance, a glucoside, which, when exposed to the action of certain reagents, particularly acids, splits up, yielding indigo and a kind of sugar, with other products of no special interest. This substance I called *Indican*, and I succeeded, approximately at least, in determining its composition, so far as to enable me to explain the way in which it is decomposed under the influence of acids. The matter is, however, not quite so simple as at first sight it might appear. Indican, like chlorophyll, seems to retain, if one may say so, some of the susceptibility belonging to its vital organic origin. It is decomposed, so to say, by a mere touch. It is not a matter of indifference, however, by what agent it is influenced; acids produce one effect, alkalis a totally different one, and this in the presence even of small quantities of these reagents. If an aqueous solution of indican is boiled, then mixed with acid, and then boiled again, indigo-red is obtained in place of indigo-blue. If the solution is heated with the addition of alkali, then, on adding acid and boiling, brown resinous products are formed bearing no resemblance to and having apparently no connection with indigo. In every case, however, the resulting products, unlike the parent substance from which they are derived, possess a remarkably stable character, in this respect resembling the products derived from chlorophyll. All this has been clearly set forth in various papers published in the Memoirs of the Literary and Philosophical Society of Manchester. It is therefore somewhat surprising to find men of science, who in treating this subject, go on babbling away on the old lines as if nothing had been done herein during the last 50 years. A German chemist complains of not being able to find a certain paper on account of its being buried in the huge catacomb of the Philosophical Transactions. The Memoirs of the Manchester Literary and Philosophical Society might perhaps be called a cemetery, certainly not a catacomb, and comparatively very little labour therefore would suffice to enable anyone to disinter what lies buried there.

A German chemist named Molisch professes to have discovered that the process of formation of indigo in the aqueous extract of indigoferous plants is one of fermentation, and that it is the presence of a certain bacterium that is the immediate cause of decomposition. In former days it was electricity that was called in to explain anything we did not understand, now-a-days it is fermentation and a bacterium or bacteria. In this case the

bacterial hypothesis is, I believe, without foundation. It is possible, of course, that in the experiments of Molisch the bacteria did indeed cause the formation of indigo, but only, I believe, in consequence of the secretion of some acid, or other substance by the bacteria. The weakest acid will do this, but I have shown that even in the absence of any acid, indican is decomposed under a variety of circumstances in which spontaneous decomposition can alone account for the change. If, for instance, the leaf of an indigoferous plant be exposed under water to a temperature below freezing, the parts of the leaf that become frozen turn blue, the indican being decomposed, while the parts not frozen remain green and unchanged. A leaf immersed in cold spirits of wine, turns partially blue in like manner. Here there can be no question of bacteria; for what bacterium can live and act in ice or spirits of wine? All the facts described seem to me to be easily explained without calling in the aid of bacteria or any extraneous agency. Indican, the glucoside of indigo, is a body of extremely unstable character, so much so that if only left to itself at ordinary temperatures it undergoes spontaneous decomposition. In the vegetable cell it is only preserved from decomposition by the vitality of the cell, destroy the vitality by cold, mechanical injury or chemical agency and the indican is decomposed.

Messrs. Darwin and Meldola have recently (*Nature*, Nov. 12, 1896) given an interesting account of the manufacture of indigo from woad, which is still carried on in a remote corner of Lincolnshire for the use of the woollen dyers of Yorkshire. These observers say that the process they describe consists in a "zymolitic decomposition of glucosides." This is, however, mere hypothesis. By a series of well-conducted experiments carried on in the indigo manufactories of India, Mr. Bridges-Lee* has proved that there is no particular indigo-producing process of fermentation whatever. He states as the result of his experiments that "there is no special distinctive kind of fermentation for the indigo plant, so far as I have been able to discover by careful observation and very numerous oft-repeated experiments. The indigo plant, like nearly every other kind of green plant, will rot if left in water long enough, and while rotting it will give rise to all sorts of foul offensive products of decomposition, and like other rotting plants will give off foul smelling gases, but at no stage is there any specially distinctive kind of fermentation apart from progress on the road to decomposition and decay, *i.e.*, there is no fermentation of a kind corresponding to the alcoholic or acetic or butyric fermentation." I fully endorse all that Mr. Bridges-Lee says on this subject, as it is quite in harmony with what I have always maintained.

Indigo-blue, as all chemists know, has been produced artificially by Baeyer and others, and hopes were at one time entertained that the natural product would be replaced by some artificial one, but these hopes were not, and under prevailing conditions could not, be realised. Indigo, considering the large percentage of colouring matter it contains, is one of the cheapest of dyestuffs, and in order to replace it by an artificial product, a very cheap material to begin with, and a simple inexpensive process of manufacture would be required. Improved processes applied to the treatment of the indigo plant in India might, moreover, lead to a much greater yield of colouring matter than heretofore. Improvements in this direction have already been effected in consequence of the application of machinery to the manufacture and further improvements will doubtless take place. There is one improvement, or rather modification, of the process of manufacture for a special purpose which I would suggest. I have shown that indigo-red, or indirubin—a red crystallised colouring matter which always accompanies indigo blue in commercial indigo, and is, in fact, isomeric with it—may be produced in much larger quantities than usual by modifying the process whereby the indican of the plant is decomposed. Would it not be possible, it may be asked, by suitable contrivances to produce in India a real red indigo—a product in which the indigo-blue shall be largely, if not entirely, replaced by indigo-red—a product which I am told would be of value? It appears, according to information kindly supplied to me by Mr. C. Rawson, of Bradford, that this end has, to a certain extent, been attained in India by adopting processes in which a base, such as lime or ammonia, is made to intervene at a certain stage of the manufacture. Mr. Coventry's patent process depends on the use of lime, and yields a product containing about 12 per cent. of indirubin, ordinary indigo having little more than 1 per cent., and of which a firm of dyers in Yorkshire say: "For vat dyeing it is an ideal indigo, the shades obtained in dyeing being very fine and bloomy."

That it has been found impossible successfully to replace natural indigo by an artificial product may from a moral and esthetic point of view be regarded as not altogether a misfortune. To replace a manufacture depending on an interesting organic process carried on under healthy conditions mostly in the open air, a manufacture which brings wealth into poor districts, and introduces system and order and civilisation among uncultured peoples, by one carried on perchance in some dingy sepulchral cave in a chemical works by some fixed and unalterable process, might from a higher point of view, be a doubtful advantage.

I may here interpolate a few remarks on a similar subject, the formation and employment of artificial alizarin. From a sentimental point of view it may perhaps be regretted that the process of dyeing with artificial alizarin has so entirely taken the place of dyeing with the natural product madder-root. The latter process is in many respects most interesting. Beginning with the glucoside of alizarin naturally contained in the root and extracted with water, we have first the decomposition of this glucoside under the influence of an enzyme peculiar to the plant and extracted at the same time, with formation of alizarin and other colouring matters; then the fixation of the latter on the mordants; lastly the removal of the colouring matters other than alizarin, together with other impurities, by various reagents, such as soap, acids, chloride of tin, &c., leaving a ternary compound of alizarin, fatty acid, and mordant as the final product. I can conceive no process more elegant and complete in this department of technical chemistry. Artificial alizarin, on the other hand, is supplied ready made to the dyer and printer, and though its formation is owing to a most interesting process, the elaboration of which is due to the genius of several distinguished chemists, there is nothing especially interesting in its application.

I may mention, what is sufficiently evident but is sometimes forgotten, that the various processes by which alizarin, indigo, and other organic substances are formed artificially throw no light whatever on what goes on in the plants yielding them. It is absurd to suppose that the plant should employ such substances as anthracene and phenylglycocoll as bases on which to build up its alizarin and its indigo. The natural laboratory of the plant must differ very widely from the chemical one, and we have as yet not the least conception of what goes on in it. At one end we have CO_2H_2O and NH_3, at the other end perhaps indigo; the intermediate stages are involved in mystery.

I have now come to a point at which it may probably be expected that I should say something about modern, *i.e.*, more especially artificial colouring matters and their application in the arts of dyeing and printing. This is a very extensive subject, and I must, for reasons already given, confine myself to a few general remarks, and these will refer chiefly to a comparison of modern methods with those employed when I was conversant with the arts I have named. I had recently an opportunity of visiting the works of an eminent calico-printing firm near Manchester, and, though my visit was too short to enable me to go much into details, I arrived at the conclusion that this branch of manufacture had, during the last 40 years, been entirely revolutionised. I say nothing as regards mechanical contrivances, but on the chemical side everything is new. There are new mordants used, such as salts of chromium, tannin, and tartar emetic—all unknown in my day. To these may be added salts of titanium, the use of which has recently been recommended; then a host of artificial colouring matters, basic colouring matters, direct colouring matters, and so on, which are of recent introduction and require in their application new and refined

* "On Indigo Manufacture," by J. Bridges-Lee, M.A. Calcutta: Thacker, Spink, and Co. 1892.

processes. The number and variety of artificial colouring matters introduced have been so great that at one time it was thought the natural compounds would be entirely superseded and replaced by artificial ones. This, however, has not been entirely the case. I was pleased on my visit to the printworks alluded to, to meet with an old friend—the indigo vat, essentially the same as I knew it, though somewhat altered as to details; also the Schlieper and Baum process of printing indigo, which was invented in my day, though not much used then. Alizarin, too, still maintains its ground, in the form, however, of the artificial product, associated occasionally with another artificial product, flavo-purpurin, a substance which was first prepared in my laboratory, and is, therefore, specially interesting to me. Alizarin colours, more especially Turkey red, will have, however, to undergo severe competition from a new and brilliant red dye, paranitraniline red, of which much has been said and written of late. In spite of opposition, however, alizarin dyes will probably, like indigo, continue to hold their ground for some time to come. That there still remains something to be done in the chemistry of natural colouring matters is proved by the memoirs which appear not unfrequently in English and foreign scientific journals. Not long since there was published an able paper on Curcumin, the colouring matter of Turmeric, by L. G. Radcliffe, of the Manchester Technical School. Quite recently the same investigator has taken up the subject of safflower. He has obtained its colouring matter in a crystalline state, and he informs me that he has little doubt of its being a glucoside, a form under which most natural colouring matters appear in the organisms containing them.

The most useful of all the artificial colouring matters, both as regards variety of tint and other properties, are the azo colouring matters. It would appear that the great importance assumed by these colouring matters in various directions is entirely justified by their manifold properties, and the possibilities of this wonderful group seem to be still unexhausted. Among the colours belonging to this group are several blacks, such as Diazo-black, Diamine-black, Columbia-black, and others, which, together with Aniline black, form perfect substitutes for the logwood black of former days, so that the discovery of a method of producing hæmatoxylin artificially is not such a desideratum as it was once thought to be.

I will not here enter on the vexed question of the comparative stability towards light and other agents of natural and artificial colouring matters and the dyes derived from them, about which opinions vary, but I may be permitted to allude *en passant* to the theory of dyeing, as to which much has been done and written of late, chiefly in order that I may enter a renewed protest against the chemical and in favour of the physical theory of dyeing, so far at least as regards cotton. Dr. Knecht, by ingenious experiments, has proved that the dyeing of wool is to a large extent a chemical process, *i.e.*, that wool contains organic substances which act as mordants and unite with colouring matters to form lakes. Cotton fibre, on the other hand, does not contain anything of a lake-producing nature, except it be a minute quantity of an albuminous substance which I found in my experiments on the constituents of cotton. Dr. Knecht, however, who has had much experience in this kind of investigation, is still of opinion that a substance or substances may be obtained from cotton which have the property of forming lakes with colouring matters. Prof. Mayer, of Naples, has lately sent me a copy of his memoir on the coloration of the cell nuclei of plants by various reagents, wherein he advocates strongly the chemical theory of coloration or dyeing in the cases coming under his observation. I am, however, not convinced by his arguments. A cell nucleus may be, and I suppose is, a nitrogenous body, and may therefore behave very differently to the cell wall, which consists of cellulose, towards colouring matters. I am at a loss to understand how, on the chemical theory, the union of such a body as carthamin, which is nearly insoluble in water and absolutely neutral with cotton fibre, which is also neutral and insoluble, to form a brilliant carmine dye can be explained—it seems to me to be a case of surface attraction. Again, in the case of the indigo vat, where we have a very weak acid united to a strong base, can we suppose that the cotton fabric dipped into it seizes hold of the reduced indigo by chemical attraction, setting free the lime, soda, or other base with which it was combined? It seems to me that surface attraction, or whatever it may be called, is the only agent that can account for what takes place, and that the process is not chemical, but physical or mechanical. What I have said applies to cotton only, not to wool or silk. The question, however, is one mainly of academic interest. As regards practical dyeing, it can be a matter of little importance which theory is adopted.

Adulteration of Food and Impurities in Water and Air.

It is pleasant to be able to report that as regards adulteration of articles of food, a great improvement has taken place within the last few years in this district. In his report to the Cheshire County Council, Mr. Carter Bell, the able analyst for the county, says: "As I have now completed my 20th year as your analyst for the county of Chester, it will be interesting to look back upon these two decades and notice the vast improvement which has taken place in the supply of food to the public. Before the passing of the Food Adulteration Acts nearly every article of food was more or less adulterated. The difficulty then was to find purity, the difficulty now is to find impurity. In former times flour was found adulterated with chalk, bone earth, powdered flints, and plaster of Paris. Bread was adulterated with alum, sulphate of copper and sulphate of zinc, the two latter being very poisonous salts." This is a very satisfactory result so far as the various articles of food are concerned. As regards water supply the city of Manchester, with its neighbouring town of Salford, enjoy most favourable conditions, the supply being abundant, pure, and very soft. There is, therefore, no occasion for a periodic examination of its quality, as there is in the case of London water, the sources of which are always more or less suspect. The case is, however, very different as regards our effluents, the sewage and waste water of all kinds from the town or, rather both towns, and the drainage area generally. Here we seem to be surrounded by difficulties almost insurmountable. At the time when Manchester was much smaller, its population less, its trade prosperous, and sanitary conditions not much thought of, it seemed only natural that our one river should convey away the sewage and waste water of the town to the sea, what else could be done with it? But now circumstances have changed, more especially in consequence of the effluent water being impounded and stored up in the Manchester Ship Canal, where it ferments and decomposes, giving off fetid gases and objectionable odours. All attempts to remedy this state of things by purifying the water before allowing it to run into the canal seem to have been more or less in vain. All that seems certain is that a successful scheme, even if possible, is sure also to be a very costly one. Some of you will be conveyed to-morrow, I expect, down the Ship Canal, and you will be able to judge for yourselves what the present state of things is; no doubt it is highly disagreeable, at least in the upper part of the canal. Seeing what it is, there may perhaps be among you someone, who, by a sudden inspiration of genius, may see at once what is the proper remedy for this great annoyance and nuisance. The solution of that hitherto insoluble problem—the purification of our rivers and streams—may indeed be near at hand. In the meantime it may occur to some one of a sceptical turn to ask whether there is really evidence such as would satisfy a scientific inquirer, to prove that the waters of our polluted streams really have such a deleterious effect on the health of the riparian dwellers as is sometimes asserted. No doubt the mortality of this city, to take an example, is high, no doubt disease is rife, and its sanitary state somewhat unsatisfactory, but to what extent these defects are due to the state of our rivers and canals has to be shown. Objections to the present state of things are sometimes raised on sentimental and esthetic grounds. Such objections, however, are not to be hastily brushed aside as being of no importance, for there is no doubt that what depresses the mind affects also the bodily health, but the question is whether we are justified, except on the highest and most urgent grounds, in saddling the towns and manufacturers of Lancashire—in face of a declin-

ing trade—with the enormous outlay, which, according to experts, would be necessary in order to effect the purification of our rivers to the satisfaction of the authorities.

The pollution of our rivers and streams may perhaps not affect many persons, but there can be no doubt that impurities in the atmosphere, especially in that of our large towns, are very sensibly and injuriously felt by the population generally. Impure water we may escape from—we have no occasion to use it, owing to the abundant supply of a wholesome article in most of our large towns; we need not dwell by polluted streams unless we choose; but impure air we cannot avoid; we are constantly enveloped by it; we draw it in with every breath, and are always exposed to its evil effects, and yet less seems to be done to neutralise its injurious influence than that of impure water. Distinguished investigators, such as the late Dr. Angus Smith and Dr. G. H. Bailey, of Owens College, have done so much in establishing the composition of pure and impure air that it would be useless to add a word to what they have said. Whether in this city any improvement has taken place as regards the less obvious impurities of the atmosphere I am not prepared to say, but as to the most obvious and obnoxious impurity—I mean that of coal smoke—I have no doubt that matters are much worse than they were years ago. In consequence of my dwelling occupying an elevated position, some 200 feet above the level of the River Irwell, in Manchester, I enjoy the advantage of surveying a considerable portion of the two towns of Manchester and Salford, and smoke and its emanation come in for a large amount of attention. That the amount of smoke has largely increased is evident from the fact that distant objects, such as the hills of Cheshire and Derbyshire, and even buildings on these hills, were formerly visible on clear days, and are now no longer seen on any occasion whatever. That this increased amount of smoke is due, as is sometimes asserted, to the number of small tenements erected of late years is, I think, not correct. Any one looking down from a height on the town, with its numerous chimnies, must be convinced that the chief part of the smoke comes from factories and works generally, whether new or old, not from small tenements. The state of things now prevailing is a disgrace to our manufacturers, a disgrace to our municipalities, a disgrace to the community generally. So much has been said as regards the best means of abating the nuisance that it would seem to be useless to add another word. I am assured by those familiar with the subject that the amount of black smoke emitted by our chimnies might be much diminished by judicious and systematic firing on the part of workmen; but whether this is so, and to what extent, I am unable to say. Of course, the contamination of our atmosphere by coal smoke is not at all times equally offensive. In dull, close—particularly foggy—weather, whether in summer or winter, it is peculiarly objectionable, the more so since at such times the carbon of the smoke is mixed with sulphurous acid from the pyrites of the coal. On the other hand, during or after rainy weather, the atmosphere in Manchester, and perhaps in other places, is comparatively pure and pleasant, the impurities being washed down by the rain. This would suggest the possibility of producing in our streets something like artificial showers of rain. A rose with small holes being attached to the end of a water-hose, water might be made to descend in fine rain, washing down the suspended carbon, the sulphurous acid, and other impurities, at the same time watering our streets quite as efficiently as it is done at present by the driblets proceeding from our water-carts. I fear, however, that in spite of all that has been said about the unlimited supply of water in this town, there would not be a sufficient quantity to do this efficiently. The great distress caused to those suffering from pulmonary complaints by the dense fogs so often prevailing in this and other large towns has, I daresay, more than once suggested to inquirers the possibility of filtering the air before allowing it to enter our dwellings. The difficulties of such a device do not seem great in themselves; the real difficulty arises from the habits and prejudices of the people, who cannot understand the possibility of ventilation except it be through windows and doors. In his paper "On the Effects of Urban Fog upon Cultivated Plants," Prof. Oliver mentions with favour a plan devised by Mr. Thorp, a horticulturist of Stepney, who causes the air entering his hothouses to pass through boxes containing trays with sticks of charcoal before impinging on the hot-water pipes, an out-draught of air being secured by means of "exhaust caps" placed on or near the ridge of the house. The plan, in Prof. Oliver's opinion, is an efficient one. That any such plan would succeed in private houses, as at present constructed, is very doubtful, but in institutions, such as hospitals, especially consumption hospitals, some such scheme might be tried, an essential condition of success being that all entrance of air except through the ventilating boxes be entirely prevented. How necessary this would be may be seen by the fact that when a fog prevails the atmosphere inside our houses is effected though the doors and windows are as effectually closed as they can be with our present arrangements, and, on the other hand, the fog inside a house disappears simultaneously with that outside, showing that there is a ready communication between the exterior and interior atmosphere, even when an absolute calm prevails, which is usually the case during fogs.

To impurity of the atmosphere are sometimes ascribed effects with which I believe it is not fairly chargeable. Of the disappearance of several species of birds in the neighbourhood of Manchester during the last 20—30 years, even in places where vegetation has not apparently suffered, there can be no doubt; but I am not sure whether it is due to the greater prevalence of smoke. Birds as a rule are not sensitive to impurities in the atmosphere. I was assured by a chemical manufacturer at Basle, that swallows come every year to his works and build their nests above the aniline stills, where human beings can hardly breathe. Insects, on the other hand, are very sensitive to atmospheric impurities, and cannot live in contaminated air; on this is founded the practice of gardeners in smoking their houses with tobacco, whereby the insect pests are destroyed without injury being done to the plants. Insects having disappeared in particular neighbourhoods in consequence, say, of increase of smoke, insectiveous birds would of course disappear too, for birds generally dislike to have to seek their food at a distance from home. I venture therefore to suggest, though I do it under fear of censure on the part of hygienists, that a moderate amount of smoke may actually be beneficial to vegetation, by covering the leaves and other parts of plants with a thin coating of carbonaceous or tarry matter, and thus rendering them unpalatable to insects. Of course the amount would have to be exceedingly small; for, if excessive, we should soon experience deleterious effects such as are seen in the scanty and blighted vegetation in the immediate neighbourhood of our large towns.

Foreign Competition.

A great deal has been said of late as regards the dangers to our manufactures from competition on the part of foreign producers. Indissolubly as we are wedded to the principles of free trade, it nevertheless becomes a matter of some importance to ascertain whether we are losing ground in our competition with foreign producers, not only outside but it may be even within our own country. From the speeches which are now and then made, one would infer that the danger is real and imminent, and that it behoves us with all possible speed to build and endow technical schools in which our workmen and others shall be trained so as to enable them to compete with the more highly educated class of similar standing in Germany and France. I am myself too little in touch with the manufacturing industries of the present day to say whether the danger is real and present, or whether it is merely apprehended and distant. If, however, it be permitted to draw conclusions from what one sees in travelling on the Continent, one might infer that competition with this country in many articles of trade is, at present at least, quite chimerical. Who that has seen what is offered to us in France or Germany in the shape of furniture, glass, or china, can help thinking that were free trade established between these countries and ours, many of the poor inferior articles of their commerce would be swept away to give place to

really good, substantial ones such as we could furnish or which they could themselves produce if exposed to the healthy influence of competition. In some departments of industry, especially the chemical and more especially that of colouring matters, foreign competition is, I suppose, a very serious matter, but whether the evil is one that can be remedied by mechanical and educational measures seems to me doubtful. The fact is that successful foreign competition may be due not only to superiority of education and training, but also to certain inscrutable, perhaps moral qualities, of which it is difficult to estimate the weight and bearing. As long as human beings exist there will always be differences of talent and disposition between individual and individual, and one nation and another. In technical, as in other matters, special talents show themselves. The English have always been noted for great mechanical undertakings, the Germans and French for the manufacture of chemicals, especially those of organic nature. In the middle ages Germans were the great alchemists, and the alchemy of former times is represented, one may say, by the organic chemistry of to-day, a branch of science which may almost be said to have had its origin in Germany. That specialities such as we see developed on a large scale in the case of different nations are also seen on a smaller scale in our own and other countries is well known to everyone. Boots and shoes are made in one place, hats in another. The woollen trade flourishes in Yorkshire, and different branches of it in different towns. In this country likewise different branches of the cotton trade have localised themselves in various places. Why we should deplore this state of things, as we might in adopting the principles of protection, I am at a loss to understand. Why should we not, in accordance with the principles of free trade, buy what we want where we can buy it best or cheapest? Why should we not buy silks in France and chemicals in Germany? Why should we determine to make all we require ourselves? Why should we imitate the example of the Grecian sage of whom we read in our school days, who considered it the height of philosophy to make all one wanted oneself and who appeared at the Olympic games in garments which, as well as the rings on his fingers, were of his own manufacture? These questions have often been asked and been answered by us in this country in one sense, and in other countries in a contrary sense. I believe myself that the principles of free trade are unassailable and will eventually everywhere prevail. Nevertheless, there is one article of commerce as to the introduction of which into this country the most pronounced free-trader might hesitate—I mean that of beetroot sugar, which is largely imported into this country and offers a very formidable competition to the cane-sugar of our Colonies. The exportation of this sugar from the countries where it is produced is bolstered up, as everyone knows, by heavy bounties, making the competition of cane sugar in countries such as ours, where free trade prevails, exceedingly difficult. Into the policy of this system of bounties this is not the place to enter. But from a chemical point of view, a few questions may well be put as to the properties of the various sugars entering into consumption in this country. The chemistry of sugars having attained a high degree of development, and the distinction between various kinds of sugar being, it is supposed, well understood, it may nevertheless be asked whether cane sugar and beetroot sugar are in reality perfectly identical, and whether, though their general behaviour is the same, there may not be some recondite physical properties in which they do not coincide. In the opinion of some experts beetroot sugar is much sweeter and more soluble in water than cane sugar. I have been told of one, who has worked much with sugars, and who states that there is a great difference between cane sugar and beetroot sugar as regards the rapidity with which they undergo change, the latter being inverted much more rapidly than the former. I have been informed also, that when the two sugars are presented to bees the latter prefer cane sugar to beet sugar. I do not attach much importance to these observations even if correct, since, as regards human food, differences such as those referred to would be of little importance. Differences of a serious nature do, however, subsist between the two sugars as regards their respective mother-liquors or molasses—differences which might possibly be considered serious. The molasses of cane sugar may be and are largely consumed in the shape of treacle; those of beet sugar contain substances which render it entirely unfit for human food, and they are utilised accordingly in another way. Among these substances betaine is mentioned, and this there is good reason to suppose is a poison. Now it is quite possible that minute traces of this and other deleterious substances may be present in the finished product or sugar, and considering the enormous quantities of sugar consumed in this country the effect even of a minute poisonous impurity on the public health might be serious. However, poison or no poison, it might be maintained by purists—people who say that soda-water should contain soda, though the consumer may not want it, and arsenical soap sufficient arsenic to justify its name—that cane sugar and beet sugar should be respectively labelled as such. The whole subject is one that merits exhaustive inquiry. While on the subject of sugar, I may mention the fact, not generally known, that a distinguished chemist, Dr. Leon Marchlewski, a former collaborator of mine, has succeeded in preparing cane sugar artificially (see page 622). Following the lines laid down by Michael, who was the first to prepare an artificial glucoside, he brought together pure acetochlorhydrose and freshly prepared potassium levulosate in presence of alcohol, and obtained a small quantity of a product which had the properties of cane sugar. This important discovery, though it is not likely to have any immediate practical consequences, opens out a vista stretching far away into the *terra incognita* of the constitution of organic compounds. For this discovery the Academy of Sciences of Cracow awarded a prize to its author; if every other academy in Europe had done the same it would not have been more than the discoverer merited, considering the importance of his work.

There are other subjects to which I might have referred on this occasion, but considering that this address is already long enough for my powers of speaking, and probably too long for your patience, which must by this time be thoroughly exhausted, I will bring it to a conclusion without extending it further, thanking you heartily for listening to what I feel is scarcely worthy of the occasion.

Prof. F. Clowes (President-elect) moved a vote of thanks to Dr. Schunck for his address. Dr. Schunck, he said, might be old, but he was still in a large degree active. His reputation was world-wide, and he conferred dignity on the Society by assuming the Presidential chair. He remembered that, years ago, in Manchester, Dr. Schunck presided over Section B of the British Association, meeting in that theatre. Many foreign chemists were present, and one and all acknowledged his eminent position as a researcher. His address that day had been delightful. If there was one word that he would add to it, it was that, since the address was written, a bacterium had been found existing in strong spirits—in fact, in rum (see page 626). But that did not in any way affect the argument of Dr. Schunck.

Mr. G. Beilby, in seconding the motion, said that Dr. Schunck had spoken of the high tone prevailing in that Society. He would say that nothing would so much contribute to the maintenance of that tone as a study (such as that they had just heard) of the biographies of the great chemists who had preceded them. They were very grateful to Dr. Schunck for the charming reminiscences he had given them of Dalton and others, and it was a matter of satisfaction that his address would be preserved in the Journal. What the President had said about foreign competition would do the greatest good.

The motion was adopted amid applause.

Dr. Schunck, in responding, said he was grateful to the assembly for their great cordiality, and for the way in which they had listened to his long address. He only regretted that he had been unable to put into his paper something that might have been original, as other Presidents had done, but that could not be expected at his age. He thanked Prof. Clowes for his words, and would say with Mr. Beilby that they ought to read as much as possible of the history of the scientists.

THE COUNCIL.

The Secretary announced that the election of the Council had resulted as follows:—

President:

Prof. F. Clowes, D.Sc.

Vice-Presidents:

David Howard.	Dr. W. Jay Schieffelin.
Prof. A. K. Huntington.	Dr. Edward Schunck, F.R.S.
Ivan Levinstein.	G. N. Stoker.
Boverton Redwood.	Dr. T. E. Thorpe, F.R.S.
Sir H. E. Roscoe, F.R.S.	Wm. Thorp, B.Sc.
A. Gordon Salamon.	Thomas Tyrer.

Ordinary Members of Council:

G. Beilby.	Prof. J. J. Hummel.
R. Forbes Carpenter.	C. C. Hutchinson.
R. Le Neve Foster.	J. S. McArthur.
Sir David Gamble, Bart., C.B.	B. E. R. Newlands.
Oscar Guttmann.	John Pattinson.
Douglas Herman.	Walter F. Reid.

Honorary Treasurer: E. Rider Cook.

Hon. Foreign Secretary: Ludwig Mond, F.R.S.

AUDITORS.

On the motion of Mr. A. E. Fletcher, seconded by Mr. F. Napier Sutton, Messrs. Miall, Wilkins, and Co. were appointed auditors at a fee of 10*l.* 10*s.*

IMPERIAL INSTITUTE REPRESENTATIVES.

Prof. A. PEDLER proposed that Mr. E. Rider Cook be the Society's representative on the Imperial Institute. As one who had been connected with India for a quarter of a century, he said he knew the good which that institution might do if properly directed. The other day he went through the Research Department of the Institute, and noted that a line had been struck out which he thought might be of considerable advantage to India and the various colonies.

Mr. T. C. CLOUD, of South Australia, seconded the motion, which was passed.

NEXT YEAR'S MEETING.

Sir JOHN TURNEY, speaking on behalf of the Mayor and Corporation of the city of Nottingham, invited the Society to meet in Nottingham next year. In view of the election of Prof. Clowes to the Presidency, their city would be an appropriate meeting-place, and the members would have every welcome.

Mr. F. J. R. CARULLA, in supporting the invitation, said this was the first time they had been asked to come to a place by a municipality.

On the motion of Mr. Ivan Levinstein, seconded by Mr. B. Redwood, the invitation was accepted.

VOTE OF THANKS.

Mr. GEO. E. DAVIS proposed a vote of thanks to the Council of the Owens College for the loan of the Chemical Theatre as a meeting-place.

Dr. KOHN seconded the resolution, which was passed.

This concluded the proceedings.

VISITS TO WORKS AND PLACES OF LOCAL INTEREST, &C.

After the conclusion of the morning's business, by invitation of the Manchester Section, luncheon was served in the hall of the Owens College Union. Mr. Tyrer then proposed the health of the Chairman of the Manchester Section, Mr. George E. Davis, warmly referring to the signal services he had rendered the Society in its earliest days, as one of its original founders.

After lunch a large number of the visitors, with their ladies, were conveyed to the residence of the President, at Kersal.

DR. SCHUNCK'S GARDEN PARTY.

On arrival, the party was cordially received by Dr. Schunck, and whilst some passed the time in the grounds, where refreshments were provided, and the noted band of Besses o' th' Barn was performing in a marquee, others went to inspect the President's splendid laboratory and scientific library, and to see the interesting exhibits there set out. Dr. Schunck's library contains about 1,000 volumes, including Liebig's Annalen, the Berliner Berichte, and the Philosophical Transactions, complete from the first volume of each work. The exhibits and specimens included the following, many of them, as it will be seen, serving to illustrate the President's address delivered in the morning.

Chlorophyll Derivatives.—Phylloxanthin, Phyllocyanin, Phyllotaonin, Methyl and Ethyl-phyllotaonin, Alkachlorophyll, and Phylloporphyrin.

Indigo and its Decomposition Products.—Indican, Indigotin, Diacetyl-indigotin, Diacetyl-indigo-white, artificial Indigotin, Diacetyl-dihydroxy-indigotin, O'Neill's acid, Indirubin, artificial Indirubin, Acetyl-indileucin. Isatin and substitution products, Hydrazones and Semicarbazones of isatin and substitution products, Amidophenimesatins, Indophenazins, Indoxyl and Di-oxindol, Anthranilic acid, Nitrosalicylic acids.

Madder and Munjeet Constituents.—Rubian. Rubiadin and derivatives. *d*-Glucose from rubiadin, Ruberythric acid and acetyl derivatives. Alizarin, Purpurin, Purpuroxanthin and derivatives. Perchlorrubiadin.

Natural Yellow Colouring Matters.—Quercitrin, Quercetin and substitution products. *d*-Rhamnose, Sophorin, Rutin, and derivatives. Xanthorhamnin and derivatives, &c.

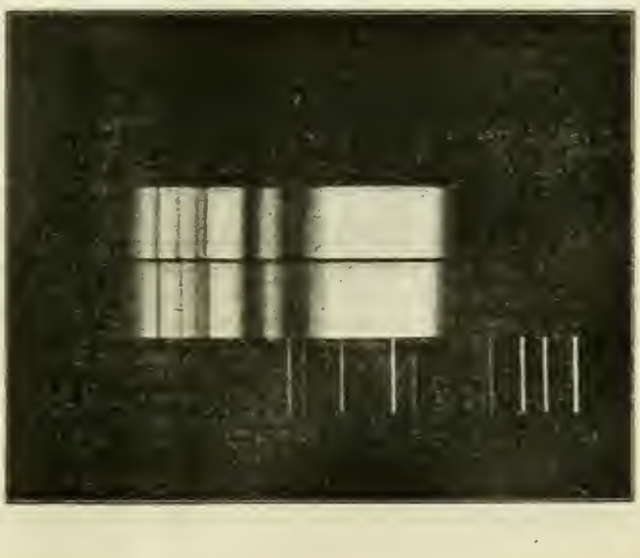
Natural Red Colouring Matters.—Carminic acid (crystallised) and derivatives. Crocetin and crocin and derivatives. Carthamic acid, Brasilin and derivatives, &c.

Glucosides.—Phlorizin and derivatives. Salicin, Arbutin, Aesculin and derivatives.

Carbohydrates and Allied Substances.—*d*-Glucose, *d*-fructose, *d*-gallactose and derivatives, Cane sugar, natural and artificial. The artificial sugar was synthesized for the first time by L. Marchlewski (see pages 594 and 622). Milk sugar, Maltose, Mannitol and derivatives. Inulin, Trahelose.

Anthracene Derivatives.—Anthracene and substitution products. Anthraquinone, &c., Alizarin, Purpuroxanthin, Quinizarin and derivatives, Anthraflavic acid, Isoanthraflavic acid, Anthrarufin and derivatives, &c., &c.

Naphthalene Derivatives.—Naphthalene and substitution products. Naphthols, Naphthylamines and ? substitution products, &c., &c.



Mr. Chas. A. Schunck demonstrated the spectra of Phylloporphyrin and Hæmatoporphyrin (see President's address, page 590), which, as will be seen from the figure on the preceding page, are practically the same; the only difference being, that in the case of hæmatoporphyrin the seven bands are slightly nearer the red end of the spectrum. Of these seven bands, the first in the red, as will be seen, is narrow, dark, and extremely well defined. It is the characteristic band. Then comes a very faint band near to the first, and just visible in the photograph but not in the figure, and then another faint band. After this appears a double one, in strong solutions looking like one uniform band, and finally two very dark bands. The wave lengths of the centre of the bands are approximately:—6250, 6120, 5950, 5750, 5650, 5250, 4900. The general absorption in the violet begins at about wave length 4370.

Instead of proceeding directly to the President's garden party (3 to 6 p.m.), a choice was offered of visits to several works and institutions of interest in the neighbourhood. These were as follows:—

The Manchester Crematorium.

Henry Simon, Esq., C.E., Chairman.

The Crematorium is situated near Chorlton-cum-Hardy. It consists of a hall or chapel and a separate contiguous chamber containing the furnace. The hall is about 50 ft. long by 25 ft. wide, and of proportionate height. In the centre of the wall, opposite to the principal entrance, is placed the aperture leading to the furnace, which, in the separate space, occupies basement and ground floor. A vestry or record room, and a retiring room, lavatories, &c., are situated at the back of the hall. The style of the building is Romanesque, similar to that of some of the churches of Lombardy and Venice, and the material is terra-cotta. The furnace is considered to be ready for cremation as soon as the cremating chambers have attained a bright orange-coloured glow. The body can then be introduced, and after about an hour the process is complete. The intense heat is such that of the wood of the coffin (light pine wood) absolutely no trace remains in the ash of cremation. At present, when the large furnace has to be heated up specially for each single cremation, the quantity of coke used amounts to something like one ton. Each cremation immediately following, however, would only require an additional two or three hundredweight of coke. In Paris, for example, where 10 or 20 cremations take place daily in the Père la Chaise Cemetery, the cost of fuel is very small indeed.

It is worthy of mention that the coffin, from its resting place in the chapel, at a given signal, passes by invisible mechanical means through the small door of the draped antechamber, to the furnace.

The intense heat used in the furnace for the Manchester Crematorium is said to be an absolute guarantee against the possibility of nuisance of any kind. In the base of the chimney—which occupies the centre of the tower attached to the Crematorium, and has a height of 75 feet—there is a second coke fire provided, through which the gases proceeding from the principal furnace can be passed. It has so far been found totally unnecessary to use this fire.

The arguments given for the crematorium and cremation are based on statements by Lord Playfair and Sir Henry Thompson: the former to the effect that "in most of our churchyards the dead are harming the living by destroying the soil, fouling the air, contaminating water-springs, and spreading the seeds of disease"; the latter to the effect that even in the case of suburban cemeteries, "these latter rapidly become urban, and will one day be found in dangerous vicinity to our homes."

The Manchester Corporation Gasworks, Gaythorn.

The Gaythorn Gasworks, thrown open to members, covers eight acres of ground. There are 65 beds of retorts, the number of mouthpieces being 776. The systems of charging are (1) by hand, (2) the Foulis-cum-Woodward system. The total carbonising power is 430 tons, and the producing power 4,300,000 cubic feet, the gas-holder capacity being 6,010,000 cubic feet. The ratio of storage to consumption is 113·5 per cent. The corresponding consumption of cannel is 18,000 tons, with 4,000 tons of coke, and the yield of tar and ammoniacal liquor is 750,000 gallons. The largest daily consumption of gas in Manchester recently, was on January 11th, 1897; it was 23,824,000 cubic feet, the largest weekly consumption being for the week ending December 24th, 1896, with 136,000,000 cubic feet. Of the works furnishing this supply of gas, Gaythorn is only one of several, the others being the Bradford Road Works, the Rochdale Road Works, and the Droylsden and Poland Street Works.

The total area of supply is: Within the city, 12,788 acres; beyond the city, 12,382 acres; and beyond the Mersey, 4,640 acres. Total, 29,810 acres. Extreme limit of supply, 10 miles. Percentage of consumption: Within the city, 86·58 per cent; beyond the city, 12·66 per cent.; beyond the Mersey, 0·76 per cent. Total number of lamps within and beyond the city, 16,000.

At the Bradford Road Gasworks a carburetted water-gas plant is being laid down on Humphrey and Glasgow's system, to make, when completed, 6,000,000 feet of gas per diem.

The Manchester Corporation Electric Lighting Works.

The generating station is in Dickenson Street, near Portland Street. The works were designed and their erection superintended by Dr. John Hopkinson, F.R.S. The generating plant had a capacity of 3,000 h.p., though the buildings were constructed of a size to admit of considerable additions. In July 1894, the resident engineer, Mr. C. H. Wordingham, took over the consulting work, and since this all extensions have been carried out to his designs. The capacity of the generating plant is now 6,200 h.p., and this will be increased to 10,600 h.p. when the extensions now in progress are completed. The system employed is entirely low-pressure, and is the multiple-wire direct current, with feeders. The greater portion is on the five-wire system, the pressure of supply to consumers being at 100 volts, whilst a portion is on the three-wire system, the pressure of supply to consumers then being at 200 volts. The following small table will indicate the rate of increase of demand for electric lighting, as well as present supply:—

Year ending March 31st.	Quantity sold.	Total Maximum Supply demanded.
	B.T. Units.	Watts.
1894 (8 months) ..	439,370	651,500
1895	1,168,382	1,187,000
1896	1,748,244	2,082,600
1897	2,508,588	2,737,400

The Hydraulic Power Supply, Gloucester Street.

The Hydraulic Pumping Station is adjacent to the Rochdale Canal, Oxford Street, and the mains are kept charged by means of six sets of powerful engines, each capable of delivering 230 gallons of power-water per minute. Steam is generated in five large boilers, fitted with mechanical stokers, economisers, coal conveyors, &c. The reservoir of power consists of two capacious accumulators, loaded to a maximum pressure of 1,120 lb. per sq. in., thus producing the same effect as if large supply tanks were placed at an elevation of over 2,500 ft. above the ground level. The power-water will be maintained in the mains at a pressure of 1,000 lb. per sq. in., and is available day and night within the area of supply all the year round, Sundays and holidays included.

The system of supply has been in operation for nearly two years, and upwards of 13½ miles of mains are now laid within the principal streets in the city, from which supplies are given to over 240 premises, and representing about 870 machines for various purposes.

The Manchester Corporation Cold-Air Stores, Elm Street.

In 1893, the Markets Committee of the Manchester Corporation resolved to establish—as an adjunct to their extensive markets—cold-air stores for the preservation of perishable goods, the main object being to minimise the hitherto great waste of the meat supplies of the city and

neighbourhood, and to furnish these supplies to the people in a sound and wholesome condition. The buildings were commenced in June 1893, and completed and equipped with machinery, &c., on January 31st, 1895. These stores consist of a square block of brick buildings, about 130 ft. long by 120 ft. wide, and 75 ft. high from the basement floor, with provision for extension upwards, should trade requirements demand it. They contain a basement, ground floor, and three upper floors. The building is constructed to accommodate 120,000 sheep, distributed as follows:—In the basement, about 20,000 sheep or other goods. In the six chill rooms on the ground floor, about 25,000 sheep, or 1,200 sides of beef or other goods. On the first, second, and third floors, about 25,000 sheep on each floor. With regard to the heights of the rooms, these are as follows:—Basement, 10 ft.; ground floor, to accommodate sides of beef, 13 ft.; the first, second, and third floors, 9 ft. each. The roadways through the building are to have a clear headroom of 17 feet, and a clear width of 19—20 ft.

The plant consists of:—Two triple-expansion vertical marine engines, each driving direct two 50-ton Linde compressors. Each engine and two compressors are capable of refrigerating the whole of the chambers in the entire building to any reasonable temperature required, as well as chilling 1,200 sides of beef in 24 hours. Three Babcock and Wilcox boilers supply the steam necessary for working the steam engines, any two of which boilers can work both the refrigerating machines simultaneously, as well as two powerful electrical plants driven by special compound condensing steam engines, which are also interchangeable—one for the purpose of driving and circulating the air through the chambers, and the other for lighting the premises.

The Manchester and Salford Technical Schools, Princess Street, Manchester, and Peel Park, Salford, were also thrown open to the inspection of the members.

Reception by the Lord Mayor of Manchester and the Lady Mayoress, in the Manchester City Hall.

In the evening, the members of the Society, with their ladies, were cordially received and entertained in the City Hall by the Lord Mayor of Manchester, Mr. Alderman J. F. Roberts, and the Lady Mayoress, Miss Roberts. An admirable programme of music was provided. Mr. Herbert Walker, Mr. Roland Hoyle, Mr. Bertram Smith, Miss Nellie Leeson, and Miss Adela Carlton took part in the singing, while some fine instrumental music was played by the band of Messrs. Forsyth Bros. in the Great Hall. Nearly 700 ladies and gentlemen were present.

SECOND DAY.—Thursday, July 15.

Sir Thos. Wardle's Leek Excursion and the Manchester Ship Canal Visit.

Thursday may be said to have been spent in two directions—one party of about 50 leaving the landing stage of the Ship Canal in Manchester, opposite the Cathedral, at 9.30 a.m. for an excursion as far as Latchford; another party of over 80 accepting Sir Thos. Wardle's invitation to Leek, Staffordshire. This party was received by Sir Thos. Wardle and Miss Wardle at Leek Station, and conveyed in wagonettes to the Dyeworks, managed by Mr. Gilbert Wardle, where the members were divided into groups and separately conducted through the different departments of the works by Sir Thos. Wardle and his son.

At one o'clock the party was driven back to Leek, and there entertained at lunch by Sir Thos. Wardle, whose efforts to entertain and please his visitors were unceasing, and, it may be added, completely successful.

After lunch, Dr. Bowman proposed, and Dr. Clowes seconded, the health of Sir Thos. Wardle and family, and Mr. Tyrer proposed a vote of thanks to Sir Thomas for his generous entertainment of the visitors, and for so kindly throwing open his works for their benefit.

Sir Thos. Wardle's Silk Dyeworks.

The visitors were shown here the various kinds of machinery for washing the silk; also various processes of dyeing black, more especially those for the dyeing of the warp and weft for the sailors' handkerchiefs ordered each year by the Admiralty, in which the weighting is stipulated to be very low, and the permanence of the dyes is secured. The dyeing of silk in various colours excited considerable interest, as also the bleaching of Tussah silk, in which the natural yellow colour is reduced to a clear and pure white, to be followed afterwards by dyeing to a variety of the most delicate and pale shades of colour. The dyeing of the Raven black, for which the firm has maintained for long a just celebrity, was next shown to the visitors. This is an unweighted colour, resembling that of the bluish-black of the raven's wing, in the production of which an extremely dilute solution of tannate of iron, produced by a mixture of iron and the bark of the alder tree, plays an important part. The dyeing of all kinds of black colours and shades of white was also demonstrated. Apparatus was seen and explained for wringing, stringing, dyeing, and lustreing of silk.

Excursion from Leek over Axe Edge.

At 3 p.m. the party proceeded, by carriages and brakes provided by Sir Thos. Wardle, over Axe Edge—2,000 feet high, and surrounded by lovely hill and dale scenery—to Buxton, the visitors thence journeying to Manchester at 5.30 p.m., so as to be in time for

The Annual Dinner.

The Annual Dinner was held on Thursday, July 15th, at 8 p.m., in the Grand Hotel. The President, Dr. Schunck, occupied the chair, and among those present were Prof. Clowes (President-elect), Sir Henry Roscoe, Mr. Tyrer, Mr. G. E. Davis (Chairman of the Manchester Section), Mr. Ivan Levinstein, Dr. Bowman, Mr. G. Beilby, Dr. Macadam, Dr. Böttinger (Elberfeld), Prof. Alex. Pedler (Calcutta), and Mr. J. Carter Bell.

The President proposed the toast of the Queen, which was duly honoured.

Prof. F. Clowes, D.Sc., proposed "The Society of Chemical Industry." He thanked the members for the honour they had done him at the recent annual meeting in electing him to succeed Dr. Schunck as President. Dr. Schunck had regretted that the burden of years had prevented him from doing as much as he would have liked for the Society. Well, he was young enough to be Dr. Schunck's son: and if comparative youth was an advantage, he hoped to be able to justify to some extent the confidence the Society had placed in him. He felt that in selecting him as their President for the new year, they were really doing honour to the Nottingham Section, which in some measure he represented. The Nottingham Section was one of the youngest members of their large and growing family. Started in connection with the University College, it had become a centre of great interest, and included members from Birmingham, Derby, Leicester, Burton, and, in fact, all the midland towns within easy access by train. The widespread interest in the work of the Section had led the committee to arrange for occasional meetings in the various towns in which they had a body of members. He believed no other Section of the Society followed this practice, but he thought it might with advantage be imitated in other Sections. It had led, in the case of Nottingham, to a very wide interest in the work of the Section—wider, he believed, than in any other Section. He mentioned these things to show that, though a young section and a small section, they were full of energy, and had done something for the general good of the Society. The membership, as the report of Council had told them, was now over 3,000. This was a large constituency, and he was glad to say it was not confined to the United Kingdom. The success which had attended the Society must be very gratifying to the men of Manchester who founded it, and especially to Sir Henry Roscoe, its first president; and it was a proof that the Society had met a want felt by chemists, not only in this country, but in other countries as well. He trusted that the classification, sometimes attempted, into professional, professorial, and industrial chemists would not be pushed too far. All chemists were now very generally trained up to a certain point in the same way, and their qualifications and

pursuits were to a great extent identical. He coupled the toast with the name of the retiring President.

Dr. Schunck, in responding, said the Society was sometimes said to be on the verge of bankruptcy, but somehow or other they not only kept out of difficulty but prospered. No society ever did die of bankruptcy, and he had no fear for the Society of Chemical Industry. During his presidency he had been struck with the want of cohesion between the different Sections of the Society. In one way it was a peculiar society, consisting, as it did, of sections, each of which met separately. Between the various Sections there was far too little connection. For example, there were the Liverpool and Manchester Sections; they were not far from each other, and yet, for any connection there was between them, one of them might be meeting at Timbuctoo. Of course, the Journal of the Society helped to keep the Sections together, and let them know what was going on; but this was not enough—where it could be managed Sections should occasionally have joint meetings. They in Manchester, for example, should go to Liverpool now and then, and have an evening there, and the Liverpool Section should, in the same way, occasionally come to Manchester. Such joint meetings would add to the unity, concord, and good fellowship of the members. A good deal had been done in this direction by his predecessor in the chair, who was in the habit of making visits to the Sections. This, of course, was very good so far as it went, but could not have all the pleasant consequences one would anticipate from a visit of one section in a body to another section. Another thing which struck him as a defect in the constitution of the Society was the lack of opportunity for the discussion by the Society of important questions which cropped up from time to time. There was only one general meeting during the year, and as an immense quantity of business had to be got through in a short time, there was no opportunity for taking into consideration questions of general interest. An example of this had occurred on the previous day, when a highly esteemed member of the Society brought forward a question which had to be answered very shortly, though it might well have been discussed for an hour or two. There ought, he thought, to be a regular meeting of the Society for the discussion of such questions—questions of vital interest, and not merely business questions. These were, perhaps, the only points of importance which had struck him during his presidency. Unfortunately he had not been able, like his predecessor, to go about among the Sections, but he had done what an old man could to advance the general interests of the Society.

Mr. T. Tyrer proposed the toast of "Science and Industry." He said that no technological work could be successfully carried on without the application of scientific principles; that was why in some cases chemical industries did not succeed so rapidly as they might. He believed we were rapidly getting out of the region of rule of thumb. Foremen were beginning to believe that masters knew as much as they did themselves, and it would be a happy day in the chemical industry when the workmen came to be of the same opinion. The want of success in chemical industry was often due to the obstinacy of the British workman. There was certainly great need for better discipline among the men—better discipline and a more ready and prompt obedience. A good deal was said from time to time about foreign competition, and particularly German competition. His own view was that with equal conditions we had little reason to fear; but it was worth bearing in mind that in some respects the conditions were not at present equal—for instance, in the education and discipline of our workmen. He was glad to have the privilege of connecting Sir Henry Roscoe's name with the toast, as representing the first branch of it. No worthier representative of chemistry could be found than the first President of the Society, to whom they were under a great debt of gratitude for his early work in connection with the Society. Sir Henry saw that if it was to become an international society it must have its headquarters in London. Still they could never forget that its was born in the energetic atmosphere of Manchester. Mr. Tyrer also spoke of the work done by Sir Henry Roscoe at Owens College, and the large number of good men he had turned out from his classes. One secret of his success was that, like Bunsen and Hofmann, Sir Henry worked side by side with his students in the laboratories, and was not content with a mere perfunctory supervision.

Sir Henry Roscoe, in responding, said this was the third time the Society had visited this great hive of industry, and it was a fact on which both the Society and Manchester were to be congratulated. It was true, as one of the speakers had said, that he had taken some share in the work of starting the Society. He remembered Mr. Muspratt, Mr. Carey, and one or two other gentlemen interested in the chemical industry, coming to Owens College to consult with him as to how they could best bring together the men of science and the men of practice. The first idea was to make it a local institution, but he suggested that it would be better to take in the whole country. The result had been extremely encouraging. Indeed he knew of no society of an industrial and scientific character which in this generation had been so successful. Perhaps no other chemical society had so many members. The existence of the Society was of itself a proof of the need for a close connection between science and industry. But he need not preach to the converted. They all knew as well as he that industry without science was nothing but rule of thumb; and rule of thumb, he was glad to say, was dead. If we were to maintain the supremacy of England in chemical and other industries, we must unite industry with science in its latest and best forms. A good deal had been said about German industries, and particularly chemical industries. Well, they all admired the work the Germans were doing in that direction, but it was not to be supposed that we were doing nothing of the same kind. It was, indeed, not too much to say that the greatest discoveries in science had been made by Englishmen; but in saying that he had no wish to belittle the work done on the Continent. There were many things in which we could learn from our German friends; they had certainly produced some wonderful results in regard to colour.

Dr. F. H. Bowman also responded to the toast, speaking on behalf of industry. He said that science was, after all, only the rational interpretation of the nature of things; and if they worked contrary to the nature of things they could not hope for the best results. It was a good omen for the future of the country that we were now thoroughly aware of the fact that our success would depend on the scientific training of the rising generation. He had been asked specially to reply for cotton. At one time he employed in that industry nearly a thousand hands, and he had found many directions in which the scientific spirit could be applied. He had the same experience in the woollen industry; and, indeed, it would not be easy to find an industry in which science had nothing to say. The Society, he was glad to think, was doing much to stimulate the scientific spirit in this great country.

Mr. G. Beilby proposed the health of "The Guests," coupling the toast with the name of Dr. Böttinger, of Elberfeld, the head of one of the largest chemical works in Germany.

Dr. H. T. Böttinger said that though he was a member of the Society, he might, being a foreigner, fairly enough consider himself a guest in Manchester. Meetings of this kind seemed to him to be of great value, if for no other reason than that they enabled them for a time to get away from the small jealousies of commerce. England had good reason to be proud of the position it occupied in the chemical industry. So far as inorganic chemistry was concerned England had the first place; in Germany they had gone in more for the development of organic chemistry. He hoped the spirit of commercial and scientific fraternity would always be the governing spirit of the Society.

Dr. Macadam proposed "The Local Section." He said the Manchester Section was an extremely strong one, and had done much good work. To say that it was managed by Manchester men was another way of saying that the management could not be improved.

Mr. H. Grimshaw and Mr. Carter Bell responded, and this brought the speaking to a close.

THIRD DAY.—Friday, July 16.

Visit and Excursion to Beeston and Peckforton Castles.

Still favoured, as all through these meetings, with exceptionally magnificent weather, a very large party of members and their ladies assembled at London Road Station at 10 a.m., and took train to Beeston, *viâ* Crewe. After ascending the hill crowned by the ruins of the ancient castle, the party partook of lunch in a marquee. Later on a small party visited Peckforton Castle. The party then drove in wagonettes to the Iron Bridge, where the ladies and those not electing to return to the Smoking Concert, remained for tea, the rest of the party driving on to Chester direct, and then taking train for Manchester. After tea, whilst one portion walked on to visit Eaton Hall, the seat of the Duke of Westminster, the remainder were entertained, *al fresco*, with humorous recitations, readings, and songs, by Messrs. Watson Smith, A. H. Allen, Dr. Bowman, and Mr. Turner. They then sailed down the river to Chester, returning by train to Manchester at about 10 p.m.

The Smoking Concert.

Those who left Chester for Manchester at an earlier hour, joined in a Smoking Concert at the Grand Hotel, at 8.30 p.m. This function was presided over by Dr. J. Lewkowitsch, whose name will be remembered in connection with the successful smoking concert of 1895, in Leeds. Here, as in Leeds, an attractive programme of recitations, songs, humorous speeches, and narratives was carried out, and much amusement was caused by Dr. Lewkowitsch's humorous and suggestive proposal for a "new process, based on strictly scientific principles, for treating and purifying alkali and air." In this the services of a variety of waste products, such as spent soap lyes, canal, street, and kitchen greases, and various works' residues, vapours, &c. were to be enlisted, most, if not all, the recovered or resuscitated articles of value being shown upon the table!

FOURTH DAY.—Saturday, July 24.

Visit to the Works of Ivan Levinstein and Co., Limited.

By special invitation a party of about 40 assembled at the Grand Hotel at 10 a.m., and proceeded in wagonettes to Crumpsall, where the extensive works of Levinstein, Ltd., are situated. Here they alighted and were shown round the various departments of the factory by Mr. Levinstein himself, who explained in outline the leading processes involved in preparing from crude naphthalene, the naphthols, α-naphthylamine, and naphthionic acid, as well as some of their azo derivatives, which form naphthalene dyestuffs.

He also showed the company that so large is the firm's business in the manufacture and export of naphthol and naphthylamine, &c., that it was deemed necessary to engage in the manufacture and rectification, on the spot, of sulphuric acid, and the sulphuric acid chambers, pyrites kilns, and rectifying plant were exhibited and explained.

Mr. Ivan Levinstein commenced coal-tar colour manufacturing at Blackley, about 30 years ago, on a very small scale, the range being then confined to Magentas, Violets, Bismarck Brown, Chrysoidine, Blackley Blue, &c. These works were then transferred to Crumpsall about 12 years ago. About 400 people are employed in the present works, and the chemical staff, inclusive of that in the dyeing laboratories, comprises 18 head chemists, 12 assistant chemists, and about 15 youths, apprentices, and boys. There are special laboratories, solely designed for scientific research, in which about eight chemists are engaged.

As the party proceeded to the inspection of the vitriol plant, attention was drawn to a large number of casks containing naphthalene derivatives intended for Germany, all of which were conspicuously marked "Made in England"—a fact which seemed to afford considerable amusement to the visitors.

The vitriol plant, which had been designed by Mr. Geo. E. Davis, was producing 100 tons of acid per week, all of which was used on the works. The rectifying plant consists of lipped glass beakers arranged in descending series, and brings the chamber acid up to 96 per cent., 97 per cent., and 98 per cent. acid. Glass retorts are used. The remarkable freedom of the retort house from the irritating fumes of sulphuric acid was the subject of remark by several of the visitors, and Mr. Levinstein added that remarks to the same effect were made either by the district or the chief alkali inspector whenever he visited the works. Mr. Levinstein drew attention to a notable feature of the works, namely, that there was no transport or handling of vitriol in carboys anywhere, but the acid was collected in a central cistern near the rectifying house and was forced by means of air pressure and acid eggs into secondary reservoirs, situated in the various departments, where it was simply necessary to draw off into measuring boxes the required amount of acid for the individual operations. Wrought-iron pipes were used, and were preferable, on account of their lightness, for the overhead transport of the acid. The present installation had been in use six years, and was still good. After the nitric acid plant, which was of the usual type, though characterised by several minor conveniences in regard to arrangement, had been visited, the inspection of the plant for the manufacture of the organic products was proceeded to.

The naphthalene stills were first visited. These are charged with naphthalene from a separate store, and are wholly separated from the condensing and rectifying plant, thus minimising the risk of fire which always attends the distillation of this product. Between 400,000 and 500,000 lb. are rectified per month, the product obtained from the first distillation being subsequently twice distilled, finally refined, and crushed, when it is ready for use in the various departments.

The plant for the manufacture of nitronaphthalene was next visited; the importance of preventing the formation of the dinitro product was explained by Mr. Levinstein, who stated that 180,000 to 200,000 lb. of the nitro-compound were produced per month. Proceeding, the party passed through the reduction house, where the nitro-compound is reduced with hydrogen (obtained from iron filings and hydrochloric acid) to form naphthylamine. It was shown that great efforts are made to remove all vapours of the ill-smelling α-naphthylamine from the atmosphere by means of a vacuum apparatus, which draws off the air of the room and forces it to a fireplace or furnace.

In the next department visited, the process has to be carried on in retorts under a pressure of a ton to the square inch, and the elaborate precautions and tests made to ensure the safety of those working in this department were explained by Mr. Levinstein. Visiting the naphthol distillery, Mr. Levinstein dwelt upon the difficulties of dealing with this product, and he showed the ingenious methods that had been exercised in devising this plant. It is significant of how greatly chemical engineering affects the progress of chemical manufactures; for, despite the difficulties of preparation, the consumers were demanding greater purity every year, although the product—which is now produced in what is practically a state of chemical purity—sells at fewer pence per pound than it fetched shillings some years ago.

On the way to the colour-making plant the central air-compressing station was passed, from whence, apart from the numerous small compressors employed on the different plants, a supply of compressed air is furnished to all parts of the works by a splendid compressor built by Messrs. Klein, Schanzlin, and Becker, of Frankenthal. At the colour-drying stoves also a new vacuum drier was shown, which, besides having the advantage of occupying 15 times less space than the ordinary stove, enables the colours to be dried at as low a temperature as 30° C.

The visitors were then shown the actual manufacture of naphthol scarlet—a colouring matter which has now almost entirely superseded cochineal.

Before leaving the works, specimens of cotton and woollen materials dyed with the Company's patented colours were inspected.

As already indicated, Messrs. Levinstein's firm is chiefly concerned in the manufacture of naphthalene derivatives, and in this special line, it is said to be the largest in Europe, two-thirds of the total production being exported to foreign parts. The different departments, each of which is managed by a staff of chemists are :—

Intermediate Products.

	Lb. per Month.
Naphthalene, with a production of about	400,000
Naphthol " " "	100,000
Naphthylamine " " "	180,000
Sodium naphthionate " "	130,000
Isomeric naphthol and naphthylamine sulphonic acids, with a production of about.....	80,000

	Tons per Month.	
Sulphuric acid, with a production of about..	400	For the firm's consumption.
Nitric acid " " " ..	70	
Benzidine derivatives " " ..	10	

Dyestuffs.

The special dyestuffs produced, are in the main derived from naphthalene, such as diazo colours, Scarlets, Fast Red, Yellows, Oranges, Clarets, Browns, &c., and Tetrazo colours, also direct dyeing colours, such as Congo Red, Benzopurpurin, Benzo Blue, Chrysamine, and other Salicylic acid dyestuffs.

Messrs. Levinstein's own patented dyestuffs are :—

Coomassie Black ; Coomassie Navy Blue, for wool ; Crumpsall Yellows and Oranges, for dyers and printers ; Union Fast Claret ; Dianol Red ; Dianol Brown ; Direct Blacks, for cotton and Union cloths ; Crumpsall Direct Fast Browns and Reds.

After the inspection of the works was concluded, the visitors were driven back to the Grand Hotel, where Mr. Levinstein entertained them at lunch.

In an address, Mr. Levinstein said that he had thrown open the works of Levinstein, Limited, to members of the Society of Chemical Industry for the purpose of demonstrating to them—especially to his friends from the South—that the coal-tar industry was not only in existence, but that it was expanding in this country in spite of all that had been said to the contrary. He acknowledged that we were behind Germany in the quantity and variety of products manufactured from coal tar, but we were gaining ground, and with an amended patent law which would not, as hitherto, give all advantages to our foreign rivals at our expense, our progress would be still more noticeable. He referred to the International Congress on Technical Education, and thought that some of the professors had been rather hard on the manufacturers in blaming them for our backward position in regard to the chemical industry. It was stated at the Congress that this country was suffering from the fact that manufacturers did not appreciate the importance of highly trained men, and if they would only recognise the value of such work, we could turn out men as well as any other country in the world. Mr. Levinstein was, however, of opinion that in science the maxim that "the demand regulates the supply" does not hold good—the very opposite was the truth. Long before Germany possessed a chemical industry of any importance great schools were established in different parts of the country, directed by masters such as Woehler, Liebig, Kekulé, Hofmann, Baeyer, Victor Meyer, Fischer, and others, and to them and their disciples belonged the credit that German manufacturers were in a very much better position by having at their command a large number of highly trained men to take advantage of the discovery and the development of the coal-tar industry, first founded by an Englishman. It was undoubtedly the fact that the position which Germany held to-day in the chemical industries was due to their great masters in organic chemistry, and to their patent laws ; whilst we, on the one hand, did not possess the highly trained staff of men, and, on the other hand, stifled our inventive genius and handicapped our industry by absurd laws. That our manufacturers did appreciate highly trained men was best proved by the fact that many hundred foreign chemists were employed in British works. Mr. Levinstein also disputed the statement that we at the present time could turn out, in sufficient numbers, organic chemists of such training as were required by our manufacturers. Our whole educational machinery was yet insufficient for that purpose. The only laboratory that existed in this country which was solely devoted to the study of organic chemistry was at the Owens College. Its existence was only of recent date, but in spite of that, under the direction of his eminent friend Prof. Perkin, it had already made its mark by turning out men of a very superior class compared with those of former years, and those who had devoted sufficient time to their studies could readily find suitable employment ; indeed, not long ago the director had more applications from manufacturers than he could satisfy. What they required was a number of organic chemical schools under eminent masters, similar to the Schorlemmer laboratory at Owens, a better preparatory education of the students when entering, and a more prolonged term of study. An organic chemist could not be sufficiently trained and prepared for works, either in Germany or here, under five years. Our manufacturers were, however, suffering from more serious causes than the insufficient training of our chemists. They were handicapped by the iniquities of our patent laws, to which he had repeatedly drawn attention, and unless these were amended all our efforts in regard to improved education would never bring back to us an industry which ought to be solely and truly a British one.

In illustration of his point Mr. Levinstein mentioned the fact that a new organic acid was invented by a large chemical firm in Germany. That firm did not know what to do with it. Three years afterwards another chemical firm in Germany made an important use of it. The inventor brought an action against them, but the German Government upset the patent. The Government said that the first inventor did not know what to do with his patent, and it was therefore of no use to commerce and industry. But in England the monopoly of the original firm was maintained. They never manufactured an ounce of acid in England, but they threatened with proceedings any English firm that produced the article. The consequence was that our chemists here were prevented from using this acid in new combinations, and thus our inventors' genius was stifled. Our manufacturers were prevented from producing it, whilst in Germany the invention was common property. A very important industry had, in Germany, been based on this acid, but here manufacturers up to this date could not use it. It was an example of unfairness in our patent laws.

Dr. Schunck, in proposing the health of Mr. Levinstein, said the day had been one of great enjoyment; it had been an intellectual day. It was most interesting to have seen what was in progress in these large and extensive works.

Mr. Thos. Tyrer said he had never heard the case for British manufacturers, and why they were a step or two behind, put so well as it had been by Mr. Levinstein. There were schools admirably fitted in the country, but few schools in which quite a complete chemical training was given, including physics. Those things were wanted if they were to cope with foreign competition, and meet future needs. He regarded it as a thousand pities that Hofmann was ever allowed to leave England. We were mean in the extreme to let him go. If he had stayed our patent laws would not have been what they were to-day. In conclusion he proposed the health of Dr. Schunck and Sir Thos. Wardle.

Dr. Schunck said he hoped to attend many meetings of the Society, but he was afraid, at his age, the probabilities were against it.

Sir Thomas Wardle said that we could only export two millions sterling of silk from this country, while we had to import 20 millions from the Continent. Manchester was the only place in which their silk students could get a proper curriculum, and without more of such institutions as the technical school, the industry would not be restored to them. Collateral chemical industries would flourish if only the silk manufacture were restored.

Mr. Levinstein proposed "The Manchester Corporation."

Mr. Alderman Hoy, in responding, said the new technical school the Corporation was building was large—some thought too large, but he was convinced that it was not more than adequate to the present and prospective needs of that great industrial centre. In regard to the remarks of Sir Thos. Wardle, while they had not been able to give him all the encouragement he wanted, they yet had done much to meet his views.

Prof. W. H. Perkin, F.R.S., whose health was toasted, said he was grateful for the remarks made about his father. It was to be hoped that the coal-tar industry would come back to this country. It was perfectly useless for a man to send his son for three years as a student in chemistry. The least period was five years. The money laid out on a student for six or seven years would be well repaid. That he would ask the company to bear in mind. In that time a student would have a good grasp of the subject, and in the works the advantage to him and to the manufacturer would be enormous.

Mr. Levinstein proposed the health of Mr. Tyrer, who, he said, had given more time to the establishment and development of the Society than any other member. Also he proposed the health of Mr. G. E. Davis, who had done great service.

Mr. Tyrer and Mr. Davis responded. Mr. A. H. Allen made a humorous reply to the toast of his health, and Mr. Carter Bell also had response to make for his energetic services.

Mr. Councillor Frankenburg's Garden Party.

In the afternoon a large company assembled at Overbrook, Kersal, and were received by Mr. and Mrs. Frankenburg. In the grounds and also in the drawing-room, refreshments were provided for the visitors, and in a marquee an excellent band performed a selection of music. This gathering, aided by the continued splendid weather, formed a most happy conclusion to the week's proceedings, and enabled all, not only to compare notes on the events of the week, but afforded an opportunity for a general leave-taking amid most pleasant scenery and surroundings.

Journal and Patent* Literature.

* Any of these specifications may be obtained by post by remitting 8d.—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

I.—PLANT, APPARATUS, AND MACHINERY.

PATENTS.

Evaporating Solutions [Brine, &c.] with Multiple Effect, Impts. in and Apparatus for. T. Glover and J. Brock, both of Exchange Buildings, Liverpool. Eng. Pat. 14,952, July 6, 1896.

The first part of the apparatus comprises a flat-bottomed, covered pan, in which the liquid is heated by a fire, or by a steam coil, the steam from this vessel being employed to evaporate the liquid in the second part of the apparatus. The latter consists of three superposed chambers, the uppermost and lowest of which are connected by a series of pipes passing through the middle chamber. The liquid is contained in the uppermost and lowest chambers and in the pipes, and the steam from the first apparatus is admitted into the middle chamber around the pipes. Currents of air are passed upwards through the pipes from the lowest chamber, to obtain the effect of ebullition without raising the liquid to the boiling point. The third part of the apparatus consists of a vessel or a series of vessels containing the liquid, and each provided with a coil of pipes, through which the air and uncondensed steam from the second apparatus are passed. The primary evaporation takes place in the third apparatus, and the final evaporation in the first apparatus, the liquid and steam being passed through the multiple effect in opposite directions. The apparatus is provided with suitable agitators, and with scrapers and elevators for the removal of the deposited salts.—R. A.

Evaporating Apparatus [for Glycerin, Salts, &c.], Impts. in and relating to. J. Foster, Pollokshields, Glasgow. Eng. Pat. 17,053, August 1, 1896.

The vapour chamber of a vacuum evaporating apparatus is provided with a number of hinged or movable plates, each of which is provided on its under surface with a series of perforated baffle plates adapted to trap and return any glycerin, &c. which may be carried off with the vapours. The inclination of the hinged plates can be adjusted to any desired angle, or they can be turned into a vertical position to facilitate cleaning, &c. When salts are deposited by the evaporation, a hopper chamber and valves are provided at the lower end of the evaporator, with a trapping chamber and pipe connection to the vapour chamber, to permit the removal of the salts without admitting air to the evaporator. —R. A.

Distilling, Evaporating, or Concentrating Liquids [Sugar Solutions, Fats, Oils, Acids, Saline Solutions, &c.], Improved Process and Apparatus for. W. P. Thompson, 322, High Holborn, London. From A. Testelin, Brussels. Eng. Pat. 7108, March 18, 1897.

The process described by the patentee provides for the production of a maximum of vapour (as it appears at the commencement of violent ebullition) in the previously heated liquid, by bringing the liquid by mechanical means into the state of spray (with or without the application of steam) before or after its entrance into the evaporation, distillation, or concentration space, so that the escape of the steam bubbles or of the easily volatilised constituents of the liquid takes place upon a surface increased to its maximum by the division into spray. In the apparatus described, the heat of the escaping vapours is utilised for the preliminary heating of the liquid before its entrance into the spraying apparatus, and the spraying is effected by means of an injector. Steam can also be admitted for heating the liquid, either at the commencement of, or during, the spraying process, as may be required.—R. A.

Cooling, Concentrating, Evaporating, and Graduating Liquids; Impts. in Apparatus for. J. Klein, Frankenthal, German Empire. Eng. Pat. 9941, April 21, 1897.

The apparatus is of the kind in which the liquid is passed downward through or over successive layers or bundles of brushwood, &c., the bundles in the lower layers being made of greater length than those in the upper layers, to prevent

liquid from dropping from the ends of the upper bundles directly into the receiver. The bundles are also arranged to break pitch vertically, the supporting rods for the bundles being arranged in the form of trellis-work.—R. A.

Cooling Liquids [Water, &c.], Impts. in and connected with Apparatus for. J. Klein, Frankenthal, German Empire. Eng. Pat. 10,025, April 22, 1897.

Improvements are described in the specification of Eng. Pat. 11,246 of 1890 (this Journal, 1890, 1024), in which the liquid is run downward between ranges of plates set at different angles, and is cooled by a current of air passed upward between the plates. In the present invention the whole or part of the ranges of cooling plates are arranged within a pit, to reduce the lift necessary for the liquid to be cooled, the plates within the pit being enclosed, except at the bottom, by open or close boarding or walling. Each lower range of plates is made of greater length than the range above, and the projecting wings are covered with hinged pent-roofs. The plates are constructed in grid form, the lowest member of the grid being notched beneath, so that the edges formed by the notches conduct the liquid directly on to the edges of the plates beneath. The top edge of the grid is provided with a narrow notched trough, through which the liquid is delivered on to the plates. The cooling plates are surmounted by a chimney-like structure adapted to induce a current of air through the apparatus.—R. A.

Vapour Condensers, and Apparatus for Destroying Noxious Fumes, Recovering By-Products, from Distillation, and like Purposes; Impts. in. S. G. Merrick, Philadelphia, and H. L. Washburn, U.S.A. Eng. Pat. 12,572, June 8, 1896.

The apparatus consists of a chamber divided into two compartments by a partition, which, however, does not reach down to the floor of the chamber. Suitably suspended in the chamber is a rotary conveyor consisting of a frusto-conical casing having spiral vanes which extend from the axis to the sides of the casing, the smaller end of the casing working in an opening or stuffing box in the partition.

Water or other solution is placed in the chamber to just above the level of the axis or shaft, so that on rapid rotation of the conveyor, fumes or gases in one compartment are drawn into the casing and forced along with the water into the other compartment, whence they escape.

A modification is also described, in which the vanes are replaced by a spiral pipe.—O. H.

Condensing, Absorbing, Heating, Distilling, and otherwise Treating Gases [Benzol, &c.] and Liquids; An Improved Column for. H. Hirzel, Leipzig-Plagwitz, Germany. Eng. Pat. 2900, Feb. 3, 1897.

The column comprises a series of superposed chambers or dishes, each provided with an arrangement of pipes adapted to receive or circulate the cooling or warming gases or liquids, the pipes in each chamber being either in communication with those of adjacent chambers or forming a separate series for each chamber, and in conjunction therewith a direct steam inlet arranged below the lowest dish of the column, to serve for the admission of steam for distilling, &c. purposes.—R. A.

Smoke or Noxious Gases or Vapours, An Improved Respirator or Protective Device for Preventing the Inhalation of. C. A. A. Miller, Syracuse, New York, U.S.A. Eng. Pat. 5512, March 2, 1897.

The respirator consists of a mask of non-combustible material in a suitable frame, and provided in the front with a sighthole protected with mica, &c., and with a perforated cup or holder to receive a sponge, &c., through which respiration is effected. The mask has a gathered or elastic backing, to enable it to be fitted closely to the head, and may be secured in position by a strap at the back.—R. A.

Reducing the Pressure of Liquids and Gases, Improved Apparatus for. R. Matchett, Melbourne. Eng. Pat. 4762, Feb. 22, 1897.

A flexible diaphragm of rubber, &c., is employed. It controls a valve on the inlet pipe of the apparatus. Spiral springs and plates bearing against each side of the diaphragm enable the pressure to be reduced or adjusted as may be desired. The apparatus is also fitted with a safety-valve and pressure-gauge.—O. H.

Drying Moist Materials, A Method of and Apparatus for. E. Stauber, Berlin, Germany. Eng. Pat. 9665, May 6, 1896.

The moist material—peat, for example—is first pressed, and then fed into a revolving drum fitted with spiral vanes on the inside. Through this revolver pass the direct products of combustion from a steam-boiler furnace. After passing through this apparatus the material is lifted by an elevator (and, during its passage through the open air, cooled) into a steam drying apparatus, heated by means of superheated steam generated and superheated in the boiler and revolver. The material is carried through the apparatus on travelling bands and laths.—O. H.

Filtering Apparatus, Impts. in. S. K. Welch, London. Eng. Pat. 11,633, March 28, 1896.

A frame or table is employed, on which is stretched perforated metal or wirework, and beneath which is a shallow tank connected with an air pump. An endless band of filtering material, suitably carried on rollers, rests on the perforated metal work, so that on lowering a frame of about the same dimensions as that on which the metal sheet is stretched, a shallow tank is formed with a porous bottom. Into this tank passes the liquid to be filtered, the filtration being expedited by the air pump. When the pores of the cloth become choked, a fresh portion of the band is used, the previously used and fouled portion being in the meantime cleaned and freed from the deposit.—O. H.

Gas [Oxygen, Hydrogen, &c.] or Gaseous Fluids under Pressure, Impts. in Cylinders for Storing. W. Lawson, Newton-le-Willows, Lancaster, and T. Prescott, Sale, Chester. Eng. Pat. 13,331, June 17, 1896.

Cylinders for storing and conveying compressed gases and the like are constructed of corrugated metal, fluted either longitudinally, diagonally, or with transverse corrugations, and are provided—in addition to the ordinary inlet and outlet valve—with a regulating or safety valve, whereby any excessive pressure sufficient to cause an extension of the cylinder is automatically released. The exterior surfaces of the corrugations may be protected by india-rubber or other flexible coverings.—R. A.

Cooling Water and other Liquids, Impts. in Apparatus for. D. Halpin, Victoria Street, London, J. B. Alliott, Bloomsgrove Works, Nottingham, and L. Sterne, Victoria Street, Westminster, London. Eng. Pat. 11,321, May 23, 1896.

The liquid to be cooled is made to issue under pressure in the form of finely divided jets from ejection nozzles, which are arranged in conjunction with induction nozzles at the bottom, &c. of an open tower or chimney, &c., so as to cause induced currents of cold or cooler air to be brought intimately into contact with jets of liquid, to take up the heat therefrom.—R. A.

Still-worm Pipes, and Supports for the same, Impts. in. Edwards and Co., London. From L. Rohrmann, Krauschwitz, Germany. Eng. Pat. 1658, Jan. 21, 1897.

The inventor claims "the combination, with helical earthenware still-worm pipes, of short supports in which the separate coils of the pipes rest, the supports having double feet and being entirely separate from each other, and the upper part of each support being separate from the lower part.—V. C.

II.—FUEL, GAS, AND LIGHT.

Benzene, and its Importance as an Illuminating Agent. G. Kraemer. Chem. Zeit. Rep. 1897, **21**, 136.

The coal tar from the gasworks in Germany is said to be the source of about three-eighths of the total consumption of benzene in that country, the remainder being imported from abroad—chiefly England. Much attention has been given within recent years to the production of coke in closed ovens, provided with means for recovering the by-products, the benzene contained in the gas being extracted by absorbing it in heavy tar oils, from which it is subsequently liberated by distillation. At the beginning of last year the quantity of benzene thus produced was put down at 4,500 to 5,000 tons per annum, which is said to represent three times the amount obtainable from the tar; so that, as regards their supplies of this material, the German colour works will soon be quite independent of foreign sources. The rapid advance in the price of benzene last year gave rise to the reconstruction of a large number of coke ovens, and it is estimated that when these, and others for which plans have been prepared, are started, at least 80,000 tons of benzene will be produced. As this quantity cannot possibly be consumed, even if, in addition to the colour industry, it were used for increasing the illuminating power of gas, the author suggests that it should be employed as a direct lighting agent. Trials made with xylene in an incandescent spirit lamp have given the following results:—

	Candles.
95 per cent. alcohol	6,100
Mixture of 15 per cent. xylene and 85 per cent. alcohol	6,700
" 25 " " 75 " "	7,200
" 33⅓ " " 66⅔ " "	8,226

For incandescent lamps the first proportion should not be exceeded, otherwise the mantle is blackened. Commercial 90 per cent. benzene gives equally good results. With regard to the illuminating effect, it is shown that for every 100 parts of alcohol *per se*, only 81·4 parts of mixture are required when adding 10 per cent. of xylene, and 73·2 parts when 15 per cent. is added. The latter represents a saving of 26·8 per cent., assuming the price of the mixture to be the same as that of denatured alcohol. In case of need a mixture of 66⅔ per cent. of alcohol and 33⅓ per cent. of benzene can be used in ordinary petroleum lamps in the place of petroleum.—D. B.

Gas-Igniting Appliances, Automatic. H. Drehschmidt. J. für Gasbeleucht. 1897, **40**, 297—301.

Electrical appliances for igniting gas automatically are either complicated or cumbersome. Platinum sponge glows in a stream of hydrogen and air, and effects ignition. The hydrogen being much diluted in coal-gas, the latter cannot be thus ignited. The sponge, however, becomes sufficiently heated to warm a platinum wire embedded in it, so that the latter is made incandescent by the gas, and ignites it. Platinum sponge soon loses its activity, and Duke substituted for it, platinised meerschaum or other porous material, with good results. A pellet of this material with a protruding platinum wire, if suitably fixed with regard to the slit of a flat-flame burner, will ignite the gas whenever the gas-tap is opened, and will retain its efficiency for a long period, provided the wire is not displaced in cleaning.

This device is less readily applied to incandescent burners, as, if suspended above the mantle, it becomes spoilt by the hot gases. S. Elster placed the pellet above a special jet at the side of the burner, and by careful manipulation of a two-way cock he was able to first ignite the flash-jet, to ignite therefrom the principal flame, and then to extinguish the flash-jet. The intelligent manipulation of the gas-cock, which was essential to its proper working, rendered this device valueless for general use. Canellopoulos extinguished the flash-jet automatically through the expansion of a solid, a liquid, or a gas, by the heat of the principal flame. In the first application of his patent, the expansion of gas, confined in a sealed limb of a U-tube, forced mercury from the bend of the tube up the other limb, which terminated in the flash-jet. The mercury sealed the gas inlet to this limb, and thus extinguished the jet very soon after the ignition of the principal flame. This arrangement was faulty, in that the gas flowed simultaneously to the jet and the burner, and collected in the chimney before the jet was lighted by the pellet; and thus the principal flame was ignited with an explosion which damaged the mantle. The expansion of platinum wire was then made to actuate a two-way valve on the gas supply. The normal position of the valve left the way to the flash-jet only open. On opening the gas-tap, gas therefore flowed to the latter and was ignited by the platinised pellet. The heat of the flash-flame expanded a platinum wire which supported a lever attached to the valve-plug, and its expansion partially opened the way to the burner. The principal flame ignited from the flash-jet and further expanded the platinum wire, thereby closing the way to the jet and extinguishing it. When the burner was extinguished, the contraction of the wire automatically brought the valve to its original position. A failure on the part of the pellet to ignite the gas is not dangerous, as only the small quantity of gas for the supply of the flash-jet can escape to the air.—J. A. B.

Sulphur and Hydrogen, Conditions of Direct Combination of. H. Pélabon. Comptes Rend. 1897, 686.

See under VII., *page* 610.

Illuminating Gas, Determination of Oxygen in. O. Pfeiffer. J. für Gasbeleucht. 1897, **40**, 354.

See under XXIII., *page* 635.

PATENTS.

Ovens for Drying and Burning Tiles, Pottery, and other Plastic or Semi-dry Ware; Impts. in. R. Stanley, Nuneaton, Cheshire. Eng. Pat. 7899, April 14, 1896.

An oven or kiln, having a chamber underneath the floor in direct communication with the fire-holes, and openings leading from the chamber through the floor into vertical flues. The floor of the oven is carried upon fire-bricks arranged in such a way that the fire from the fire-holes may collect and combine. A casing is employed between the walls of the oven and the goods to be burnt, forming a flue or flues down which the fire, after having passed up the vertical flues, may descend and be led off to an outside chimney.—V. C.

Metallic Threads, Refractory [Incandescent Gas-burners]; Impts. in or relating to the Manufacture of. M. L. Ross, London. From E. Oberlé, Paris. Eng. Pat. 12,056, June 2, 1896.

This specification refers to the preparation of oxide threads for incandescent gas-burners. A 50 per cent. solution of thorium oxychloride ($ThOCl_2$) in alcohol is mixed with a 3—20 per cent. solution of nitrocellulose in alcohol-ether if the thread is to be spun under water, or with a 20 per cent. solution if it is to be spun under pressure in the air. The oxychloride may be spun when mixed with one-fourth part of nitrocellulose. The proportion of alcohol must exceed that of ether in a homogeneous solution, because ether precipitates ThO_2 or a basic salt. The spinning is effected with either a weak or a strong solution. In the former case the solution is passed through glass tubes with capillary openings immersed in water, the solution being drawn by clockwork or otherwise through the water, in which the alcohol and ether dissolve; the thread is then dried in the air. In the latter case, the solution, consisting of 20 per cent. of nitrocellulose and 80 per cent. of thorium oxychloride, $ThOCl_2$, with 0·75 of cerium oxychloride, dissolved in ether and alcohol, is passed under pressure into the open air. The threads are finally decomposed by heating in a closed vessel or in the open air at a low temperature, whereby the nitrocellulose is burned and the salts are broken up into oxyhydrates, which are then converted into oxides by means of a Bunsen burner. The threads may be denitrated by means of ammonium hydrosulphide if preferred. By using a salt that may be metallised, in place of thorium compounds, threads for incandescent electric lamp filaments may be made in the same manner.—W. G. M.

Incandescing Media for Lighting Purposes, Impts. in the Manufacture of. R. Langhans, Berlin. Eng. Pat. 14,845, July 4, 1896.

The claims are for the manufacture of incandescent media composed of zirconia with about 1 per cent. of vanadium pentoxide. Mantles may be made in the ordinary way by impregnating a fabric with an aqueous solution of zirconium and vanadium salts, and then burning off; or a zirconia body may be first made (say according to Eng. Pat. 22,396 of 1893), then impregnated with an aqueous vanadium solution and calcined. A paste may be made by impregnating zirconia with an aqueous vanadium solution, and the bodies made from this paste are then dried and calcined in a Bunsen flame.—H. B.

Filaments for Use in Electric Glow Lamps, Impts. in. [Impregnation with Rare Earth Oxides.] F. de Mare, Paris, France. Eng. Pat. 16,534, July 25, 1896.

The improved filament is formed of a vegetable fibre or thread impregnated with an aqueous solution of an organic salt of one or more of the rare earths, the object being to obtain a mixture of carbon and the oxide of the rare earth, the latter acting as the irradiating substance, and the former as the conductor of electricity. The solution preferably employed is one of the citrates obtained by precipitating with ammonium citrate, a solution composed of thorium nitrate and cerium nitrate, the proportions being 99 per cent. of the oxide of the former, and 1 per cent. of the oxide of the latter. The carbonised filament is soaked a second time in the solution, and then dried and annealed electrically. Instead of the soaking, a mixing process may be employed, in which the organic salt is mixed with the vegetable substance before it is drawn into a filament, the latter being then soaked several times in the solution of the salt, and annealed as before.—G. H. R.

Incandescence Bodies for Illuminating Purposes, Impts. in the Manufacture of. W. Nicholls, London. Eng. Pat. 16,966, July 30, 1896.

Mantles are to be prepared in the ordinary way by impregnating a fabric with a solution prepared as follows:—"2 grms. of palladium chloride dissolved in 100 grms. of water and 3 grms. of platinum chloride dissolved in 100 grms. of water are each mixed with 25 grms. of tartar emetic, and 25 drops of each or 50 drops of their mixture are added to a solution consisting of" 12·5 grms. of zirconium nitrate, 6·25 grms. of calcium nitrate, and 8·5 grms. of ammonium nitrate. Other suitable soluble salts of zirconium, calcium, and antimony may be used. It is stated that a mantle so prepared, if placed on a Bunsen burner, will ignite the gas when the latter is turned on. The claims are for "a self-igniting incandescence body" and for the above-described impregnating solution.—H. B.

Incandescent Light produced from Benzine Gas. An Apparatus for Producing and Using an. P. W. von Gehlen, Prussia. Eng. Pat. 2194, Jan. 27, 1897.

The single claim is for "an arrangement for the production of benzine gas incandescent light, characterised by the fact that compressed air is forced over the surface of benzine, and becoming impregnated with hydrocarbon thereby, flows to an incandescent gaslight burner of which the annular cone surrounding the nozzle is solid or imperforate." The "annular cone surrounding the nozzle" is not perforated with air inlets, because the gaseous mixture contains sufficient air for combustion.—H. B.

[Mantles.] Incandescent Gas Burners, Impts. in. D. Meyer, Paris. Eng. Pat. 8759, April 6, 1897.

The improved burner consists of incandescence material, which is formed into beads or particles of any suitable shape, these being strung upon a double wire and arranged in network form over a Bunsen burner, so as to form a mantle. The air inlets of the burner are much larger than usual, and are fitted with wire gauze.—H. B.

Bunsen Burners and Incandescent Gas and Similar Lamps in which such Burners are Incorporated, Impts. relating to. A. H. Catherwood, Bermondsey. Eng. Pat. 11,106, May 21, 1896.

The combination illustrated and claimed, is that of an incandescence lamp of the usual type, with a globe surrounding the chimney, the globe resting upon a gallery which forms the upper part of an imperforate casing that surrounds the burner and passes down to a point below the air-inlet holes, where it is fastened by an air-tight joint to the burner tube. Thus the air supply—both that portion which enters by the air-inlet holes and that which passes in at the bottom of the chimney—must all pass down between the globe and the chimney, receiving a preliminary heating, and being unaffected by draughts. Another claim is for a device consisting of a tubular piece of glass filling up the space between the "lighting-back" plate and the lower edge of the chimney; in this way no air can enter at that point, and the entire air supply must enter through the air-inlet holes.—H. B.

Incandescence Spirit Lamps, Impts. in. E. Becker, Berlin. Eng. Pat. 10,710, May 18, 1896.

The improvements relate to lamps of the type in which a small heating flame from a central wick, vaporises the spirit which is drawn upwards from the reservoir by two or more main wicks, the tops of the latter being in the gasifying chamber. The improvements are embodied in the claims, which are for a spirit incandescence lamp in which the heating gases of the vaporising flame pass through the Bunsen burner by means of a central pipe serving as a draught cylinder and passing through the gasifying chamber, and are led by a lateral branch pipe up into the chimney surrounding the mantle burner; the objects being to avoid smell, to heat the mixture of gas and air, to keep external parts cool, and to give a steady flame when the lamp is carried about.—H. B.

Acetylene, Treating Prior to Combustion; Impts. in and connected with the Method and Apparatus for. T. Thorp, Whitefield, near Manchester, and T. G. Marsh, Manchester. Eng. Pat. 12,942, June 12, 1896.

The invention refers to methods of and apparatus for diluting acetylene with air or other gases for the supply of ordinary gas burners at low pressure. Acetylene at a pressure of about 6 ins. of water, feeds an injector which draws in the diluent, and the mixture is stored in a low-pressure holder, the bell of which actuates a tumbler arrangement which opens or closes the valve on the injector inlet pipe as required. Or the acetylene drives a motor—preferably of gas-meter type—which pumps the diluent along with the exhaust acetylene into a mixing chamber.
—J. A. B.

Acetylene Gas, Impts. in Automatic Apparatus for the Manufacture of. J. E. Atkinson and J. M. Labouchere, both of Liverpool. Eng. Pat. 13,147, June 15, 1896.

Two generators, automatically acting in succession, are supplied with water from a reservoir through a non-return valve. A gasometer and safety devices are provided. In another pattern the carbide is placed in a receptacle within the gasometer, which has a dished top, with door and seal for the introduction of fresh carbide. In a portable pattern, the bell of the gasometer is weighted so that it does not rise.—J. A. B.

Acetylene Gas, Impts. in Apparatus for Generating, Storing, and Cooling. F. S. Thorn and C. Hoddle, both of London. Eng. Pat. 15,962, July 18, 1896.

Close cylindrical carbide containers with external mouthpieces are situated at the foot of the tank of a gasholder. The fall of the gasholder bell opens a ball cock, and permits water to enter one compartment of one container. As each compartment is exhausted, the water flows on to the next. When one container is exhausted, another ball cock operates and admits water to the next container.—J. A. B.

Acetylene, Apparatus for Generating and Storing. G. de Roussy de Sales, Lyons, France. Eng Pat. 2292, Jan. 28, 1897.

A close vessel contains a fixed bell with contracted upper part. The water in the bell acts, until its level is depressed by the gas produced, on carbide introduced in cartridges into projecting receivers, which can be shut off from the bell by valves. The gas passes through a non-return valve and washer to an elastic bag, and thence to the burner. Safety devices are provided.—J. A. B.

Acetylene Gas, Impts. in Means for Generating, Holding, and Burning. Don Carlos B. y Franco, San Gervasio, near Barcelona. Eng. Pat. 5236, Feb. 26, 1897 (under International Convention).

The invention comprises a fixed or portable gasogene consisting of two connected reservoirs for water, the lower of which contains a carbide chamber, to which the water gains access by means of wicks of any porous material.
—J. A. B.

Acetylene Gas, An Improved Apparatus for the Manufacture of. H. E. Alexandre, Barcelona, Spain. Eng. Pat. 5913, March 5, 1897.

The apparatus consists of a generator with vertically separated compartments which come into action successively; a bell gasholder with rack which operates the water-supply tap to the generator; a gas-inlet pipe sealed in the water of the gasholder tank, so that the passing gas is washed; and a signal device giving notice of the exhaustion of the carbide.—J. A. B.

Acetylene, Impts. in Apparatus for Manufacturing. H. Cousin, Paris. Eng. Pat. 8552, April 3, 1897.

A stationary hopper containing carbide is supported above a gasholder bell which floats in an annular tank. To the bottom of the hopper is attached a small bell which dips in an annular tank riveted to the crown of the gasholder bell. The valve at the bottom of the hopper is kept closed by means of springs, until the weight of the gasholder bell in its lowest position is thrown upon it, when it is opened and carbide drops through into water at the base of the apparatus. The gas is purified in four compartments, containing, in order, sulphate of copper, oxide of iron, carbide of calcium, and chloride of calcium.—J. A. B.

Acetylene Gas, Impts. in Apparatus for Developing. W. P. Thompson, Liverpool. From The Deutsche Acetylengas Gesellschaft mit beschränkter Haftung, Berlin. Eng. Pat. 10,199, April 23, 1897.

A gasholder bell supporting a carbide container moves in the annular space between two concentric tanks, the inner of which contains water for the decomposition of the carbide. An air lock for admission of carbide is provided.—J. A. B.

Acetylene Gas, Impts. in Apparatus for Generating. D. C. Moreney, Levis, Canada. Eng. Pat. 10,186, April 23, 1897.

The gas is generated by water admitted to carbide through the top of a vessel of bell gasholder pattern. It escapes to a bell gasholder, in the crown of which is a subsidiary holder, the bell of which operates its inlet valve. Burners are supplied from this subsidiary holder. Depression of the level of the liquid in the main holder cuts off the supply of water to the generator.—J. A. B.

Carbides of the Earth Metals and Alkali Earth Metals, Impts. in the Manufacture of. L. M. Bullier, Paris, France. Eng. Pat. 16,255, July 22, 1896.

In the improved process for the commercial manufacture of carbide of calcium, or of any of the carbides of the earth metals and alkali earth metals, the temperature at which fusion takes place is lowered by the addition of some suitable flux, such as 10 per cent. fluorspar, fluoride of aluminium, cryolite, or other fluxes, such as alumina, silicates, &c., according to the nature and purity of the carbide required. The process may be carried out by the method described in Eng. Pat. 2820, Feb. 8, 1895 (this Journal, 1896, 265), or by any other method, whether electrical or otherwise.—G. H. R.

Treatment of Gas for Illuminating and other Purposes, Impts. in and relating to the. A. Arter, Hammersmith. Eng. Pat. 12,130, June 6, 1896.

The gas is dried and expanded before passing to the burners. This is done by passing it through a vessel heated by one or more ordinary gas burners.—R. S.

Removal of Sulphuretted Hydrogen from Gases or Gaseous Mixtures, An Improved Process and Apparatus for the. H. Strache, Vienna. Eng. Pat. 14,943, July 6, 1896.

The sulphuretted hydrogen is removed by means of nitric acid, nitrous acid, or oxides of nitrogen, with or without the addition of air or oxygen. The best plan, it is stated, is to have three vessels arranged at different levels. The gas to be treated, mixed with air, is led into the lower vessel, in which it is treated with dilute nitric or nitrous acid, which is the mixture which runs away from the third vessel. It then passes to the second vessel, in which it is treated with concentrated nitric acid, and is finally washed with water in the third or uppermost vessel. The sulphuretted hydrogen is recovered as sulphur from the vessels and discharged liquid.—R. S.

Manufacture of Gas, and Apparatus therefor; Impts. in the. W. L. Wise, London. From Prof. E. Thomson, Swampscott, U.S.A. Eng. Pat. 16,692, July 28, 1896.

To manufacture a combustible gas consisting mainly of carbon monoxide, hydrogen and nitrogen, a vertical, lined gas producer, having a vertical central division rising some distance from the base, is employed; or two such chambers connected together near their upper ends may be used. Instead of the usual bricks employed as regenerative material, or in addition thereto, the ashes of the fuel used in the chambers are employed. Air is passed through this heated incombustible inert material, thence upwards through a mass of combustible fuel, over the top of which water is discharged. The gases produced are then drawn downwards in the second portion of the chamber (or the second one when two are used), through the incandescent fuel and ashes therein, thereby abstracting the heat therefrom; and finally the gaseous mixture is collected, or used at once in a gas engine. The direction of inflow of air and outflow of gas is reversed from time to time, either automatically or by hand. The two forms of producer are illustrated, showing suitable valve mechanism, and, in the case where two chambers are employed, the connections with the gas engine to be driven, and the mechanism for operating the reversing valves automatically from that engine.—R. S.

Condensing, Absorbing, Heating, Distilling, and otherwise Treating Gases [Benzol Vapours, &c.] and Liquids An Improved Column for. H. Hirzel, Leipzig-Plagwitz Germany. Eng. Pat. 2900, Feb. 3, 1897.

See under I., page 602.

Producer- and Water-Gas, Improved Process and Apparatus for Manufacturing. C. Whitfield, Kettering. Eng. Pat. 12,771, June 10, 1896.

Producer gas having the smallest percentage of nitrogen, and a high percentage of carbonic oxide, hydrogen, and of the lighter hydrocarbons, is obtained by raising the fuel at the bottom of a generator or producer to a high temperature by forcing in air, and by injecting into the incandescent fuel, at a point above the combustion zone, steam and the volatile compounds given off from the green fuel at the upper part of the generator. The products of combustion of the fuel and of the decomposition of the steam and the volatile products, are withdrawn from the producer at a point—say 2 or 3 ft.—below the top of the generator and below the point where the volatile compounds are given off from the green fuel. The latter are withdrawn through a suitable connection pipe, and are passed into the column at another point along with the steam of the steam injector.
—R. S.

Gas Furnaces, Impts. in. T. Fletcher, and Fletcher, Russell, and Co., Ltd., Warrington. Eng. Pat. 7067, March 18, 1897.

The gaseous fuel employed, is composed of air under pressure mixed with a light hydrocarbon, such as gasoline or benzoline. For an oxidising flame, the air is not fully saturated with hydrocarbon; for a reducing flame, it is supersaturated. The mixture is caused to come into direct contact with the material to be heated, and is injected at a tangent to the inner diameter of a ring or cylinder enclosing the articles to be heated.—R. S.

Enriching Gas, An Improved Composition and Apparatus for. A. A. Stephenson, Sydney, New South Wales. Eng. Pat. 7991, March 29, 1897.

The composition consists of ¼ of a pint of oil of cloves, 2 pints of "best ether," 2 pints of petroleum, 2 pints of gasoline, and 2 pints of kerosene, mixed with the following dry substances: ¼ lb. of powdered camphor, ¼ lb. of powdered resin, ¼ lb. of powdered whiting, and ¼ lb. of powdered glue. The enriching apparatus, which is charged with the above composition, and through which the gas is passed, consists of a box having inlet and outlet unions, a charging hole, and, in its interior, rows of wires for supporting canvas, wicks, or other similar absorbent material.—R. S.

Lighting and Heating by Means of Hydrocarbons, Apparatus for. S. Pitt, Sutton, Surrey. From La Compagnie Internationale des Procédés Adelphe Seigle, Paris. Eng. Pat. 8177, March 30, 1897.

The apparatus, which is specially applicable to heating steam boilers by the use of light or heavy liquid hydrocarbons, consists, 1st, of a retort in which the hydrocarbons are gasified; 2nd, a sprayer (optional); and 3rd, a permanent lighter placed between the jet and the furnace to be heated. The retort is a cylindrical vertical column, through the hollow walls of which, or through a helical tube arranged inside the wall, the hydrocarbons are passed from above downwards, and provided with a lamp in its lowest part, which at first is fed with an easily inflammable light hydrocarbon, but afterwards solely by some of the vapour produced. The sprayer is omitted when using light hydrocarbons. The lighter is a casing having an inner metallic or refractory wall, which slightly pinches the gas, while its axis is coincident with that of the flame.—R. S.

Furnaces for Use with Light Hydrocarbons as Fuel, Impts. in. T. Fletcher, and Fletcher, Russell, and Co., Ltd., Warrington. Eng. Pat. 9042, April 9, 1897.

The furnace, to be used instead of a muffle or the like, is circular in plan, with any suitable centrally perforated cover. In the centre of the furnace bottom there is a movable or permanent circular wall, or a flat-bottomed, open-topped, cylindrical container, leaving an annular passage between it and the circular wall. Into this annular passage the benzoline or gasoline flame is injected, through an opening in the furnace casing at a tangent thereto. The work—such as enamels and dentists' work—is placed in the inner container or space.—R. S.

A Photometer for Automatically Measuring and Registering the Intensity of the Light of a Gas Flame, and Apparatus therefor. W. and B. Cowan, Ltd., Westminster. From C. V. L. Martenson, Rio de Janeiro. Eng. Pat. 9041, April 9, 1897.

See under XXIII., *page* 634.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Petroleum, Caucasian; New Constituents of. W. Markownikoff. Ber. **30**, [8], 974—977.

Contrary to his previously published opinions (J. Russ. Chem. Soc. 1890, 23), that the fractions boiling below 60° C. consist exclusively of paraffins, the author now finds that in some of these the specific gravities are higher than is consistent with this assumption, and indicate the presence of cyclic hydrocarbons. This is the case with the fractions boiling between 58° and 48°, and from 87° to 81°, where the sp. gr. rises continuously as the boiling point recedes; and since in the fraction 68°—70°, the sp. gr. is 0·692, whilst that of normal hexane is 0·663, and in the fraction 36°—38° the sp. gr. is 0·643, that of normal pentane being 0·6263, it is assumed that the methylpentamethylene known to be present in the fraction 68°—73° is concentrated in the fraction 71°—75°, the presence of another cyclic hydrocarbon (trimethyltetramethylene or tetramethyltrimethylene) being conjectured in the fractions between 79° and 85°.

In the fraction 50°—51° the sp. gr. (0·751) indicated cyclopentane, the secondary nitro-product obtained from this fraction yielding the characteristic benzoyl derivative (glutaric acid) indicative of pentamethylene. From this and the adipinic acid obtained by nitrating the hexamethylene fractions, it is concluded that the cyclic hydrocarbons having no branch are converted on oxidation into the corresponding dibasic acids.

The fraction 91°—93° (sp. gr. 0·730) was examined and found to contain a branch chain hydrocarbon, C_7H_{14}, yielding a tertiary nitro-product, $C_7H_{13}NO_2$, boiling at 131°—132°, the corresponding tertiary alcohol boiling at 144°—145°. In preparing the latter, an unsaturated hydrocarbon, which can only be C_7H_{12}, was also formed, and yielded, on prolonged heating with hydrogen iodide, a hydrocarbon distilling at 88°—93°, the polymerised products boiling at 94°—130° and 130°—240°. When refractionated, the bulk had a boiling point of 91°—93° and a sp. gr. at 4°—20° of 0·7231, with a vapour density of 3·45, this sp. gr. being lower than that (0·7543) found for synthetic dimethylpentamethylene by Zelinsky and Rudsky.—C. S.

Petroleum, a South American; Composition of. C. F. Mabery and A. S. Kittelberger. Amer. Chem. J. **19**, [5], 374—381.

The sample examined was obtained from the vicinity of the Magdalena River, in Colombia. The crude oil had a sp. gr. of 0·9480 at 20° C., with 0·70 per cent. of sulphur, about 0·320 per cent. of nitrogen, and 0·011 per cent. of ash (containing much iron); the percentages of carbon and hydrogen being 85·45—85·80 and 11·79—12·02 per cent. respectively, and the bromine absorption 12·09 per cent. The colour was dark and the consistency thick and viscous. On distillation under ordinary pressure, the first fractions came over at 260°, and the highest (those distilling between 310° and 345°) were badly cracked, as evidenced by the high bromine absorption (34·96 per cent.), thus differing from North American oils. *In vacuo* the first fraction passed over at 100° C., the largest individual fractions above that temperature being those at 140°—150° and 150°—155°. On repeated distillation, under atmospheric pressure, of the fractions up to 150°, the fraction collected at 170°—172° yielded a hydrocarbon approximating to the formula $C_{10}H_{20}$, and that at 190°—192° a hydrocarbon corresponding to $C_{11}H_{22}$ (sp. gr. 0·8381) or $C_{12}H_{24}$. The next distillate of importance came over at 212°—214°: formula $C_{12}H_{24}$, sp. gr. 0·8483; and as the sp. gr. of the oil after treatment gave 0·8484—the same value as the crude distillate—it appears that this petroleum consists mainly of a single series of hydrocarbons (only a trace of aromatic hydrocarbons being present): the C_nH_{2n} series, similar to the naphthenes in Russian petroleum, which is the first instance noticed of such a constitution in American oils.—C. S.

PATENTS.

Ammoniacal Liquor, A Process for removing Carbonic Acid and Hydrogen Sulphide from. A. Feldmann, Bremen, Germany. Eng. Pat. 10,501, April 27, 1897.

See under VII., *page* 611.

Oil [Mineral], Impts. in the Process of and Apparatus for Deodorising. J. R. Whiting, Connecticut, and W. A. Lawrence, New York. Eng. Pat. 10,526, April 27, 1897.

The process is one for refining and deodorising the lighter products of the distillation of petroleum and coal, such as petroleum ether, gasoline, benzine, naphtha, &c.

The light oil is converted into vapour in a steam-heated boiler, the vapour then being forced by the pressure thus generated through a vertical cylinder packed with bone charcoal. After passing through this, the vapour is led into a cylinder charged with lime water, and is discharged into a condenser, from which it may be collected.—W. P. S.

IV.—COLOURING MATTERS AND DYES.

Dyewood Extracts in Powder. Leipziger Färber- u. Zeugdr. Zeit. **46**, [6], 211.

Sumach, fustic, and Persian berry extracts have recently been put on the American market in powder form. These extracts are said to be very pure, stable, and to effect a saving in carriage in cases where the articles have to be forwarded long distances.—I. S.

o-Nitro-p-phenylene Diamine. C. Bülow and E. Mann. Ber. **30**, 977—988.

By the action of nitrite on nitro-*p*-phenylene diamine, Bülow (Ber. **29**, 2284) observed that only one of the amido groups is attacked, even when a large excess of hydrochloric acid is present, and the second group is only diazotised after the first has been combined with some dyestuff component. In continuing their investigation of the substance, the authors find that with regard to acetylation with acetic acid or benzylation by the Schotten-Baumann method, substitution only takes place in one amido group. Nitrodiacetyl-*p*-phenylene diamine is obtained by nitrating diacetyl-*p*-phenylene diamine in sulphuric acid solution, and one acetyl group can be very readily removed by the action of very dilute alkalis or even ammonia. The nitro-acetyl-*p*-phenylene diamine is a weak base, which, however, gives a stable diazo compound. On combining this with β-naphthol disulphonic acid R and hydrolysing the product, a dyestuff is obtained isomeric with that produced from the non-acetylated base and R salt. Hence the acetyl group, which is not at first attacked, is attached to that amido group which is most easily acted on by nitrous acid, *i.e.*, which has the greatest basicity. By suitably reducing nitro-diacetyl-*p*-phenylene diamine, it is converted into *p*-diacetyltriamidobenzene, which, on treatment with nitrite in an acid solution, yields diacetylamidobenzene-azimide. This substance, even on standing in acetic acid solution at the ordinary temperature, loses the imido acetyl group and is converted into acetylamidobenzene-azimide. The dyestuff obtained by combining the diazo compound of nitro-acetyl-*p*-phenylene diamine with R salt crystallises in long, thin needles and is easily soluble in water. It dyes wool bluish-red from an acid bath. The hydrolysed product has practically the same dyeing properties. When this latter substance is treated with hydrochloric acid and nitrite, it yields a very stable bluish-red diazo compound, which, on combination with R salt, gives the same dyestuff as that obtained from (nitro-*p*-phenylene diamine + R salt) + R salt. As already mentioned, nitro-*p*-phenylenediamine (obtained by hydrolysing the diacetyl compound with concentrated hydrochloric acid) gives a diazo and not a tetrazo compound when treated with nitrous acid. This substance combines with R salt in an alkaline solution, forming greenish crystalline needles, which by reflected light appear blue. The compound dissolves easily in water; alkalis produce a crystalline precipitate, whilst acids change the shade to red. The colour is, however, too dull to be of use in wool-dyeing. The diazo compound has much the same properties as its isomeride, and can be employed for the production of symmetrical or mixed dyestuffs. For instance, nitro-*p*-phenylene diamine + 2 R salt dissolves in sodium carbonate solution with a blue colour, and dyes unmordanted cotton the same shade; but on washing and soaping, this turns violet, and hence is technically of no value, as the salt is dissociated also by a large quantity of water.—T. A. L.

Rhamnazin. A. G. Perkin and H. W. Martin. Proc. Chem. Soc. 1897, [181], 139.

Rhamnazin was isolated from Persian berries by one of the authors and J. Geldard (this Journal, 1895, 565), and shown to be a quercetindimethylether. The present investigation was instituted to determine the position of the methoxyl groups. On methylation, it yielded quercetintetramethylether, and from this result and other experiments described in the paper, it evidently contains no methoxyl group in the phloroglucol nucleus in the ortho-position relatively to the carbonyl group. By fusion with alkali at 200°, rhamnazin yielded phloroglucol and protocatechuic acid, and digestion with boiling alcoholic potash gave vanillin, vanillic acid, and a non-crystalline phloroglucol derivative. Oxidised by air in alkaline solution, vanillic acid and a similar phloroglucol derivative were obtained. No free phloroglucol resulted from either of these decompositions. Taking into consideration that though the dyeing properties of rhamnazin are extremely feeble, it must still be considered a colouring matter, these results indicate that it has the constitution of a rhamnetinmonomethylether.

New Oxyazo Compounds and Triphendioxazine Derivatives. K. Auwers and H. Röhrig. Ber. **30**, 988—998.

Requiring for cryoscopic investigations some *o*-hydroxyazo compounds containing para to the hydroxyl, a strongly negative group, such as NO_2, CN, CO_2R, CHO, the authors have made use of Bamberger's reaction by combining nitrosobenzene with suitable *o*-amidophenols, and have obtained in this way a number of new derivatives. The o-amidophenols employed, were *o*-amido-*p*-nitrophenol, the methyl and ethyl ethers of *m*-amido-*p*-hydroxybenzoic acid, and *o*-amido-*p*-cyanophenol. The method consisted in dissolving equimolecular proportions of the reacting substances in the smallest quantity of glacial acetic acid at a medium temperature, and mixing the solutions, which as a rule turned dark and after a time deposited crystals either as long reddish needles or small plates. The product generally consisted of a mixture of two substances which could be separated by their solubility in acetic acid, the soluble portion being the hydroxyazo compound, whilst that insoluble in acetic acid was insoluble also in other usual solvents, and could only be dissolved by boiling nitrobenzene, aniline, or phenol, &c. These substances formed red or brownish-red plates, having a metallic appearance, and melting above 300° C. A characteristic property is the blue or bluish-violet colour they give with acids, and this is especially marked by dissolving them in sulphuric acid. These compounds are derivatives of triphendioxazine—

$$C_6H_4\left\langle\begin{matrix}N\\O\end{matrix}\right\rangle C_6H_2\left\langle\begin{matrix}O\\N\end{matrix}\right\rangle C_6H_4$$

and, as a matter of fact, by the action of nitrosobenzene on o-amidophenol, a dyestuff has been obtained identical in all respects with the above. Hence nitrosobenzene not only condenses with o-amidophenol, but acts as an oxidiser. The proportion in which the two substances are formed depends upon the particular amidophenol. In the case of the ethers of *m*-amido-*p*-hydroxybenzoic acid, about 50 per cent. of the theoretical amount of crude or 40 per cent. of pure azo compound is obtained, whilst the yield of triphendioxazine dicarboxylic ether amounts to 25 per cent. of the theoretical. The yields in the case of *p*-nitro- and *p*-cyano-o-amidophenol were not so good, and in the latter case the azo compound was not obtained pure. Other new products were also obtained, and are described.—T. A. L.

Amido Amidines. C. Lauth. Comptes Rend. **124**, [20], 1105—1106.

The analogy existing between the amidines—

$$C_6H_4\left\langle\begin{matrix}N\\NH\end{matrix}\right\rangle C.R$$

and the thiazoles—

$$C_6H_4\left\langle\begin{matrix}N\\S\end{matrix}\right\rangle C.R$$

led to the investigation as to whether the former, like the latter, were capable of giving dyestuffs. Hübner and Stoever, by nitrating benzenyl-phenylene amidine—

$$C_6H_4\left\langle\begin{matrix}N\\NH\end{matrix}\right\rangle C.C_6H_5$$

and reducing the nitro product, obtained an amido base, but its constitution was uncertain, since it was not known into which nucleus the nitro group entered. The author has prepared an amido amidine of known constitution by heating 21·4 grms. of o-nitraniline with 32 grms. of p-nitrobenzoyl chloride to 100°—105° C. for six hours. The product obtained, having the constitution—

$$O_2N.C_6H_4.NH.CO.C_6H_4.NO_2,$$

crystallises from toluene in yellow laminæ melting at 216° C. In order to reduce it, 30 grms. were heated with 300 c.c. of hydrochloric acid and 75 grms. of tin for one hour. The tin was then removed with sulphuretted hydrogen, and on evaporation of the filtrate, prisms of the hydrochloride of p-amidobenzenyl-phenylene-amidine were obtained. The base—

$$C_6H_4 \begin{matrix} \diagup N \diagdown \\ \diagdown NH \diagup \end{matrix} C.C_6H_4.NH_2$$

is slightly soluble in boiling water, easily soluble in alcohol, and melts at 240° C. On repeating Hübner and Stoever's experiments, a base was obtained melting at 281° C., and not at 245° C. as stated. The two bases differ, not only in their melting points, but also in their properties. The base (melting point 240° C.) crystallises in long prisms, its alcoholic solution shows a violet dichroism, and its hydrochloride—sparingly soluble in water—crystallises in prisms; whilst Hübner and Stoever's base separates in spherical granules, its alcoholic solution shows a green dichroism, it is much less soluble in water than the isomeric base, and its hydrochloride—very soluble in water—crystallises in laminæ. Both hydrochlorides diazotise easily, and on combination with various amines and phenols give azo dyestuffs which dye cotton directly, the shade varying from yellow, through red, to black, according to the nature of the dyestuff component. The shades, which are very similar from the two bases, are said to be fast to ordinary reagents, but not to light.—T. A. L.

Tetramethyldiamidodiphenylcarbinol on Para- and Meta-Sulphanilic Acids, Products of the Action of. Suais. Bull. Soc. Chim. 1897, 517—519.

On heating together in a litre of water, 46·2 grms. of sodium p-sulphanilate, 18 c.c. of hydrochloric acid, and 54 grms. of tetramethyldiamidodiphenylcarbinol on a water-bath for six hours to 80°—90° C., a number of products are obtained in practically equal quantities. 1. A yellow crystalline substance dyeing tannin-mordanted cotton yellow, similar to Auramine; it is decolorised by acids even in the cold. 2. A sulphonated leuco base converted by lead peroxide into a green dyestuff for wool, decolorised by alkalis. 3. Hexamethyltriamidotriphenylmethane (Crystal Violet). 4. A substance soluble in water, which does not yield a dyestuff on oxidation. With m-sulphanilic acid the following quantities were taken:—34 grms. of m-sulphanilic acid, 51 grms. of hydrol, 57 c.c. of hydrochloric acid, and 530 c.c. of water. After heating on a water-bath for 10—12 hours, the liquid contains several substances corresponding to those obtained from p-sulphanilic acid, with, however, certain differences. 1. The yellow substance only forms about 4—5 per cent. of the mass. 2. The sulphonated leuco base is the principal product. On oxidation it yields a violet dyestuff, which is turned green by caustic soda. When diazotised, boiled with water, and then oxidised, it yields a green dyestuff, which is reddened by alkalis. If, however, the diazo compound be boiled with absolute alcohol and the product oxidised with lead peroxide, a dyestuff is produced which dyes wool bluish-green shades remarkably fast to caustic soda. According to its formation the substance is to be regarded as an o-sulphonated Malachite Green and the two following analogously constituted acids—

$$1.3.5.C_6H_3.NH_2(SO_3H)_2 \text{ and}$$
$$1.3.5.C_6H_3.CH_3.NH_2.SO_3H$$

are being examined in a similar manner. The author regards the stability to alkalis as due to the o-position of the sulphonic acid group relative to the methane carbon—a constitution also possessed by the derivatives of the Patent Blue series.—T. A. L.

Xylidines, Commercial; Estimation of. W. Vaubel. Zeits. Anal. Chem. 1897, **36**, [5], 285.

See under XXIII., *page* 639.

α-Naphthol from β-Naphthol, A Reaction permitting the easy Distinction of. E. Leger. J. Pharm. Chim. 1897, **5**, 527.

See under XXIII., *page* 638.

PATENTS.

Aromatic Nitro-Derivatives, The Manufacture and Production of New Products from. J. Y. Johnson, London. From "The Badische Anilin and Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 14,955, July 6, 1896.

By the action of slightly fuming sulphuric acid on 1.1′- or 1.4′-dinitronaphthalene, products are obtained which, by further treatment in various ways, yield dyestuffs. For instance, about 10 kilos. of 1.1′-dinitronaphthalene are gradually added to about 50 kilos. of fuming sulphuric acid (12—23 per cent. SO_3), the whole being stirred and the temperature maintained at 40°—50° C. until a sample is soluble in dilute caustic soda with a yellow colour. The melt is then poured into water and the precipitate filtered off. It may be purified by crystallisation from glacial acetic acid. The new product is not a dyestuff, but may be converted into one by treating it with fuming sulphuric acid (40 per cent. SO_3), preferably in presence of sulphur. It then dyes chrome-mordanted wool brownish-yellow shades fast to light and milling. Brown to red dyestuffs are produced by the action of sulphuric acid of various strengths, with or without the addition of boracic acid or similar reagents. Dyestuffs are also obtained by treating the product with caustic soda or by nitrating it. On reducing the product it yields a base which can be employed in the manufacture of azo dyestuffs.—T. A. L.

Colouring Matters [Cotton Blacks], Impts. in. H. R. Vidal, Paris, France. Eng. Pat. 16,449, July 24, 1896.

The patentee finds that not only does sulphur react with disubstituted benzene and naphthalene derivatives, but that certain trisubstituted derivatives, such as diamidophenols and diamidonaphthols, react in a similar manner. Thus 3·2 kilos. of sulphur and 12·6 kilos. of 1.2.4-diamidophenol are heated at 80°—120° C. for 6—8 hours, when a product is obtained which dissolves to a blue solution in alkaline sulphides and dyes cotton black. Sodium sulphide is added preferably to the melt, in order to dissolve the sulphur and diamidophenol. The diamidophenol may be replaced by the corresponding dinitrophenol or by dinitronaphthol, but in that case it is necessary to first reduce the dinitrophenol with sodium sulphide, and afterwards add sulphur. For instance, 1·840 kilo. of dinitrophenol (1.2.4 or 1.2.6, or a mixture of the two) is heated with 3 kilos. of sodium sulphide to 140° C. in a vessel provided with an agitator. After reduction, 350 grms. of sulphur are added. When the melt is finished, it is run out, cooled, and ground.

—T. A. L.

Colouring Matters [Blues, Blacks], Production or Manufacture of New. I. Levinstein and Levinstein, Ltd., Manchester. Eng. Pat. 17,064, Aug. 1, 1896.

A further development of Eng. Pat. 2946 of 1896 (this Journal, 1897, 136). By heating with glacial acetic acid and anhydrous sodium acetate, the 1.4.2-diamidonaphthalene sulphonic acid there described, it is converted into an acetyl derivative having the formula—

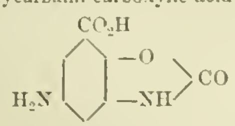
$$C_{10}H_5.NH_2.NHCOCH_3.SO_3H(1.4.2),$$

which can be used for the preparation of dyestuffs. It is prepared by heating 1 kilo. of naphthalenediamine sulphonic acid with 3 kilos. of glacial acetic acid and 1 kilo. of anhydrous sodium acetate under a reflux condenser for about 12 hours. The reaction is complete when a sample is completely soluble in water. After distilling off the excess of acetic acid, the product remaining is sufficiently pure for technical purposes. It yields a sparingly soluble yellow diazo compound, which gives dyestuffs with amines and phenols. These, after saponification, can be again

diazotised and combined, yielding diazo dyestuffs; or, if the amine employed in the first combination be capable of further diazotisation after combination, trisazo dyestuffs can be obtained. The following is an example of the first method:—30·2 kilos. of amido-acetnaphthalide sulphonic acid are dissolved in 300 litres of water, cooled with ice, acidified with 24 kilos. of commercial hydrochloric acid, and diazotised with 7·2 kilos. of sodium nitrite in aqueous solution. The sparingly soluble yellow diazo compound separates, and, if combined with 35 kilos. of β-naphthol disulphonic acid R in an alkaline solution, a deep red dyestuff is formed, which may be salted out. In order to saponify it, the press cakes are boiled with 500 litres of 10 per cent. caustic soda until a sample on adding acetic acid gives a pure reddish-violet colour. An excess of hydrochloric acid is then added, and the whole is diazotised with 7·2 kilos. of sodium nitrite at a low temperature, and combined with 14·5 kilos. of β-naphthol in an alkaline solution. A dark blue dyestuff is thus produced, which gives greener and brighter shades on wool than the isomeric colour described in Eng. Pat. 2946 of 1896 (*loc. cit.*), and also possesses a greater affinity for vegetable fibres. If in this example the 35 kilos. of R salt be replaced by 24 kilos. of γ-amidonaphthol sulphonic acid, the dyestuff, after saponification, contains two diazotisable amido groups, and, moreover, dyes wool a deep violet black from an acid bath. In order to convert it into its tetrazo derivative, it is dissolved in 10 litres of water, cooled with ice, acidified with 70 kilos. of hydrochloric acid, and treated with an aqueous solution containing 14·4 kilos. of sodium nitrite. After stirring for about 12 hours the diazotisation is complete, and the whole is mixed with an aqueous solution of 25 kilos. of *m*-tolylenediamine, the excess of hydrochloric acid being neutralised with sodium acetate. The mixture is then made alkaline and the dyestuff salted out, filter-pressed, and dried. It dyes animal and vegetable fibres a deep black. The base can be employed for other combinations also.—T. A. L.

Amino-oxycarbanil-carbonic Acid, and Azo Dyestuffs [Blue] therefrom, Fixing on Chrome Mordants; Manufacture of. O. Imray, London. From G. H. Weiss, Charlottenburg, Berlin, Germany. Eng. Pat. 17,207, Aug. 4, 1896.

The specification refers to the production of dyestuffs from the base amido-oxycarbanil carboxylic acid—



which is obtained by the action of phosgene on nitro-amido salicylic acid (m. pt. 220° C.) (from dinitro salicylic acid by partial reduction) and subsequent reduction of the condensation product. The nitro-amido salicylic acid is dissolved in water together with 4½ times the quantity of alkali theoretically necessary for its solution, and phosgene is passed in (below 35° C.) until a sample is no longer diazotisable, when hydrochloric acid is added to the melt to complete the precipitation. After purification by recrystallisation from hot water, the product forms needles melting at 263° C., easily soluble in water and alcohol, but insoluble in benzene. It is reduced by heating together 4·23 kilos. of the nitro body, 8 kilos. of tin, and about 16 kilos. of hydrochloric acid and the same quantity of water. The tin is removed by sulphuretted hydrogen or caustic soda, and the base is separated by sodium acetate or acetic acid. It forms white needles and melts at 252° C. It can be easily diazotised, and gives azo dyestuffs with the usual components, which produce very fast shades on chrome-mordanted wool; and better results still are obtained by a subsequent treatment with potassium bichromate. By combining the base with α-naphthylamine or with 1.3′- or 1.2′-naphthylamine sulphonic acid, re-diazotising, and combining with naphthol, dihydroxynaphthalene or amidonaphthol mono- and disulphonic acids, dyestuffs are produced which give black shades on chrome-mordanted wool fast to soap, light, and fulling. The following example gives the quantities employed:—19·4 kilos. of the base are dissolved in 500 litres of water by means of 12 kilos. of 33 per cent. caustic soda lye and 7 kilos. of sodium nitrite are added. The whole is then cooled to 5 C. and run into 500 litres of water and 40 kilos. of hydrochloric acid, 22° B. A solution of 18 kilos. of α-naphthylamine hydrochloride in 1,000 litres of water is then added, with constant agitation, to the diazo solution, and after adding sodium acetate, the reaction is complete in about six hours, when the amido-azo compound is filtered off. The press cakes are dissolved in 1,000 litres of water, together with the calculated amount of caustic soda lye mixed with 7 kilos. of sodium nitrite and slowly run at 15° C. into 50 kilos. of hydrochloric acid (22° B.) cooled with ice. The diazotisation is complete in about four hours, and the compound is then added to a solution kept alkaline with sodium carbonate, of 35 kilos. of β-naphthol disulphonic acid R. The dyestuff forms at once, and is separated by salting out and filter pressing. It dyes chrome-mordanted wool a deep and fast blue-black.—T. A. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

PATENTS.

Vegetable Fibres, Improved Method of Extracting. W. Warburton, Stockport. Eng. Pat. 13,815, June 23, 1896.

China grass and similar fibres are extracted from the plants by placing the latter in galvanised wire baskets and subjecting them in an ordinary high-pressure kier to the action of soap, soda, and petroleum under a steam pressure of 50—55 lb. per sq. in.—R. B. B.

Horsehair, The Manufacture of a Substitute for. H. W. Langbeck, Loughton. Eng. Pat. 29,168, Dec. 22, 1896.

Cocoa-nut fibres or "Mexican fibres" are boiled in an alkaline solution, well washed, and then dyed. When dry they are coated with a coloured spirit varnish and again dried. When the fibres are intended for upholstery this last drying is effected at 220°—240° F., but fibres to be woven are dried at the ordinary temperature.—R. B. B.

Textile Fabrics, An Impt. in. T. Zimmermann, Gnadenfrei, Germany. Eng. Pat. 10,077, April 22, 1897.

Threads are manufactured, composed of metal filaments and textile fibres twisted together, and these are woven or knitted into fabrics, being used either as warp or weft threads, or for both warp and weft.—R. B. B.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Turkey-Red Oil, Importance of, in Fixing Dyes. P. Wolff. Chem. Rev. Fett- u. Harz-Ind. 4, [8], 103—106.

Although in some cases the action of the oil in brightening colours on the fibre appears to be due to a merely mechanical envelopment of the particles of dyestuff, in others (alizarin, for example) it would seem that the fatty acid enters into combination with the alumina and alizarin forming the colour lake, the result being in accord with the law that the brightness and fastness of a colour increase with the complexity of the molecule. Confirmation of this chemical action of the oil is afforded by the resistance towards discharging agents exhibited by Alizarin-red when oil has been used in the dyeing. Analogous results are obtained with the Diazo-red prepared from *p*-nitrodiazobenzene and β-naphthol, the use of oil being found to develop the brilliant blue shade characteristic of Turkey red.

Additional confirmation of the part played by the oil in the formation of a lake is afforded by the very fast brown produced by treating the *p*-Nitraniline red with cupric salts, warm, and the resistance of this red to stannous chloride when developed with oil.

It is also found that Turkey-red oil in conjunction with a cupric salt is essential in the production of a marine blue colour from tetrazodianisol, the resulting dye being said to be superior in fastness to all other blues; and, moreover, the shade obtained is in close relation with the oil used, ordinary Turkey-red oil giving reddish shades, ammonium ricinoleate

greener shades, and still greener and darker shades being produced by desulphurised oxyoleic acid. No action, however, is exerted by the oil on the developed colour—a circumstance pointing to a reaction between the oil and the naphthol, which is confirmed by an observation made in connection with cloth impregnated with sodium-naphthol. This was found to turn brown when exposed to air, and in this condition gave impure shades; but when the sodium naphthol was associated with Turkey-red oil or antimony oxide, or both, the oxidation of the first-named was retarded, especially in the latter case, when pure shades were obtained on development after four weeks' exposure to the air.

To judge of the quality of samples of Turkey-red oil, the dyer is recommended to have recourse to sample dyeings with *p*-Nitraniline Red, employing an oil of known quality for comparison.—C. S.

Cement Water Pipes for Dyeworks. Leipziger Färber- u. Zeugdr. Zeit. **46**, [6], 257.

See under IX., *page* 613.

Sumach, Adulteration of, and Methods for their Detection. M. Spica. Gaz. Chim. ital. **27**, 349; Chem. Central-Bl. 1897, **1**, [22], 1101.

See under XXIII., *page* 639.

PATENTS.

Calico Printers' Blanketing, Floor-Cloth Printers' Blanketing, and the like; Impts. in the Method of and Means for Cleansing. A. B. Wimpenny, Hayfield. Eng. Pat. 23,642, Oct. 24, 1896.

The blanket is brought into light contact with a revolving highly polished metal roller, which removes from it the greater part of the dirt and colouring matter. The colour, &c., is taken from the roller by one or more "doctors" and led into a trough. The blanket, after this treatment, is further cleansed in the ordinary washing machine by means of revolving or reciprocating brushes.—R. B. B.

VII.—ACIDS, ALKALIS, AND SALTS.

Sulphur and Hydrogen, Conditions of Direct Combination of. H. Pélabon. Comptes Rend. 1897, [13], 686.

M. Hautefeuille has shown that when sulphur and hydrogen are heated together at a temperature near to 440° in a closed vessel, they combine to form sulphuretted hydrogen, and also that sulphuretted hydrogen is dissociated at 440°. Furthermore, that at temperatures above 440° the combination is prevented by the inverse reaction (Comptes Rend. **64**, 610).

Below 215° there is no combination.

Between 215° and 350° the combination is limited, but this is not owing to any inverse reaction. The author has already shown that when sulphur and hydrogen are heated together, the quantity of sulphuretted hydrogen produced increases as the mass of sulphur employed is more considerable, owing to the fact that molten sulphur is capable of absorbing H_2S and gives it off again on solidifying (H. Pélabon, this Journal, 1897, 142).

The direct combination is quicker in proportion to the elevation of temperature.

The maximum quantity of H_2S produced (that of the sulphur employed being the same) increases very rapidly as the temperature employed is higher. Thus, taking in each case 0·02 grm. of sulphur, when the temperature was 220°, only 2·10 per cent. of the hydrogen present was converted into H_2S, even after 624 hours of heating; while, with a temperature of 350°, 97 per cent. was so converted on 27 hours' heating.

When, however, these same experiments were made, using 50 times as much sulphur, very different results were obtained, owing to the absorption of H_2S by molten sulphur already referred to.

Beyond 440° the results are the same, whether a mixture of sulphur and hydrogen or the gas H_2S alone be employed. If, instead of pure H, a mixture of H and N be used, the quantity of H_2S produced at any given temperature, is lessened, but, other conditions remaining the same, the difference is less as the temperature is increased.—J. H. C.

Aluminium Chloride, The Preparation of. R. Escales. Ber. 1897, [10], 1314—1317.

In the preparation of aluminium chloride by the action of hydrochloric acid gas on aluminium, it is not necessary to apply heat during the whole process. If the metal be heated at the commencement, the heat generated by the reaction is sufficient for its completion. A bell-jar with two tubulures is placed air-tight on a glass plate; on this plate is a glass dish, which should be as large as possible; above the dish, supported on a glass tripod, is an asbestos sheet covered with 20 grms. of aluminium filings. Through the lower tubulure, hydrochloric acid gas is introduced; when most of the air is expelled, 0·5—1 grm. of heated aluminium is thrown in through the upper tubulure. This is conveniently heated in a crucible, and must be as hot as possible; if the reaction does not commence, a second quantity is added. The aluminium chloride produced sublimes on to the sides of the bell-jar. If the stream of acid be carefully regulated, very little chloride issues from the tubulure; to prevent any loss, this may be fitted with a tube.—A. C. W.

Metastannyl Chloride. R. Engel. Comptes Rend. 1897, **124**, 765—768.

It is well known that the reactions of stannic chloride vary according to the nature of the solution used, a freshly prepared aqueous solution of the anhydrous chloride not giving the same reactions as a similar solution when old, or as one prepared by the action of hydrochloric acid on metastannic acid ("β-chloride"). Regarding the distinctive reactions and true nature of the β-chloride, different observers are, however, by no means in agreement. Schützenberger considers it to be a chlorhydrin of metastannic acid. This view the author confirms, but at the same time concludes, from his observations, that there exist two distinct β-chlorides, differing in properties, and corresponding to two distinct metastannic acids. One of these chlorides is obtained when metastannic acid (prepared by the oxidation, at a comparatively low temperature, of tin by nitric acid) is treated with one-fourth of its weight of hydrochloric acid, and the resulting solution, after slight dilution, precipitated with an equal volume of hydrochloric acid. The precipitate, when dried *in vacuo*, forms translucent masses having the composition expressed by the formula $Sn_5O_9Cl_2.4H_2O$ (*Metastannyl chloride*). The salt is deliquescent in moist air, and is very soluble in water and absolute alcohol. The aqueous solution remains bright when diluted, providing a few drops of hydrochloric acid be added. But if largely diluted, the acid being omitted, a white gelatinous precipitate of metastannic acid ($Sn_5O_{11}H_2.4H_2O$) is obtained, from which, when dried, the chloride may readily be regenerated.

Stannic chloride, in dilute solution, is gradually converted into the meta-chloride in accordance with the equation $5SnCl_4 + 9H_2O = Sn_5O_9Cl_2 + 18HCl$.

An acidulated solution of metastannyl chloride is not precipitated by dilute sulphuric acid. In a note to follow, the author will describe the properties and mode of preparation of another chloride, corresponding to a second metastannic acid, the solution of which *does* yield a precipitate on addition of sulphuric acid.—H. T. P.

Magnesium Sulphate, An Adulteration of. P. A. Lamanna. Boll. Chim. Farm. **36**, 198; Chem. Central-Bl. 1897, 1100.

See under XXIII., *page* 636.

New Vinegar Bacterium. Zeidler. Central-Bl. für Bakt und Parasitenkunde, 1896, No. 23—24.

See under XVII., *page* 626.

PATENTS.

Electrolytic Decomposition of Liquids [Alkali Chlorides, &c.], Impts. in or connected with Apparatus for the. G. and G. W. Bell, Liverpool. Eng. Pat. 11,133, May 22, 1896.

See under XI. A., *page* 617.

Water-Glass, Impts. in the Manufacture and Production of, with Valuable By-Products [Hydrochloric Acid, Chlorine, Nitric Acid] of the said Manufacture. H. Propfe, Mannheim, Germany. Eng. Pat. 14,953, July 6, 1896.

When it is desired to obtain hydrochloric acid as a by-product, a mixture of 100 parts of sand, 60 parts of sodium chloride, and 4 parts of charcoal is heated, and 60 parts of sulphuric acid are admitted. The HCl is evolved at 200° C. The residual mixture is then transferred to the melting furnace and fused to water-glass. Saturated brine may replace the salt; sawdust or peat, the charcoal; and sodium bisulphate, the sulphuric acid.

To obtain chlorine, a mixture of sand, rock salt, and saltpetre is treated with sulphuric acid, and charcoal is added to the residual mixture before fusing it to water-glass. Nitric acid is obtained by similarly treating a mixture of sand and saltpetre, charcoal being, as before, added to the residue before fusion. To obtain nitrobenzene or similar nitro compounds, by a corresponding method, benzene is added to a mixture of sand and saltpetre, and then, slowly, sulphuric acid. The use of the spent sulphuric acid of the mineral oil industry for obtaining hydrochloric acid as a by-product is claimed; and in this case the impurities present serve to replace the reducing agent otherwise required.—E. S.

Oxalic Acid, Improved Process for Producing. G. F. Zacher, Hamburg, Germany. Eng. Pat. 2308, Jan. 28, 1897.

Sawdust or other cellulose material is placed in a steam-jacketed vessel connected with an air-pump. After exhaustion of air, the temperature is slowly raised to about 70° C., to deprive the sawdust of moisture and air. Hot alkali lye is then admitted, and stirring is maintained, and the vacuum kept constant until a temperature approaching 180° (but not exceeding that) is reached. Near the end of the process, either an oxidiser, such as sodium peroxide or hydrogen peroxide, is added; or air enriched with oxygen is slowly sucked in. The finished product is dissolved in water in the same vessel, and the solution is forced into an open stirring cylinder, in which it is thinned and treated with lime. The calcium oxalate obtained, practically free from carbonate, is decomposed by sulphuric acid; and it is stated that "a good white oxalic acid is produced by this improved process with only one crystallisation."—E. S.

Ammoniacal Liquor, A Process for Removing Carbonic Acid and Hydrogen Sulphide from. A. Feldmann, Bremen, Germany. Eng. Pat. 10,501, April 27, 1897.

Gas liquor is heated in distilling columns to from 94° to 97° C., at which temperature "a simultaneous and sufficiently complete removal of carbonic acid and hydrogen sulphide will be effected." It is supposed that at the stated temperature ammonium carbonate is dissociated, and that the carbonic acid set free, decomposes ammonium sulphide, liberating hydrogen sulphide. A column apparatus suitable for the conduct of the process is shown.—E. S.

Acetates of Lead, Copper, and the like; Process and Apparatus for the Production of the. A. Schmidt, Cassel, Germany. Eng. Pat. 7192, March 19, 1897.

The apparatus employed to produce neutral lead acetate consists of a pair of closed iron cylinders, lined internally with acid-resisting materials, connected at the lower part by tubes worked by an intermediate pump, and above by a valved arched pipe. Each cylinder has a steam-heating compartment at the bottom, a man-hole at top, and other accessories. The cylinders are half-filled with shot or small pieces of lead, and the second of them is charged until about three-fourths full with dilute acetic acid, and heat is applied. When the upper space of the cylinder is full of the acid vapour, the man-hole is closed, and the acid is pumped into the first cylinder, while oxygen under pressure is admitted. After the oxygen has acted upon the moist lead surfaces for about half an hour, the acid is pumped back, and the oxygen passes into the other vessel, where it acts similarly; and the process is continued, at a temperature of from 50 to 60° C., as long as necessary. After withdrawing the solution of lead acetate, fresh acid is added and the process repeated. Neutral copper acetate is similarly obtained, copper being substituted for lead.

To obtain subacetate of copper, a single cylindrical vessel, having a perforated false bottom above the steam-heating compartment, is used. Thin copper plates, separated by wooden laths, are placed upon the false bottom, below which acetic acid is introduced, the vapour of which, on applying heat, displaces air; oxygen is then forced in, to act jointly with the acetic acid vapour on the copper surfaces. By regulating the proportions of the oxygen and acid, a salt of any required basicity is produced.—E. S.

VIII.—GLASS, POTTERY, ENAMELS.

Pottery, Studies in the Art of; especially the Influence of the Furnace Atmosphere on the Colours given by Metallic Oxides. M. E. Damour. Bull. Soc. d'Encouragement, 1897, 322—325.

The author finds (1) that the coloration varies in a regular and continuous manner with the composition of the furnace atmosphere: excellent plates are given showing the colours obtained in the experiments; (2) that the tints obtained are constant with a constant atmosphere; (3) that with a naked fire the coloration is uncertain and unequal even when the greatest care is exercised.

The opinion is expressed that for fine porcelain the attempt should be made to introduce in commercial practice a muffle furnace heating to 1,300° C. The question of heating is still the key of all important progress in the potter's art, and an exact knowledge and control of temperature are subjects demanding the closest attention.
—V. C.

Glass, Colouring by Direct Penetration of Metals or Metallic Salts. L. Lemal. Comptes Rend. 124, [20], 1097—1099.

The author finds that by applying a salt of silver to the surface of glass at a temperature of 500° to 550° C., the glass becomes coloured pale to dark orange-yellow according to its composition, the former shade being produced in ordinary glass and the latter in glass prepared from:—sand 100 parts, lime 40, sodium chloride 30, sodium sulphate 34, sodium carbonate 5, arsenic 0·5, alumina 3, and carbon 1·5 parts, the colouring agent in both cases being a mixture of 10 grms. of silver sulphide and 100 grms. of red ochre.

The amount of silver salt required is very small, a 1 per mil. solution of silver nitrate sufficing to reproduce in dark yellow the design of a piece of lace dipped therein, and, after immersion in potassium sulphide, applied to the heated glass. The same result was obtained from a collodion photographic print.

The penetration effected by the silver salt increases with the strength of the solution and the time of exposure to heat. After heating for 5 minutes the glass was penetrated to a depth of 0·17 mm., and, after another hour's heating, to 0·32 mm.; and by renewing the colouring composition at intervals of 6 hours, a sheet of glass 1·57 mm. thick was found completely permeated after heating for 18 hours.

The coloured glass is dichroic, reflecting a phosphorescent tinge ranging from yellowish-green to bluish-violet—a property intensified by the addition of powdered copper (reduced from copper sulphate by zinc). In the case of glass coloured red by means of Cu_2O, a photographic reproduction by the new process, gave the dichroic image by reflected light, but it was invisible by transmitted light, the colour being masked by the copper red.

Coloured glasses, except those coloured with manganese oxide, transmit the imprint produced by the new process, and analogous results to those in the case of silver are obtained by using gold, copper, or iron.

In a note to the paper, A. Gautier states that he has already, in conjunction with Helier, observed a penetration of glass by metallic silver in vessels containing hydrogen

(or oxy-hydrogen) gas when exposed for several hours to a temperature of about 450° C. The silver disappeared, leaving the glass opaque, but the latter afterwards recovered its transparency.—C. S.

PATENTS.

Glass-Etching Plates for Use in Reproducing Designs on Stone, Zinc, Copper, Brass, and the like ; Method of Producing. T. Löhler, Mannheim. Eng. Pat. 9901, May 9, 1896.

A method for producing a non-transparent white coating upon glass plates for etching, in which the plate is first covered with a layer of "collodion and iodine," then treated with a solution of silver nitrate, next with an aqueous solution of green vitriol containing sulphuric acid and alcohol, and finally with a solution of mercury and albumin. The plates can be removed by treating with "the dry vapours of volatile chemicals."—V. C.

Glass, Impts. in the Manufacture of Sheets, Cylinders, and other Forms of. P. T. Sievert, Dresden. Eng. Pat. 17,391, Aug. 6, 1896.

The inventor claims the employment of a glass-melting furnace with slits of the required shape for the molten glass to pass through by gravity, in order to be drawn into a form corresponding to the slits, in combination with an arrangement of furnace for heating the glass at its exit. —V. C.

Glass-melting Furnaces or Troughs, Impts. in or connected with the Construction of. W. P. Thompson, Liverpool. From M. Mühlig, Teplitz, Bohemia. Eng. Pat. 2315, Jan. 28, 1897.

The inventor claims glass-melting furnaces constructed of an improved brick, "which is provided with recesses or bulgings, with the object of producing a more intense cooling of the walls of the furnaces or troughs and the joints of the bricks."—V. C.

Ceramic Masses, Improved Process for Producing. C. C. Schirm and O. Lessing, Berlin. Eng. Pat. 4539, Feb. 19, 1897.

The process consists in mixing fired, powdered materials (porcelain, earthenware, &c.), plaster of Paris, and flux, firing the mixture, and subsequently saturating it in a water-glass solution.—V. C.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Puzzolana and its Practical Use. J. Zervas. School of Mines Quarterly, **18**, [3], 226.

As its name implies, puzzolana was formerly found principally at Puzzuoli, in the neighbourhood of Naples, though at the present time a similar material is obtained from many other places, as from the valley of the Rhine (where it is known as tuffstone, or in the powdered condition as trass), the Azores, Schemnitz, Auvergne, &c. The Italian puzzolana is mostly obtained from shallow open pits, but large deposits are also found. Besides being indifferently manufactured, the material itself is inferior to the products from the Rhine and the Azores. French engineers use less of it than they formerly did in their marine constructions, and Dutch engineers employ "trass," which is selected and manufactured with greater care. Both puzzolana and trass are the products of former volcanic eruptions, and, though different in appearance, are substantially the same in chemical composition, as will be seen from the following table of analyses :—

—	Trass (Germany).						Puzzolana.					
Origin	Plaidt. Tuffstone.	Kruft. Tuffstone.	Brohl. Tuffstone.	Brohl. Mountain Trass.	Weibern. Leucit-tuff.	Seven Mountains. Trachyt-tuff.	Azores, St. Miguel.	Azores, Terceira.	Azores, St. Miguel "Temu."	Italy, Naples.	Italy, Civitia Vecchia.	Santorin.
Analyst.....	Zervas.	Mengerschausen.	Berthier.	Kyll.	Kyll.	Kyll.	Chateau.	Zervas.	Zervas.	Stengel.	Berthier.	Feichtinger.
Silica	53·58	55·28	57·00	61·10	67·60	59·40	54·70	57·73	60·90	60·91	44·50	66·37
Alumina....	19·11	17·34	16·00	12·70	11·30	22·70	20·50	13·81	11·14	21·28	15·00	13·72
Ferric oxide	9·24	3·90	5·00	10·20	5·20	2·50	6·30	12·02	12·78	4·76	12·00	4·31
Lime	3·21	3·17	2·60	8·10	8·20	3·10	2·20	3·74	2·57	1·90	8·80	2·98
Magnesia ...	0·30	0·87	1·00	1·90	2·80	0·80	1·70	1·73	1·45	0·00	4·70	1·29
Potash	4·84	4·70	7·00	2·10	0·60	3·50	2·20 (Potash and Soda)	3·21	2·64	4·37	1·40	2·83
Soda........	1·87	3·80	1·00	2·10	0·50	2·80		2·76	2·74	6·23	4·10	4·22
Water	7·50	10·63	9·60	1·40	3·10	4·80	12·40 (Water and Loss)	4·67	5·78 (Water and Loss)	0·55 (Water and Loss)	9·20	4·06
Loss	0·35	0·31	0·80	0·40	0·70	0·40		0·33			0·30	0·22

All that is needed to make a good hydraulic mortar is lime, and this is added with the sand. Probably equal parts of puzzolana (or trass), lime, and sand give the best results. The proportions used in the Kiel harbour works were :—

Trass	3	parts by volume.
Fat-lime paste	2	" "
Sand..........................	7	" "

In Holland the following are the standard formulæ for hydraulic works :—

—	Trass.	Shell-lime Paste.	Sand.	Stones.
Strong trass mortar.....	1 vol.	1 vol.	..	..
Second quality..........	2 vols.	4 vols.	1 vol.	..
Third quality	2 vols.	6 vols.	3½ vols.	..
Concrete................	1 vol.	1 vol.	..	1¾ to 1½ vols.

As trass and puzzolana commence to harden only after the addition of the lime, they can be stored, even in damp places, without deterioration ; and this renders it possible to export them without the risk incurred in shipping Portland cement, whilst it also enables the engineer to make better arrangements with regard to purchase and storage. If the lime used be perfectly slaked, mortars made with puzzolana or trass do not suffer the expansion or contraction so commonly observed with cement. Mixed with 20 per cent. of water, puzzolana mortars composed of 1 volume each of puzzolana, lime, and sand, 28 days old, break at more than 200 lb. per sq. in.

To test impermeability to water, a circular conical disc of 20 cm. surface at the smaller face and 15 mm. thick, is made of standard mortar and hardened in water. By means of a rubber ring, the disc is tightly fitted into a cylinder, and while water is filled into the cylinder above the disc, the air is exhausted from below. After 24 hours, the percolated water is weighed. Comparative experiments made with puzzolana and trass, and with slow-setting cement, show results greatly in favour of the former, and this important quality is fully recognised by Dutch and German engineers in the construction of sluices and basins.

Experiments made to test the adhesive strength of mortar to stone in concrete, showed that, unlike cement, puzzolana and trass mostly yield a higher strength by the

addition of the stones. The same experiments also showed that a larger addition of lime should be made to puzzolana or trass when made into concrete than when used without stones.

In order to test the adhesive strength of separate layers of mortar, experiments were carried out as follows:—Specimens, prepared in the same way as for testing tensile strength, were made and immersed in water to harden. After lying in water for certain definite times, some of the specimens were taken out and divided into halves, the remainder being allowed to remain in the water, to be used later on for testing for tensile strength. Each of the halves was placed again in a mould, the empty half of the mould filled up with fresh mortar, carefully pressed to the old half specimen, and the whole re-immersed in water. The results obtained are shown in the following tables:—

Table I.

Mixture.	Medium Temperature of Water.	Tensile Strength after 28 Days. Kilos. per Square Centimetre. Regular Specimens.	Tensile Strength after 28 Days. Kilos. per Square Centimetre. Specimens joined after 24 Hours.
	° C.		
1 trass, 1½ fat-lime paste, 3 sand, without water.	19¼	15·00	12·00
Cement, standard mixture ..	19½	11·25	6·25

Table II.

Mixture.	Temperature.	Regular Specimens.	Joined after 36 hours.
	° C.		
Trass. The same as in Table I.	15¼	13·25	9·25
1 trass, 1 fat-lime paste, 1 sand, and 5 per cent. of water.	15	14·50	11·00
Cement, standard mixture ..	15	9·50	4·00

Puzzolana or trass mortar sets harder in sea-water than in soft water. Taking the tensile strength of the mortar hardened in soft water at 100, the same hardened in sea-water showed the following results:—

Trass.	Fat-lime Paste.	Sand.	After 28 Days' Hardening.
2 parts by weight	1 part by weight	.. ..	111
1 part „	„ „	1 part by weight	168
1 part by volume	„ volume	2 parts by volume	132

Trass has been used in the construction of most of the bridges over the Rhine, Weser, and Elbe, in the maritime works of the German Government, and in Holland and Belgium, 60,000 tons having been used in the construction of the Antwerp docks alone.—H. H. B. S.

Cement Water Pipes for Dyeworks. Leipziger Färber- u. Zeugdr.-Zeit. **46**, [6], 257.

Water pipes made of hydraulic cement will stand a high pressure, and buried in moist ground are indestructible. They are made locally, where wanted, as follows:—On the well-prepared foundation a gutter is made by means of two boards. In the centre of this, lengthwise, a smooth sheet-iron pipe, of the thickness of the desired bore, is laid, and the prepared cement, consisting of two parts of hydraulic cement to one part of pure sand mixed with water, is poured over it. After about 15 minutes, when the cement has partly set, the iron tube is carefully turned round and pulled out, leaving the end only in the casting, and the previous operation is repeated. In this wise a pipe of any length can be made in one piece. Pipes of larger diameter may be constructed in a similar manner, by using for the core a tube consisting of wooden staves, so constructed that one of them can be pulled out when the cement is sufficiently hardened, whereupon the others can be easily removed.

—I. S.

PATENTS.

Silica Bricks or other Highly Refractory Bricks, Impts. in the Manufacture of. J. Dixon, Oughtibridge, and A. Reynolds, Bolsover Hill, near Sheffield. Eng. Pat. 11,682, May 29, 1896.

To suitable siliceous material is added "half-stuff," cellulose, or other like fibrous material, which acts as a binding agent during the process of moulding.—V. C.

Artificial Stone, Marble, and the like; An Improved Manufacture of. W. Owen, Brixton. Eng. Pat. 12,368, June 5, 1896.

A method of manufacturing artificial stone and the like, in which the component parts are subjected to pressure under water free from air. A cylinder of special construction, which is described and figured in the specification, is also claimed.—V. C.

Paving Blocks, Tiles, and the like; Impts. in the Manufacture of. W. S. Wilkinson, Baltimore, U.S.A. Eng. Pat. 5439, March 1, 1897.

In the manufacture of comprecssed paving blocks, &c., the inventor claims a preparatory treatment of the asphalt, which consists in pulverising it and then macerating and agitating it in water. The ingredients besides the asphalt, are non-calcareous hard-stone *débris* and powdered limestone.—V. C.

Fillings for Coating Wood before Painting the same, Impts. in. Ph. Phillips, Balan, Colony of Victoria. Eng. Pat. 8686, April 5, 1887.

"Tun white lead," ground pumice stone, and varnish are mixed, either alone or with powdered asbestos. Pyrites are added when more durability is required.

The proportion used for an ordinary coating is 8 lb. of white lead, 4 oz. of pumice stone, 8 oz. of asbestos, and 1¼—1½ pints of varnish.—E. A.

X.—METALLURGY.

Dredge-Work in the Siberian Placers. E. D. Levat. Eng. and Mining J. 1897, **63**, 541.

The author, in his report on the gold-fields of the Trans-Baikal, describes the use of the dredge for working the second level of placers where the subsoil is yielding and marshy. He describes the bucket dredge used by the Verkhne-Zeya Company, which, owing to its large weight and the difficulty of moving it from place to place as desired, had to be mounted on a barge or pontoon, and floated in an artificial basin, filled with water from a stream flowing through the placer. The bedrock, on and in the cavities of which, nuggets are generally found, is worked over by hand as it is uncovered by the dredge. The gravel brought up by the buckets is deposited in a sluice carried on the dredge-boat; the water for the sluice, which is provided with riffles at the bottom, is furnished by a centrifugal pump. The tailings from the sluice are discharged into a wooden float, which, when full, is hauled to the side of the excavation and there discharged. The chief defect is the shortness of the sluice, which prevents the complete washing of the gravel. The author suggests the use of a Z-shaped sluice, with one or two under-currents.—A. S.

Furnace, The Bolthoff-Wethey Improved Roasting. Eng. and Mining J. 1897, **63**, 545.

This is a modification of the Wethey multiple-deck calcining furnace, arranged for the desulphurising of ores for chlorination and cyanide mills where a "dead roast" is desirable. The furnace is of rectangular form, and being supported by the same means on both sides, is not liable to injury from expansion and contraction. The design is such that no iron-work is exposed to the fire except the rabbles, which are

constructed of heavy pipe, to which steel ploughs are clamped, and are supported at both ends by carriages and driven by wire rope and sprocket wheels. The carriage is so constructed that the rabble pipe and ploughs can be lifted out and replaced without disturbing the connection between the carriage and the rope. Half of the ploughs on each rabble are set at one angle and half at an opposite angle, thus overcoming all end thrust. The ore is charged into the furnace at the driving end, and after travelling the full length of the roasting hearth is dropped to the cooling hearth and carried back to the charging end, thus allowing the ore the same length of time to cool that was required to roast it. In order to facilitate the cooling of the ore and ensure its temperature being sufficiently low to prevent injury to the conveyors and elevators, water pipes are laid the full length of this hearth, flush with the top: the amount of water necessary for the cooling of the ore can be regulated by the discharge from these pipes. The pipes are laid loose in grooves between the brick in such a manner that they can expand and contract without injury to the hearth or themselves. A valve is provided for shutting off the stream of ore from the feeder while the rabble is passing under it. All the ironwork of the furnace is exposed to the atmosphere, and is thus less liable to injury from the corroding action of the fumes than where the ironwork is enclosed or passes through the brickwork. It is claimed that this furnace is cheaper and requires less space than other types of furnaces. —A. S.

Zinc-Box Precipitate, The Treatment of. T. H. Leggett. Eng. and Mining J. 1897, **63**, 541. From the J. Inst. of Mining and Met., London.

The author describes a process for the cleaning up and drying of the zinc-box precipitate from the cyanide process. The slime sediment from the zinc extractor boxes is placed in a wooden tank, into which the zinc shavings are also washed through a coarse screen. The cyanide solution brought into the tank with the slimes and shavings is then pumped out by means of an india-rubber hose pipe attached to the pump suction. It is forced through a press, leaving the slimes at the bottom of the tank in the form of a thick mud. Concentrated sulphuric acid is then poured on, a bucketful at a time, until all the zinc is dissolved, a hood being lowered on to the tank for the purpose of leading the fumes outside the building. Boiling water is now run on to the slimes, which are kept agitated, until the tank is filled, when the hot slimy solution is drawn off by the pump, and forced through a filter press; the solution is kept stirred all the time, and more hot water is run in as required until the tub is empty. The slime cakes in the filter press are further washed by hot water until all acidity and zinc sulphate are removed, after which air is pumped through and the cakes finally taken out and dried in a closed muffle without stirring, so that no dust is made.

The following results were obtained by this method at the Treasury Mine, Transvaal. Weight of dried slimes, 108 lb. av. = 1,575 oz. troy; sulphuric acid used, 234 lb. av.; flux used — borax 5 parts, soda 3 parts, precipitate 10 parts; slag produced, 48 lb.; gold produced, 446·81 oz. fine; silver, 55 oz. fine. The bullion produced assayed 750·3 fine gold and 92·9 fine silver, or 843·2 total per 1,000. The solutions escaping from the filter press assayed 8 c. per ton in gold; and 400 gals. of hot water per 1,000 oz. of precipitate were used for washing. The total losses by this method are under 0·1 per cent., while much less and far cleaner slag is produced than by the nitre method, together with a higher grade of bullion.—A. S.

Metallic Sulphides, Formation of, by Mechanical Means. L. Franck. Bull. Soc. Chim. 1897, 504—506.

A number of instances are quoted in which small quantities of metallic sulphides are obtained by rubbing together, between paper, flowers of sulphur with certain metals in powder. When aluminium powder is rubbed together with flowers of sulphur, sulphuretted hydrogen is given off, and a similar reaction occurs with magnesium. On rubbing a silver plate or coin with flowers of sulphur, a yellow and then a black colour is produced, due to the formation of silver sulphide. On continuing the friction, the surface becomes uneven, and excrescences are formed, which, on removal, are found to contain silver sulphide. A gold coin shows a similar reaction, whilst with copper the effect is even more distinct. A freshly-cut surface of lead, when rubbed with flowers of sulphur, gradually turns black from the formation of lead sulphide.—T. A. L.

Metallic Sulphides [Cu, Bi, Ag, Sn, Ni, and Co], Influence of a High Temperature on. A. Mourlot. Comptes Rend. 1897, **124**, 768—771.

The author has submitted various sulphides—mostly prepared artificially—to the high temperature of an electric tube furnace, as used by Moissan. The conclusions are as follows:—

1. Copper and bismuth sulphides are completely desulphurised—in the case of copper, with distinctly greater difficulty than with bismuth.
2. Silver sulphide is similarly decomposed, but the reduced metal obstinately retains traces of sulphur.
3. Cobalt and nickel sulphides are reduced to relatively stable CoS and Ni_2S respectively, which at the maximum temperature of the furnace are further desulphurised, but not completely, a trace of sulphur being obstinately retained.
4. Stannous sulphide is not decomposed at the highest temperatures attainable, but is partly volatilised, partly converted into a crystalline mass of SnS, and must therefore be classed along with zinc, cadmium, and aluminium sulphides, which are stable at high temperatures.—H. T. P.

Nickel Steel, Metrological Properties of. E. Guillaume. Comptes Rend. 1897, **124**, 752—755.

The author has previously referred (Comptes Rend. 1897, **124**, 176) to the very small expansibility by heat of certain alloys of nickel and steel, and he pointed out the value of such metal for the construction of scientific apparatus, &c. The resistance of nickel steel to corrosion by moisture increases with the percentage of nickel present, and is already sufficiently marked in case of the alloy possessing minimum dilatability (containing about 36 per cent. Ni). Polished bars of this metal retain their lustre for months in a damp atmosphere, and even for a considerable period when submerged in water. Unpolished surfaces, however, rust in the course of a few days; and in any case, the attack, when once initiated, proceeds rapidly. All the alloys are very readily attacked by dilute hydrochloric acid.

As regards density and elastic modulus of nickel steel, it appears, roughly speaking, that minimum dilatability is accompanied by low density (as compared with the calculated mean density) and low modulus, and *vice versa*. (The most expansible alloy contains about 24 per cent. Ni). In order to determine the effect of annealing, test bars were repeatedly heated, and their length measured at intervals when cool. At 100° C. the alloys containing less than 25 per cent. of nickel contract permanently, whilst those richer in nickel expand. The absolute amount of expansion is greater at low than at high temperatures; but the rapidity with which the change takes place is greater, and the time required for a fixed and definite length to be attained, shorter at high temperatures. In confirmation, the curious fact is cited of a bar, which, when annealed at a given temperature, again expands when subsequently heated to a lower temperature, or contracts at a higher temperature. As regards the least dilatable alloy, a condition of equilibrium is arrived at in 20 hours at 150° C., 100 hours at 100° C., 300 hours at 60° C., 700 hours at 40° C., and in about two months at the ordinary temperature.—H. T. P.

Silver-Copper Group of Alloys. F. Osmond. Comptes Rend. **124**, [20], 1094—1097.

From the curves of fusibility obtained by Heycock and Neville (Trans. Roy. Soc. **189**, 25), it appears that the silver-copper alloys are merely mixtures of the two metals, and the existence of Levol's alloy, Ag_3Cu_2, as a definite compound is thus called in question. By subjecting this alloy to microscopic examination the author finds that the metals are distinctly separate, being recognisable by their colour, especially if the oxide colours of the copper be developed by heating; and that the structure of the alloy

resembles that of perlite, the copper being distributed in curved lamellæ, like the Fe_3C in iron, and producing a similar iridescence when slightly oxidised and viewed by oblique light. A power of 1,000 diameters is necessary to detect the grains of primary consolidation, which are enveloped in grains of coarser structure of the secondary order. When the cooling of the alloy is hastened, the lamellæ are still rendered visible by the iridescence, but their structure is not easy to distinguish, even under the highest powers. The results obtained, confirm the inferences drawn from the plotted curves of fusibility, and disprove the existence of Ag_3Cu_2 as a definite compound.—C. S.

Alloys, Metallic; Constitution of. G. Charpy. Comptes Rend. 1897, **124**, 957—958.

Eutectic Alloys.—These alloys have often been considered chemical compounds because they are homogeneous and solidify at a constant temperature; but the microscope shows that they consist of crystalline lamellæ of two constituents juxtaposed, and so minute that the structure is evident only under great magnification. The minuteness of the structure explains the apparent homogeneity and the conchoidal fracture. These alloys have the constitution of cryohydrates; they are not definite compounds, but resemble the mixtures of liquids distilling at definite temperatures studied by Berthelot. The pearlite in steel shows all the characteristics of such an eutectic mixture, and this is in opposition to Arnold's contention that the perlite is a definite alloy of carbon and iron, corresponding to $Fe_{24}C$.

Definite Compounds.—The existence of definite compounds of metals has been denied, and few besides Cu_4Sn and Cu_2Sb have been completely investigated. Micro-metallography shows the existence of these compounds. Thus, Cu_3Sn is seen in alloys containing more than 5 per cent. Cu in the form of white, very hard crystals forming six-branched stars (like snow crystals), increasing in number as the proportion of copper is gradually approximated to that contained in Cu_3Sn itself. Cu_2Sb is shown in the same way by the presence of hard violet-coloured crystalline forms. Microscopy also shows the existence of other definite compounds not yet isolated, and is of great use when the physical properties of the alloy give no clear indications, probably owing to phenomena connected with isomorphism. It has given evidence of the existence of the following compounds, which, however, remain to be isolated:—Of antimony and tin, with about 50 per cent. of Sn, and isomorphous with Sb; of antimony and silver, containing about 20 per cent. of Sb, isomorphous with Ag; of tin and silver, with 30 per cent. of Sn, isomorphous with Ag. The first-named of these gives beautiful crystals, with clear outline, in alloys rich in tin. In micro-metallography the form of the constituents of an alloy gives only an approximate indication, and better information is yielded by the colour, hardness, and by their behaviour towards various reagents.

Two types of structure are indicated by microscopic examination. In the first, crystals of a pure substance (element or compound) are enveloped in another substance, which is usually an eutectic mixture formed from two finely divided materials, of which one is identical with the substance of the crystals. In the second, isomorphous mixtures give a single type of crystals, which occupy the whole space, the composition and properties varying uniformly in each crystal. This type is of frequent occurrence; for, although but few metals are able to form isomorphous mixtures, there are many examples of definite compounds capable of forming such mixture with one or other of the constituent metals.—W. G. M.

Phosphor Bronze, The Manufacture of. M. H. Wickhorst. J. Amer. Chem. Soc. 1897, **19**, [5], 393—395.

Phosphor bronze is bronze containing a small amount (from a few hundredths to over 1 per cent.) of phosphorus, which may be added in substance as phosphorus, or in the form of a high phosphorus alloy. The latter method is the one described, and the high phosphorus alloy or "hardener" is made in the following manner:—90 lb. of copper are melted in a crucible under charcoal, 11 lb. of tin added, and the mixture heated.

The phosphorus to be added to the metal is weighed out in the following manner:—A stone jar half full of dilute copper sulphate solution is weighed, and phosphorus, in sticks about 4 ins. long, is added until the weight is increased by 7 lb. At the end of about half an hour the phosphorus is covered with a coating of metallic copper, which prevents ignition when it is dried and exposed to the air. The phosphorus is dried on blotting paper, placed on wire netting supported from ledges along the inside of a shallow, square, galvanised iron pan, containing about 2 ins. of water; the pan is provided with a lid, which may be put down in case of fire.

The crucible containing the molten metal is placed on the floor, and a cup-shaped instrument, called a retort or phosphoriser and fitted with a long handle, is held just over the brim. Two or three pieces of the prepared phosphorus are then placed in the retort and the latter immediately plunged into the metal before the phosphorus can fall or flow out. This operation is repeated, and after the 7 lb. of phosphorus have been thus added, the mixture is poured into thin slabs about 1 in. by 3 ins. by 4 ins.

The phosphor bronze is prepared by melting the copper under charcoal, adding the tin and lead, and then enough "hardener" to obtain a sufficient amount of phosphorus. —A. S.

Metals, Deformation of, under Strain. M. Mengin. Comptes Rend. 1897, 681.

The author refers to M. Hartman's experiments on steel and other metals (Acad. des Sciences, 1894), which proved that deformations under compression, flexion, &c. were produced in a series of waves in discontinuous zones, geometrically distributed according to well-determined laws.

The author's experiments on aluminium, nickel-steel, delta-metal, and brass, show that they obey the same general laws as steel, but he notes certain interesting peculiarities.

1. *Aluminium.*—When a bar is stretched, regular waves are produced on the surface in two conjugate systems, as with steel. But while the waves in the steel form a network of narrow furrows, in the aluminium a series of broad bands appears for a moment; the bands are not permanent, however regular the strain, but appear intermittently in certain zones, equally inclined to the direction of the strain. This phenomenon continues until one of the waves is converted into a permanent groove. Finally the bar is ruptured either along such a groove, or following the bisectrix of the angle formed by two conjugate depressions, if they are of equal depth. As in the case of steel, the undulations produced on the two sides of the bar are exactly the same, so that they are not mere surface phenomena.

2. *Nickel-steel* (25 per cent. nickel).—The phenomena are similar, the surface being covered with a network of lines, all making the same angles with the direction of strain. These lines are produced almost continuously, and sometimes they take the form of an X the branches of which have the same inclination as those of the isolated lines. Finally a permanent groove is produced in some well-determined zone of undulations. Outside this zone, the bar retains no traces of molecular movements. When the strain is relieved for an instant, the undulations in progress are abruptly arrested, but are reproduced when the strain is again exercised.

3. *Delta Metal.*—The same peculiarities as nickel-steel, but with this difference, that the X form undulations are more numerous and are propagated more continuously. The rapidity of propagation is a function of the speed of transmission of the strain, and the number of oscillations increases with this speed of transmission.

4. *Brass.*—With hammered brass a series of clearly defined rectilinear deformations is produced immediately and abruptly, which rigorously preserve the same angle with the direction of strain, and spread over the surface as the strain increases—not lines in the geometric sense of the word, but veritable depressions, of sometimes many millimetres, and which exhibit a network of very fine striæ parallel to the two conjugate systems of deformation.

—J. H. C.

Phosphor Bronze, Phosphor Copper, Phosphor Tin, &c.: Analysis of. M. Wickhorst. J. Amer. Chem. Soc. 1897, **19**, [5], 396.

See under XXIII., *page* 638.

Lead, Estimation of, in Slags and other By-Products. D. J. Williams. Chem. and Metall. Soc. of S. Africa, Proc. of Meeting, May 15, 1897, 11—14.

See under XXIII., *page* 637.

Lead, Determination of, in Lead Ores. R. K. Meade. J. Amer. Chem. Soc. 1897, **19**, 374.

See under XXIII., *page* 637.

Lead, Volumetric Determination of. J. H. Wainwright. J. Amer. Chem. Soc. 1897, **19**, 389.

See under XXIII., *page* 637.

Silica, Determination of, in Blast-Furnace Slag. G. H. Meeker. J. Amer. Chem. Soc. 1897, **19**, 370.

See under XXIII., *page* 636.

Silicic and Tungstic Acids, Note on the Separation of. J. S. de Benneville. J. Amer. Chem. Soc. 1897, **19**, 377.

See under XXIII., *page* 636.

PATENTS.

Gold and Other Metals from their Ores, A New or Improved Process for Extracting, especially applicable to the Treatment of Refractory Ores. J. A. Bruce-Fraser, London. Eng. Pat. 9524, May 5, 1896.

The gold is extracted by nascent chlorine produced by any convenient means. In the process cited, the pulverised ore is roasted if necessary, mixed with about 5 per cent. by weight of barium peroxide, placed in a closed vat with sufficient hydrochloric acid to cover the mixture, and allowed to stand for 12 hours. Water is then added till the solution contains only 3 per cent. of acid, and the whole is well agitated, allowed to settle, the liquor decanted, and the gold precipitated by a ferrous salt. If silver be present, common salt is added to the solution to dissolve the chloride, and the metal is precipitated with copper, whilst any copper present may be removed by scrap iron.—A. W.

Metals on Silver Coatings, An Improved Process for Depositing. The Improved Electric Glow Lamp Syndicate, Ltd., London. From C. Schwabe, Berlin. Eng. Pat. 9656, May 6, 1896.

In the deposition of copper or other metal on a thin film of silver deposited on glass for glow lamps, &c., it is found that the silver, together with the copper on it, flakes or scales off. This is now found to be due to traces of the silvering solution, contained in the pores of the silver and between it and the glass, reacting with the solution used for depositing the copper and producing movements of the liquid, heat, &c., which cause the silver film to become torn. To prevent this the author adds to the depositing solution of the copper or secondary metal, the remains, or the essential constituents of the same, of the silvering solution in adequate quantity, thus neutralising the secondary bath and preventing its reaction with the traces of primary solution in the pores of the silver.—A. W.

Metal or Admixture of Metals [Alloys of Al, Cu, Sn, Zn, Ag, and Sb Regulus], A certain New or Improved. W. Van Wart, F. W. Popp, and J. J. Bradley, all of Birmingham. Eng. Pat. 12,287, June 5, 1896.

To obtain an alloy which shall give sound castings, and which shall be of very low specific gravity, but strong and workable, and suitable for spinning, stamping, rolling, casting, and drawing,—85 to 100 parts of aluminium are alloyed with 1 to 7 parts of copper, $\frac{1}{2}$ to 5 parts of tin, 1 to 7 parts of zinc, 1 to 5 parts of silver, and $\frac{1}{4}$ to 4 parts of antimony. The copper and silver are first melted together (or the copper alone, if silver be omitted from the mixture), the zinc and antimony are then stirred in very rapidly, the aluminium is then gradually introduced, and when the whole mass is thoroughly alloyed, the pot is removed from the furnace and the tin is added.—W. G. M.

Alloys, Chromium and Similar, Impts. in the Manufacture of. E. A. G. Street, Paris. Eng. Pat. 13,111, June 13, 1896.

Aluminium is added to molten iron or steel in quantity just insufficient completely to deoxidise it; the desired proportion of chromium is then introduced, with a slight surplus to effect the removal of the last trace of oxygen. In this way a perfectly fluid metal is obtained free from aluminium. —W. G. M.

Bessemer or Thomas Process for the Treatment of Low Qualities of Crude Iron, Impts. in the. L. Pszczolka, Krompach, Upper Hungary. Eng. Pat. 13,451, June 18, 1896.

The difficulty of refining white crude iron, containing insufficient silicon and phosphorus to produce the necessary heat in the ordinary process, is now overcome by blowing compressed and heated air upon, or laterally into, the crude iron bath. The hot air may be obtained from a Cowper blast-furnace apparatus, working at the usual blast-furnace pressure, and the furnace may be a converter or hearth of any kind. The surface of the metal is freed from slag in places by the blast, and thus exposed to direct oxidation by the hot air.—A. W.

Metallic Alloy, An Improved Manufacture of. C. Parnacott, London. Eng. Pat. 14,006, June 24, 1896.

The composition is as follows:—Copper, 85·15 parts by weight; iron, 2·15; magnesium, 1·0; zinc, 10·45; tin, 1·10; sal-ammoniac, 0·5; and "crude tartar," 0·1. The copper and iron are mixed by melting and pouring the former into the latter in a crucible, adding the sal-ammoniac, and well stirring. The magnesium is then added. The zinc and tin are melted together and poured into this mixture through a tube which dips under its surface. The crude tartar is then added, the whole well stirred, and the alloy cast. The result is a yellowish-red or yellowish-gold metal possessing great tensile strength and elasticity. —A. W.

Annealing Metals, and for other like Purposes; Impts. in Muffles for. Elkington and Co., Ltd., Birmingham, and H. T. Fellows, Acock's Green. Eng. Pat. 16,756, July 28, 1896.

The improvements "consist essentially in arranging in or combining with a closed chamber, or arranging under a closed hood, a rotating or stationary bed or pan, in which bed or pan the ignited fuel for the heating of the metals to be annealed, &c. is placed, the combustion of the fuel in the said bed or pan being maintained by a bottom forced draught of air supplied to the said bed or pan." "The combination with annealing muffles of the kinds just referred to, of a cooling chamber provided with a slowly rotating table," is also claimed. On this table "the heated articles from the annealing chamber are placed."—A. W.

Steel, Impts. in the Manufacture of. J. de Moya, Paris. Eng. Pat. 5926, March 6, 1897. (Date claimed under International Convention, Aug. 10, 1896.)

Sodium chloride, calcium carbonate, and plumbago are added to the bath of Bessemer or open-hearth steel before pouring, in order to produce a fine-grained homogeneous metal. In the case of an 8-ton basic Bessemer converter, 12 kilos. of NaCl and 20 kilos. of $CaCO_3$ are added immediately before removing the slag. The converter is rocked with the blast on to expel fumes; it is then allowed to remain at rest for about two minutes; the slag is removed, ferro-manganese is added to the extent of half the quantity ordinarily used; and finally 6 kilos. of plumbago are introduced, the converter being still at rest with the blast turned off. After a minute the charge may be poured. In the acid Bessemer converter, the same process is followed, excepting that the slag is not removed. The use in the open-hearth furnace is similar to that above described.—W. G. M.

Lead and Lead-Zinc Ores, Impts. in the Method of Decomposing. A. J. Boult, London. From S. Ganelin and J. Block, both of Philadelphia, U.S.A. Eng. Pat. 1179, Feb. 16, 1897.

The sulphides of lead and zinc are mixed with an equal weight of calcium (or magnesium) chloride, both ingredients being thoroughly pulverised. They are then treated for two days in the cold with concentrated commercial hydrochloric acid (22° B.). Galena is thus decomposed and the lead forms a double chloride with the calcium or other chloride, and under these circumstances does not prevent the further attack upon the galena; the hydrogen sulphide evolved is sufficiently free from hydrochloric acid gas to be used for conversion into sulphuric acid. The added chloride accelerates the solution of zinc sulphide in the ore, and for this purpose, ammonium chloride is to be preferred. With ammonium chloride, the greater part of the zinc is left insoluble in the acid liquid, probably as a double salt. After drawing off the solution, from which zinc and silver are to be recovered by existing processes, the ore is dried to remove H_2S and HCl. Instead of employing a solution of hydrochloric acid, the ore may be mixed with calcium chloride and from 2 to 4 molecules of H_2O per molecule of $CaCl_2$, and then treated with hydrochloric acid gas in the cold. Or the HCl gas may be passed through a concentrated solution of a chloride containing the ore in suspension. The ease with which the reaction of the hydrochloric acid with the sulphide is completed is due to the porosity of the double chlorides formed, rather than to chemical energy; for less heat is evolved by the action of hydrochloric acid upon galena when calcium chloride is present than when the sulphide is alone; and, further, a chemically inert, porous substance, such as coke or pumice, may be substituted for the calcium chloride. Lead-zinc ores often contain silicious matter, and are then capable of being treated without foreign admixture.

The chloride residue is freed from hydrochloric acid by exposure to the air for two or three days, or by blowing air through the mass, and is then leached with water, which dissolves the added alkaline earth metal chloride and all soluble chlorides formed, including silver chloride, which becomes dissolved in the resulting salt solution. The residual lead chloride may be used either as it is left, or after recrystallisation (following filtration) from a boiling alkaline earth metal chloride solution, the mother-liquor being used repeatedly for successive recrystallisations. The purity of the crystals depends upon the strength of the liquor, calcium (or other) chloride being deposited with the lead chloride; but this impurity is soluble in water, and may thus be removed. The presence of zinc chloride in the solution is advantageous, as it renders the lead chloride less soluble, the maximum of insolubility being attained when the solution contains 12 per cent. of the former compound. There are 15 claims.—W. G. M.

Alloy of Copper and Iron, Manufacture of an. A. F. M. V. Baron. Eng. Pat. 4202, Feb. 16, 1897.

A copper-iron alloy of any desired composition is made by heating the copper in a plumbago pot to a temperature varying between a cherry- and a very bright red heat. A mixture of two-thirds of oxalic acid and one-third of resin is then added, in the proportion of (say) 50 grms. of mixture per 100 kilos. of alloy. When the copper is melted the iron is added. Other organic acids which evolve carbon monoxide and carbon dioxide on heating, may be substituted for the oxalic acid. A comparatively soft alloy would contain 5 per cent. of Fe; as the iron is increased up to 50 per cent. the alloy becomes harder. These alloys may be rolled or drawn with a facility which depends upon the proportion of iron.—W. G. M.

Copper, Obtainment of, from Ores and other Copper-bearing Substances; An Impt. in. W. Noad and W. Agate, both of London. Eng. Pat. 8873, April 7, 1897.

The crushed ore is agitated with a (5 per cent.) ferric chloride solution until the copper is dissolved. The liquid is then run into a second vessel, in which is a porous compartment containing sodium chloride solution and a zinc or other electro-positive metal electrode; in the outer vessel is a copper or other electro-negative electrode, to complete an electric circuit. When the deposition is completed the solution is returned to the first vessel to treat another batch of ore. Sulphide ores must be roasted prior to treatment.
—W. G. M.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(A.)—ELECTRO-CHEMISTRY.

Ferric Chloride Battery. F. W. Küster. Zeits. f. Elektrochem. 3, 1897, [17], 383—385.

This is a criticism of a recent paper (see this Journal, 1897, 245), in which H. Pauling recommends a form of battery wherein iron, carbon, and ferric chloride are used, and in which the following reactions are said to take place when the battery is producing a current:—

$$3FeCl_3 = 3FeCl_2 + 3Cl.$$
$$Fe + 3Cl = FeCl_3.$$

Küster says it is *a priori* improbable that ferric chloride should be formed at the anode and should then give up part of its chlorine at the cathode; and he adduces an experiment to show that only ferrous (and not ferric) chloride is produced when the cell is in action. He further criticises Pauling's sanguine estimate of the economical working of the cell. Such iron as would be used in making these cells always contains finely divided carbon or iron carbide. One would therefore expect local action to be set up between these particles and the iron in which they are embedded. The following experiment shows that such wasteful action does occur:—A cell consisting of a carbon plate, a clean iron plate of 40 sq. cm. surface, and a 50 per cent. solution of ferric chloride was closed through a galvanometer for 40 minutes. The galvanometer showed that during this time 0·297 ampère-hour had flowed through it. On the assumption that it forms ferric chloride, the corresponding amount of iron dissolved should be 0·207 grm.; or 0·310 grm., on the assumption that it forms ferrous chloride. As a matter of fact, the iron plate was found to have lost 1·66 grm. in weight. Thus only one-sixth of the iron dissolved was usefully employed in producing current. And when simply left to stand by itself in the (partially reduced) ferric chloride solution for another 40 minutes, the iron plate suffered a further loss in weight of 1·2 grm. In Pauling's battery the iron is used as the containing vessel; and it would probably come to grief before long, even on open circuit.
—D. E. J.

PATENTS.

Electric Batteries [Using Air, &c.] and Appliances connected therewith, Improved. L. B. Atkinson, Penarth, and F. G. Treharne, Llanishen. Eng. Pat. 8906, April 28, 1896.

The object is the production of electrical energy from coal, coke, or other fuel, or from hydrocarbons, solid, liquid, or gaseous. The inventors say: "Broadly, our invention is as follows. We form a battery or cell having electrodes [metals, or oxides of metals; fused or moving electrodes] which have a different affinity for oxygen: we place these in an electrolyte [one or more, or mixed: solutions of salts, or fused salts, as hydrates, nitrates, chlorates, phosphates, borates, or oxides (lead, &c.); porous partitions may be employed] capable of transferring oxygen. The electro-negative element is oxidised by means of atmospheric air, either directly, or indirectly by means of a chemical transfer. The electrodes now being connected by a conductor, an electric current is set up, the electro-negative element loses oxygen, the electro-positive element is oxidised. To maintain the action, the electro-negative electrode is oxidised at the expense of atmospheric oxygen, the oxidised portion

of the electro-positive electrode is deprived of oxygen or reduced by carbon monoxide, hydrogen, hydrocarbon gas, or mixed producer gases derived from carbonaceous or hydrocarbon fuel."—J. C. R.

Electrolytic Decomposition of Liquids [Alkali Chlorides, &c.], Impts. in or connected with Apparatus for the. G. and G. W. Bell, Liverpool. Eng. Pat. 11,133, May 22, 1896.

The improvements relate to "covered-in or hollow anodes, that is, anodes having a cover or roof above, by which all the gas liberated at the surface under such roof or cover is collected, and in which the gas so collected is conveyed away by suitable conduits." Apparatus for the decomposition of liquids (alkali chlorides, &c.), with the cathode chamber directly above the anode chamber and a diaphragm separating the two, is fully described and shown. Eng. Pat. 20,540 of 1895 (this Journal, 1896, 908) is referred to.—J. C. R.

Electrodes [Detachable] for Electrolytic Purposes, Impts. in. E. A. G. Street, Paris. Eng. Pat. 11,338, May 23, 1896.

"A cathode for use in the electrolysing of substances producing a solid deposit on the negative electrode, the characteristic feature of this cathode being that after the deposit having been effected thereon, it can be reduced in size—the object being to enable the deposit to form a film or layer which can be very easily detached; the said cathode being for this purpose formed by a thin sheet, preferably of metal, wound one or several times upon itself; the reduction of the cathode in size being subsequently obtained, after the deposit has been formed thereon, by winding the sheet constituting the cathode upon itself."—J. C. R.

Anodes [Carbon-Platinum] for Electrolytic Purposes, Impts. in. P. Jensen, London. From H. C. F. Störmer, Christiania, Norway. Eng. Pat. 14,393, June 29, 1896.

The claim is for "carbon anodes for electrolytic purposes, coated or covered with foil of platinum or other metal which is not acted upon by the electrolyte."—J. C. R.

Secondary Electric Batteries, Impts. in. [Reticulated Frame.] M. O. A. Garreau, Paris, France. Eng. Pat. 16,270, July 22, 1896.

The electrodes are composed of a reticulated frame formed of an alloy of antimony and lead, the rectangles of which are filled with flat or corrugated bands or strips of lead of suitable length, the edges of which are connected midway of their length and held in place by an autogenous solder, so as to limit their expansion to the direction of their length. Each plate is provided with long legs or supports of insulating material which is unaffected by the electrolyte, and they are wrapped in parchment paper to secure the active material. When set up in the cell, the plates are pressed closely against each other.—G. H. R.

Pasted Plates for Accumulator Batteries, Impts. in. [Helically wound Cores.] J. and H. H. S. Vaughan-Sherrin, Ramsgate. Eng. Pat. 16,516, July 25, 1896.

Each element is in the form of a bar, and consists of a solid or tubular core of ebonite or other suitable non-conducting material, round which is wound helically a lead strip, which may be crimped or plain. Round this conducting strip, and in the opposite direction, is wound an insulating helix of ebonite or the like, and the active material is pressed into the interstices between the parts. The non-conducting core may be dispensed with, and the elements be formed of a cruciform, straight, or twisted lead conductor, round which is wound an insulating helix, the interspaces being filled with active material as before. An inner springy insulating helix may be wound between the core and the conductor, or between the coils of the latter. The electrodes may consist of one or more of the elements suitably connected up.—G. H. R.

Ozone or Ozonised Air, Impts. in Apparatus for the Production of. A. Verley, Paris, France. Eng. Pat. 17,228, Aug. 4, 1896.

The apparatus consists of a sheet of polished copper or the like and a sheet of glass or equivalent material silvered on the side furthest from the metal sheet, the two sheets being separated by distance pieces capable of adjustment, and so arranged that the air in passing between them to the outlet at the centre is forced to take a spiral or other circuitous course, during which it is ozonised. The two metal sheets are respectively connected to the poles of a source of electric supply, and means are provided for cooling the apparatus, either by a stream of water or of other liquid passing over the surface of a slate table on which the copper sheet rests, or by the expansion of compressed air or other gas.
—G. H. R.

Electric Accumulators, Active Material for the Plates [Pyridine, &c.] or Electrodes of; The Manufacture of an Improved. S. Hammacher, Berlin. Eng. Pat. 17,161, Aug. 4, 1896.

The claims are for the manufacture and use of active material for the electrodes of accumulators by mixing an oxide or oxides of lead [minium or red lead or litharge] with substances or bodies of the pyridine group [pure pyridine or crude, as in bone- or animal-oil] in the presence of water, whereby the said oxides of lead are solidly bound together, so that after drying they may be subjected to the "forming" process.—J. C. R.

Electric Accumulators or Storage Batteries [Felt between Electrodes], Impts. in. D. Young, London. From La Société Anonyme "Accumulateur Eclair," Brussels. Eng. Pat. 22,069, Oct. 5, 1896.

"The manufacture of an electric accumulator in which the active material or paste is scraped off above the surface of the grids, and the plates or electrodes so formed are placed one against another, with a sheet of felt or analogous material intercalated between them . . . . ."—J. C. R.

Plates (Planté) for Secondary Batteries or Accumulators, Impts. in or relating to. [Lead Ribbon.] H. T. Cheswright, Paris, France. Eng. Pat. 9563, April 14, 1897.

The plate consists of a series of lead strips or ribbons, plain, corrugated, or embossed, arranged in groups, and depending from a conducting bar, the lower ends being left free. To prevent any chance of short-circuiting between the plates, these are enclosed in a framework, bands, cage, or perforated envelope of insulating material.—G. H. R.

Electrodes for Electric Accumulators, A Manufacture of. [Use of Butyrate or Lactate Paste.] E. Marckwald, Berlin, Germany. Eng. Pat. 9913, April 20, 1897.

The plates consist of frames or moulds filled with a paste made of a basic lead salt formed by stirring into a 10 to 12 per cent. aqueous solution of butyric acid, 5 to 7 times the percentage of finely powdered lead oxide. When the plates have hardened, they are formed in dilute sulphuric acid in the usual way. Instead of butyric acid, the same proportion of lactic acid or of the two acids may be employed.
—G. H. R.

Electrodes for Electric Accumulators, A Manufacture of. [Use of Milk Paste.] E. Marckwald, Berlin, Germany. Eng. Pat. 9914, April 20, 1897.

The electrodes consist of gratings, frames, or moulds filled with a paste made by mixing 1 kilo. of lead oxide or of red lead with about 200 c.c. of milk. The hardening of the paste may be accelerated by careful warming, and the formation of the plates is then completed in the usual way.
—G. H. R.

Accumulator Plates, An Improved Process for the Manufacture of. W. A. Boese, Berlin. Eng. Pat. 10,254, April 24, 1897.

1. "A process for the manufacture of accumulator plates, which consists in causing red lead to be reacted on within

a frame by an organic or inorganic acid which combines with red lead or which decomposes the red lead, forming an insoluble or hardly soluble lead salt, with or without the use of a binding material, in order to produce a mutual wedging of the particles of the mass in the frame by the resultant expansion of the reaction product."

2. "A process for the manufacture of accumulator plates, which consists in mixing red lead with acetic acid and a binding material soluble in the latter, such as asphalt or catechin, and allowing it to react in a frame divided into one or more parts."

3. "A process for the manufacture of accumulator plates, which consists in dry lead formed into plates being inserted in sulphuric acid, or an acid having a similar reaction, and allowing it to stand therein uninterruptedly, the acid consumed being replaced until the said plates are sufficiently hard."

Eng. Pats. 11,532 of 1892 and 4610 of 1895 are referred to (this Journal, 1893, 527, and 1895, 757 respectively).
—J. C. R.

(B.)—ELECTRO-METALLURGY.

Alloys, Electrolytic Methods of preparing Metallic. J. Walter. Zeits. f. Elektrochem. **3**, 1897, [17], 385—388.

Suppose the object is to prepare an alloy of a heavy metal, such as lead, with a light metal, such as sodium. The most obvious method is to use fused lead at the bottom of a crucible as the negative electrode: on this is placed the fused salt, into which the positive electrode (carbon) dips. The process goes on satisfactorily at first; but, after a while, globules of sodium rise to the surface in increasing quantity, and then burn or unite with the chlorine. Furthermore, after the crucible has cooled, it is found that at the bottom we have nearly pure lead, and just below the fused salt an alloy which is very rich in sodium. The alloy, being very light, does not mix readily with the bulk of the lead. The author schedules a number of methods for overcoming this difficulty. The methods depend upon one or other of the following principles:—I. Mechanical stirring of the metal which forms the negative pole. II. The use, as negative electrode, of a fluid (mercury) or fused metal, which is allowed to trickle through the electrolyte. III. In some cases the temperature of the electrolyte (fluid or fused) may be kept below that of the metal to be alloyed, but above that of the alloy; the metal may then be suspended as the negative electrode in the liquid electrolyte. The alloy as formed, melts away and a fresh surface of the metal is continually exposed. IV. In other cases the salts of two metals which are to form the alloy can be arranged in separate layers; the negative pole can be raised or lowered so as to dip alternately into the one layer or the other, while the positive pole makes contact with both. Or temporary changes can be made in the composition of the fused electrolyte; *e.g.*, if the alloy contain an insufficient proportion of a certain metal, a salt of that metal can be brought into contact with the cathode for a time. Among the applications of these methods described, the following may be given as examples:—A. Preparation of the alloy mentioned at the beginning of this abstract. The fused lead is stirred up by passing through it hydrogen or nitrogen (from a tube provided with minute apertures), so that the alkali metal meets fresh surfaces and forms a uniform alloy with the lead. B. Preparation of an alloy of aluminium and tin from the double chloride of aluminium and sodium. C. Preparation of silicon-bronze by electrolysis of water-glass. D. Preparation of aluminium-bronze by electrolysis of cryolite and copper chloride.—D. E. J.

XII.—FATS, OILS, AND SOAP.

Cholesterin Bromides. C. Cloez. Comptes Rend. **124**, 864—866.

When bromine is added to cholesterin, both in carbon bisulphide solution, the body $C_{26}H_{44}OBr_2$ is obtained; but if to 5 grms. of cholesterin in 50 c.c. of carbon bisulphide a solution of 2 c.c. of bromine in 10 c.c. of carbon bisulphide be gradually added, small crystalline needles form abundantly when 2 c.c. of the bromine solution have been added, and redissolve as more is added, disappearing completely when 4·3 c.c. (the amount needed to convert all the cholesterin into dibromide) is reached.

This suggests the formation of a mono-bromide; but in point of fact, the compound is probably a molecular combination of cholesterin with the dibromide—

$$C_{26}H_{44}O + C_{26}H_{44}OBr_2$$

Solvents could not be found to separate the new body into these two substances; but support is given to this view of its constitution from the fact that it is easily obtained by mixing carbon bisulphide solutions of cholesterin and its dibromide in molecular proportions.—J. T. D.

Castor Oil, Chemistry of. H. Meyer. Pharm. Zeit. **42**, 326.

See under XX., *page* 632.

Stearin in Spermaceti, A Simple Test to Detect. E. Hirschsohn. Pharm. Central-H. **38**, 283.

See under XXIII., *page* 639.

PATENTS.

Spent Lyes, An Improved Process for the Purification of. C. H. Simpson and F. H. T. Allan, Wakefield. Eng. Pat. 16,129, July 21, 1896.

The improvement claimed is the treatment of spent lyes with electricity. The lyes are neutralised, although this is not absolutely essential, and a current of electricity is passed through the liquid, an iron cathode and anode being used. The strength of the current should be not less than $2\frac{1}{2}$ volts. By this treatment the lower and less stable compounds of sulphur and oxygen are oxidised, and the arsenic and organic impurities are precipitated. The result is a very pure form of lyes suitable for concentration and distillation for the manufacture of pure glycerin.—W. P. S.

Liquid Soap, A New or Improved. T. H. Bateman, London. Eng. Pat. 24,090, Oct. 29, 1896.

This consists of a solution of soft soap in spirits of rosemary and lavender oil, with the addition of glycerin.

120 parts of soap are digested in 30 parts of a solution composed of 20 parts of spirits of rosemary and 1 part of lavender oil. After filtration, about 10 per cent. of glycerin is added to the compound.—W. P. S.

Glycerin, Improved Method of Concentrating. E. A. Ruch, Paris. Eng. Pat. 1355, Jan. 18, 1897. (Under Internat. Convention, First Foreign Appl., July 18, 1896.)

This method is one for distilling glycerin liquors under pressure. The liquors, clarified and filtered as usual, are introduced into a copper digester of any suitable shape, and provided internally with a coil, through which a current of steam circulates. The contents of the digester are quickly raised to boiling point at the desired pressure, which should not be less than 6 kilos. above the pressure of the atmosphere.—W. P. S.

Oil from Peat, A Process for the Production of. F. Rapp-Rosenthal, Berlin. Eng. Pat. 9314, April 12, 1897.

This process has for its object the production of an oil from peat, and consists in boiling the peat for about an hour, if necessary with the addition of water, and then pressing it in bags. The resulting liquid is allowed to stand until an oil separates, which oil is then drawn off from the extract. The oil may be used as an embrocation.—W. P. S.

XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

(A.)—PIGMENTS, PAINTS, &c.

PATENTS.

Drying White Lead, Colours, and other Powdery Granular or Nodular Substances; Impts. in Apparatus for. T. C. Sanderson, Norwich. Eng. Pat. 7868, April 14, 1896.

The apparatus consists of a long closed heating chamber containing a travelling band or a series of trays for carrying the material through the apparatus. Heating pipes are arranged in side chambers, communicating with the main chamber at top and bottom, so as to heat and circulate the air. The material is fed into the apparatus through steam-heated rollers, and when the dust is noxious, the dried material is discharged ioto a hopper in connection with a dust chamber and exhaust fan.—O. H.

White Lead [Dutch], Impt. in the Manufacture of. A. H. Sandiland, London. Eng. Pat. 10,462, May 15, 1896.

Since "Dutch" white lead as usually prepared contains a certain amount of soluble lead salts, this portion of the substance dissolves and is wasted during the washing and grinding in water. To prevent this loss, the inventor adds 7 oz. of an alkali carbonate or hydrate, or a mixture of the two, to every 20 galls. of the water employed in the treatment of the material; claiming by this device to obtain an increase of 0·5 per cent. in the yield.—F. H. L.

White Lead [Dutch], Impts. in the Manufacture of. H. C. Webster and R. Crawford, Glasgow. Eng. Pat. 14,428, June 30, 1896.

Some or all of the earthenware pots usually employed in the manufacture of white lead by the "Dutch" process are replaced by similar vessels made of lead, in order to avoid loss by breakage, &c.—F. H. L.

[Coloured] Zinc Pigments, Impts. in the Manufacture of. A. M. Clark, London. From W. Hampe and C. Schuabel, Clausthal, Hanover. Eng. Pat. 17,031, July 31, 1896.

These pigments are prepared by intimately mixing together zinc sulphate with 6 to 30 per cent. of the sulphate of some other metal (or metals) which yield a coloured oxide—*e.g.*, nickel, cobalt, manganese, and iron—drying the whole, adding finely-divided carbon, and igniting carefully at 650° C. In the manufacture of the more delicately tinted samples, the zinc sulphate should be purified by stirring zinc oxide into its solution, and passing a current of chlorine through it, by which process the iron, cobalt, and manganese present as impurities are thrown down.—F. H. L.

(B.)—RESINS, VARNISHES.

PATENT.

Treatment of Wood, Paper Fabrics, Fibres, and like Materials, to render them Impervious and Inalterable by Moisture and Climate and other Influences; Impts. in the. S. Bennett, London. Eng. Pat. 12,695, June 9, 1896.

After being thoroughly dried, the materials to be treated are first coated with a mixture of equal quantities of castor or linseed oil and turpentine, and then with 1 part of nitrocellulose, 2 parts of turpentine, and 3 of ether-alcohol or amyl acetate. Or the two operations may be combined by adding 5 to 20 per cent. of nitrocellulose in a state of solution to the oils selected.—F. H. L.

(C.)—INDIA-RUBBER, &c.

PATENT.

Treatment of India-rubber, Gutta-percha, and their Compounds; Impts. in the. E. Hornung and S. Hansel, Vienna. Eng. Pat. 9464, April 13, 1897.

As vulcanised india-rubber and gutta-percha, if not freed from excess of sulphur, gradually become hard and brittle on exposure to air or high temperatures, the inventors prefer to precipitate gelatin from its aqueous solution, mix it with 10 to 50 per cent. of "vulcanised oil," dry the whole, and incorporate it with the rubber; the proportions employed depending on the quality of the rubber, and also on the filling materials contained in the mass.—F. H. L.

XIV.—TANNING, LEATHER, GLUE, SIZE.

Fermentation Phenomena in Tan Liquors. F. Andreasch. Imp. Research Laboratory, Vienna. Der Gerber, **527**, 186; **534**, 275.

The preceding research has shown that the tannins have no direct antiseptic action. The carbon required by the micro-organisms is drawn from the soluble non-tannins. The yeasts draw their supply of carbon from the carbohydrates, and cease to grow as soon as these are used up For the bacteria, the supply of N is of more importance than C.

The amount of non-tannin in a tanning material is a fairly exact measure of the quantity of acid it will produce, In an unused tan liquor only alcohol and acetic acid are usually produced, lactic organisms not being able to develop for want of sufficient N.

In order to ascertain the exact measure of fermentable substances, the author conducted a series of experiments on nine different materials, the results of which are given in Table I. The materials employed contained an average amount of tannin, non-tannin, and sugars as compared with the mean of a large number of samples.

N was added in the form of mineral salts, so as to introduce no fresh carbon; but, as a matter of fact, peptones and similar albuminous bodies furnish no carbon to the bacteria.

The organisms used were: (1) A Saccharomyces; (2) a lactic bacillus; (3) a lactic yeast. The alcohol was estimated by Duclaux's physical method (Comptes Rend. **70**). Owing to the many ethers produced, it gives slightly erroneous results; but if the amount of alcohol be compared with the percentage of sugars, the results are concordant enough. The sugars and lactic acid were estimated as before described (this Journal, 1897, 248 and 249).

It will be seen that the whole of the sugar is decomposed by alcoholic fermentation, and that the lactic bacteria can transform almost all the non-tannin into lactic acid. These results hold good for all materials examined by the author. The dextrins and soluble starches of some materials (pine and hemlock barks, canaigre) are as important a source of carbon for bacteria as the sugars are for the yeasts.

In practice, the fermentation takes place in two stages: (1) all the sugars are fermented by yeasts; (2) fresh hides coming into the partly decomposed liquor, supply the lactic and other bacteria with nitrogen, and enable them to continue the production of acid from other carbon compounds than the yeasts can utilise.

In accordance with the table, p. 621, the materials examined may be arranged in order of acid-producing value, as follows:—1. Pine bark. 2. Sumac and oak bark. 3. Myrabolams. 4. Valonia and hemlock bark. 5. Oak wood. 6. Knoppern and quebracho wood. Knoppern liquors are remarkable for a short, quick fermentation, owing to the presence of an easily fermentable sugar. Glucose may be added with advantage to such materials as quebracho, but addition of fresh pine bark produces a quicker fermentation, owing to the fact that suitable yeasts exist on the bark, which cause a natural fermentation, like that of wines. Alcohol has been added with success to the liquors of an Italian sole-leather tannery when the acidity was insufficient. The author has investigated the action of addition of glucose, alcohol, and pine bark to tan liquors; the results are shown in Table II.

The research, so far, may be summed up as follows:—

1. In the alcoholic and acid fermentation of tan liquors, the carbon is derived from the non-tannin, in some cases the whole of it being used up.

2. The tannin is *not* decomposed by the micro-organisms taking part in the fermentation.

TABLE I.

Time of Fermentation, 14 Days. Temperature: 25° C., Alcoholic Fermentation; 30° C., Lactic Fermentation.

Name of Tanning Material.	Original Tanning Material contains in per Cent.			Fermentation I. Grms. per 100 c.c.					
				Original Liquor.			After Fermentation with		
							Bottom-Fermentation Yeast from Tan Liquors.	Lactic Bacterium from Tan Liquors.	Lactic Yeast from Tan Liquors.
	Tannin.	Non-Tannin.	Sugar, calculated as Glucose.	Tannin.	Non-Tannin.	Sugar, calculated as Glucose.	Alcohol.	Lactic Acid.	
Valonia	28·14	15·22	2·88	1·8000	0·9736	0·1843	0·0876	0·7936	0·1564
Knoppern	28·82	8·65	0·70	1·8000	0·5402	0·0437	0·0206	0·5138	0·0360
Myrabolams	30·23	15·66	6·35	1·8000	0·9325	0·3781	0·1786	0·8006	0·3334
Sumac (Sicilian)	23·24	19·95	5·93	1·8000	1·5450	0·4593	0·1788	0·8200	0·4000
Oak bark	9·88	7·21	2·74	1·8000	1·3137	0·4993	0·2518	0·7990	0·4244
Pine bark	12·08	12·52	4·16	1·8000	1·8656	0·6199	0·2950	0·8123	0·5270
Hemlock bark	10·71	7·46	1·00	1·8000	1·2538	0·1681	0·0773	0·8044	0·1400
Quebracho wood	19·88	1·58	0·30	1·8000	0·1409	0·0270	0·0117	0·1366	0·0231
Oak wood	6·07	2·94	0·46	1·8000	0·8720	0·1365	0·0593	0·7883	0·1152

Name of Tanning Material.	Fermentation II. Grms. per 100 c.c.						Fermentation IIA. Grms. per 100 c.c.					
	Original Liquor.			After Fermentation with			Original Liquor.			After Fermentation with		
				Bottom-Fermentation Yeast from Tan Liquors.	Lactic Bacterium from Tan Liquors.	Lactic Yeast from Tan Liquors.				Bottom-Fermentation Yeast from Tan Liquors.	Lactic Bacterium from Tan-Liquors.	Lactic Yeast from Tan Liquors.
	Tannin.	Non-Tannin.	Sugar, calculated as Glucose.	Alcohol.	Lactic Acid.		Tannin.	Non-Tannin.	Sugar, calculated as Glucose.	Alcohol.	Lactic Acid.	
Valonia	1·8484	1·000	0·1892	0·0900	0·8000	0·1600	0·9242	0·5000	0·0946	0·0469	0·4855	0·0804
Knoppern	3·3310	1·000	0·0809	0·0382	0·7915	0·0687	0·6655	0·5000	0·0191	0·0200	0·4752	0·0162
Myrabolams	1·9304	1·000	0·4055	0·1916	0·8216	0·3447	0·9652	0·5000	0·2028	0·0988	0·4463	0·1700
Sumac (Sicilian)	1·1650	1·000	0·2972	0·1158	0·8254	0·2506	0·5825	0·5000	0·1485	0·0610	0·4811	0·1263
Oak bark	1·3710	1·000	0·3800	0·1914	0·7990	0·3185	0·6855	0·5000	0·1900	0·0977	0·4764	0·1600
Pine bark	0·9651	1·000	0·3323	0·1582	0·8003	0·2714	0·4826	0·5000	0·1662	0·0843	0·4844	0·1413
Hemlock bark	1·4570	1·000	0·1408	0·0617	0·7913	0·1205	0·7185	0·5000	0·0704	0·0333	0·4884	0·0601
Quebracho wood	..	..	..	..	..	..	3·1955	0·2500*	0·0475	0·0200	0·2431	0·0404
Oak wood	2·0609	1·000	0·1563	0·0680	0·8000	0·1329	1·0304	0·5000	0·0781	0·0411	0·4685	0·0664

* The concentration of the Quebracho liquor was made half the strength of the other liquors, so as to avoid too great an amount of tannin.

TABLE II.

	Grms. per 100 c.c.	Original Liquor.	Acids after Eight Days. Temperature, 30° C.					
			No Addition.	Addition of 4 grms. Absolute Alcohol.	Addition of 8 grms. Glucose.	Addition of 8 grms. Glucose and Inoculation with Alcoholic Yeast from Pine Liquors.	Addition of 25 grms. Pine Bark = 1·04 grms. Glucose.	Addition of 20 grms. Pine-Bark Extract = 1·355 grms. Glucose, and Inoculation with Alcoholic Yeast from Pine Liquors.
Sour pine liquor, eight days old	Acetic acid	0·1719	0·2261	1·2501	0·5185	2·1730	0·7105	0·9065
	Lactic „	0·2742	0·4212	..	..	..	..	..
Sour pine liquor, 15 days old	Acetic „	0·1501	0·1230	1·2870	0·2355	2·2070	0·6422	0·8277
	Lactic „	0·1770	0·1755	..	..	..	..	..
Pine liquor from layer pits, four months old	Acetic „	0·2475	0·2398	1·1056	0·6032	2·6535	0·7800	0·8733
	Lactic „	Traces	Traces	..	..	..	..	..
Quebracho liquor	Acetic „	0·0842	0·0976	1·1472	0·4225	1·9738	0·5331	0·7473
	Lactic „	0·1005	0·1321	..	..	..	..	..

—J. T. W.

Hübl's Iodine Solution, The Behaviour of Tannic Acids and Tannic Extracts towards. C. Boettinger. Chem. Zeit. **21**, [16], 450.

See under XXIII., *page* 640.

PATENTS.

Soluble Colloids of the Gelatine Class, Impts. in Formation and Preparation of. E. J. Mills, Glasgow. Eng. Pat. 17,536, Aug. 8, 1896.

This comprises modifications of or improvements on the process described in Eng. Pat. 8847 of 1895 (this Journal, 1896, 462) for converting gelatin, or a gelatin-containing, or gelatigenous substance, into a compound permanently soluble in cold water.

One method is to take 100 parts of glue, preferably comminuted, 5 parts of slaked lime, and 100 parts of water. These are stirred together and allowed to remain at the ordinary temperature until perfectly soluble in cold water. With glue of good quality and moderate hardness, the conversion takes place in a week or ten days.

An alternative method is to dissolve the glue in water with the aid of heat, and allow the solution to cool and become a jelly. The comminuted jelly is mixed thoroughly with the slaked lime. In the course of an hour or more, perfect liquefaction will have been attained.

Instead of caustic lime, other caustic alkali earths may be employed, or caustic alkalis or their carbonates.

—L. J. de W.

Tanning of Hides and Skins, Impts. in the Preparation for and. E. O. Daniel, London. Eng. Pat. 11,111, May 4, 1897.

In the process of preparing hides and skins for tanning, a revolving drum is used having an entrance and exit for liquid, and the continuous passage therethrough of liquid in which the hides or skins are treated previous to going through the "bating" process; and the internal periphery of the drum may be fitted with stays or struts, or backwardly curved pegs or projections, or with pegs and struts combined, to assist in keeping the hides or skins separated from each other.

Another portion of the invention relates to the method of application of the tanning solution. The hides or skins are allowed to soak in a comparatively dilute tanning solution in suitable vats or pits, and the solution is gradually increased in strength; or the hides or skins are put into a stronger solution or gradually stronger solutions. Up to this point there is no novelty in the treatment; but the skins are next placed in a drum containing a strong tanning solution, which, when revolved, brings the solution into contact with every part of the hides or skins, by which greatly improved results are obtained.—L. J. de W.

XV.—MANURES, Etc.

PATENT.

Fertilised and Fertilising Materials, Manufacture of Improved. C. H. Thompson, Teignmouth. Eng. Pat. 9704, April 15, 1897.

In the manufacture of an improved fertiliser of the kind described in Eng. Pat. 20,971, 1894 (this Journal, 1894, 1246), to serve as a substitute for earth, in which plants may be grown without earth, or raised from seeds, or struck as cuttings and brought to maturity quicker than in earth, or to be used as "mulching" or manure—peat-moss is used, or other suitable fibrous or spongy material, impregnated with a "solution" of soot, gypsum, bone-meal, or the chemical or functional equivalents of these substances, phosphoric acid, potash (pearlash), and nitrate of soda. After boiling, the fibre is removed, dried, and fermented, so as to cause a complete chemical interchange of the ingredients.

The liquor is also fermented with yeast or other ferments, after the addition of saccharine matter, to produce a concentrated liquid fertiliser.—L. J. de W.

XVI.—SUGAR, STARCH, GUM, Etc.

Synthesis of Cane-Sugar. L. Marchlewski. Rocznik Akad. Umiej. Krakowskiej. 1996.

Cane-sugar may be obtained by the action of acetochlorhydrose upon the potassium salt of *d*-fructose. The reaction is expressed by the following equation:—

$$\begin{array}{ll} \begin{array}{l} CH_2.OCOCH_3 \\ \quad | \\ (CH.OCOCH_3)_3 \\ \quad | \\ O\!<\!\begin{array}{l} CH \\ | \\ CH{-}Cl \end{array} \end{array} + \begin{array}{l} CO_2.OH \\ \quad | \\ (CH.OH)_3 \\ \quad | \\ O\!<\!\begin{array}{l} COK \\ | \end{array} \end{array} & + 4C_2H_5OH = \begin{array}{r} KCl + \\ 4CH_3COOC_2H_5 \\ + C_{12}H_{22}O_{11} \end{array} \end{array}$$

Pure acetochlorhydrose is dissolved in alcohol, and to the solution freshly prepared sodium levulosate is added. The mixture is left to stand for about seven days at ordinary temperature; to complete the reaction it is heated for half an hour on the water-bath; next the potassium chloride formed is filtered off, the filtrate evaporated at 80° C., and the residue dissolved in boiling water. The solution obtained is next treated with a solution of $Ca(OH)_2$, and the gradually formed precipitate filtered off, stirred in some water, and decomposed with CO_2. The $CaCO_3$ obtained is filtered off, and the filtrate purified by means of $Ca(OH)_2$ in a similar manner. Finally, the aqueous solution of the sugar is extracted with ether in order to remove the saccharin, and then evaporated *in vacuo*. After some days' standing, the cane-sugar formed, crystallises in the well-known forms. All the reactions and physical properties agree with those of natural cane-sugar.—L. M.

Juice and Liquor [Beetroot], Filtration of. L. Beaudet. Bull. Assoc. Chim. 1897, **14**, 967—969.

Most sugar manufacturers are convinced of the necessity of filtering the juice carefully after each purification, but in some factories this is only incompletely carried out, either for want of space or on account of cost. The author thinks that no expense should be spared to effect this object, for fear of redissolving precipitates already formed, through alteration of temperature or alkalinity.

Contradictory results were obtained on examining liquors which were produced from diffusion juice filtered before carbonating, but this failure is attributed to the filtration having been too slow.

The filtration of the juice in filter presses is effected either with or without decantation. Decantation has the disadvantage of cooling the juice, and is thus one cause of the formation of calcium salts. As soon as a precipitate is formed, it should be separated as rapidly and completely as possible, and the juice should be exposed as little as possible to the air. This condition is far from being realised in the case of decantation.

One of the principal difficulties in filtration arises from incomplete carbonating, or from the juice being allowed to cool after the first carbonation. Another cause is the use of well- or river-water for washing, instead of distilled water, the former rapidly obstructing the pores of the cloths, through deposits which are formed.—L. J. de W.

Juice [Beetroot], Filtration of. L. Beaudet. Bull. Assoc. Chim. 1897, **14**, 973—977.

The filtration of the juice should be applied at the following stages:—(*a.*) After the filter presses from the first carbonating. This operation is indispensable; for, whatever care may be exercised at the presses, the juice will still contain precipitates in suspension. The juice of the first should be completely freed from precipitate before going to the second, or the whole principle of double carbonating is disregarded. (*b.*) To the filtration of the juice of the second carbonating previously passed to the filter press. (*c.*) To the filtration of the juice of the third carbonating. (*d.*) To the liquors between the second and third effects. This, although not indispensable, is to be recommended. (*e.*) To the filtration of liquors from the multiple effect.—L. J. de W.

Diffusion Juice, Conditions of Inversion of Sugar in. Acidity of Beetroot and Diffusion Juice. H. Pellet. Bull. Assoc. Chim. 1897, **14**, 1038—1040.

Jesser concludes (this Journal, 1897, 451) that diffusion juice possesses an invertive power, and that this power varies from year to year with the temperature to which the juice is heated, the method of culture, and the soil on which the roots are grown, &c. Consequently, from the point of view of diffusion, the greatest attention should be paid to every circumstance which may increase the invertive power, and extremes should be avoided. The author, however, sees no disadvantage in strongly heating a juice if the duration of diffusion is thereby lessened, or if the practical conditions are otherwise favourable. The best guide is to follow the quality of juice obtained by different methods of working, examining not merely the raw diffusion juice, but every stage down to the low products. It is not always diffusion juices of greatest purity which give massecuites easy to work and the highest yield. It is only after purification by lime and carbonic acid that we can have an indication of this by chemical analysis and the physical properties of the massecuites.

It is not acidity alone that determines the invertive power of a diffusion juice, but it is probably the result of all the foreign matters acting on the sugar. In diffusion juice from the sugar cane, much higher acidities are found than in that from beetroots, and no formation of invert sugar results. This has been verified the last few years in several factories in Java, Guadeloupe, and Egypt.

According to Pannenko (Bull. Assoc. Chim. 1897, 829), diffusion juice has a higher acidity than that of the beet slices from which it is produced. This does not appear to be the fact. On reducing 100 grms. of cossettes into pulp suitable for instantaneous cold aqueous digestion, and making up the liquid to a litre, it will be found to be of the same acidity as diffusion juice of the same dilution, the total acidity being calculated to 100 grms. of sugar, and not to 100° Brix. In actual work the varying character of the water used may influence the acidity very considerably. Beetroots also are not always equally clean, and if the earth adhering to them be of a calcareous character, the acidity of the juice may be affected. Unless all these conditions are taken into account, it is not possible to conclude with certainty that there is formation or destruction of acidity in the diffusion process.—L. J. de W.

Potato Starch and Potato Flour. Saare. Papier Zeit. 1897, **30**, 1059.

Potato starch, of which 200,000 to 300,000 tons are annually produced in Germany, is sold in two qualities. The best, termed "superior," is singularly white, glossy, and pure, and consists of large grains, being the first to deposit in the settling tanks. The deposits are lower in quality in succession, until only a brown powder is deposited, known as slime sand.

Potato flour is made by grinding and sifting the dried potato starch. If dried on frames in a state of repose, it is known as "hurdle" starch, or if dried on endless webs kept in motion, it gets small-grained, and is known as "machine" starch.

The best quality of starch is used for the manufacture of starch sugar and dextrin. A soluble starch has been recently made, giving in water a clear colourless solution. Another recently introduced article is ozone gum and ozone starch, in which the aromatic odour is removed, rendering the product similar to wheat starch. Attempts have also been made to obtain a nitro starch as an explosive, but without much success. Some French chemists have also tried to obtain alcohol from starch.

Good starch must be free from chlorine and acids, and the moisture must not exceed 20 per cent. It must be of a pure white colour, slight shades of yellow, grey, or blue indicating insufficient washing or bad drying. Potato starch has a peculiar smell, due to volatile oils, which may be extracted by alcohol. A sour smell indicates wrong treatment in manufacture, and the starch is deteriorated as a food-stuff. The gloss of starch, which is much esteemed, depends on the size of the grains—the larger the grains the better the gloss; and the determination of the average diameter of the grains under the microscope forms a good test for starch: a superior article having an average diameter of grain of 35·5 μ ($\mu = \frac{1}{1000}$ mm.), the "Ordinary Prima" starch 32·5 μ, the ordinary or "Prima seconds," 21 μ, and the inferior kinds 17 to 12·5 μ. The condition of the grains also affects the gloss, some kinds of starch which show a tendency to agglutinate having no gloss.

Of considerable importance is the amount of impurity present, consisting of such foreign substances as coal dust, potato skin, soot, sand, iron particles from wheels, algæ from water, particles of wood, &c. The amount is ascertained by smoothing a sample on white paper, and counting the number of particles in 1 sq. cm. The superior contains 15 to 30; average starch, 27—170; while some kinds contain as many as 800 upon 1 sq. cm. These particles affect the manufactories, showing in paper as dark dots, &c. The nature of the impurities ("Stippen") may be sometimes ascertained by means of the microscope. A sample is spread smoothly out on white paper, and under the microscope the number of fine particles of foreign substances is counted in 1 sq. cm. A "Superior" starch should only contain 15 to 30, whilst "Medium Prima" may contain 27 to 170. In potato flour, hard lumps sometimes appear, due to bad drying.

If not free from acids, especially mineral, it is liable to affect colours when used as a dressing material. The use of acids in the manufacture is necessary, but they must all be removed again. Calico-printers test for acid by printing on coloured cotton a pattern with the starch paste and observing whether any change of colour takes place. To test for acids, drops of neutral Bordeaux-red litmus solution are sprinkled upon the starch. Superior starch turns the colour light blue, most starches do not affect it, slightly acid ones turn it wine red, and strongly acid ones turn it to an onion-red colour. The test for acid by titrating the alcoholic extract with decinormal alkali is not accurate, owing to the starch grains retaining some of the acid in their parenchymæ. The following is recommended:—25 grms. of starch are ground up with 25·30 c.c. water and titrated with decinormal KOH. The alkali is added by drops, until a drop of the solution placed on filter paper prepared with litmus gives the same colour as a known neutral starch. If 100 grms. of starch need 5 c.c. of $\frac{1}{10}$ normal KOH, the slight acidity should be mentioned; if 8 c.c. KOH be required, the starch is acid, and if more than 8 c.c. be required, the starch is strongly acid.

The amount of water in starch varies from 13 to 30 per cent.—S. P. E.

Starch, Control of the Manufacture of. Hébert. Bull. Assoc. Chim. 1897, **14**, 1003—1011.

The results of the extraction of starch from potatoes appear to be identical in France and Germany; yet, in spite of the efforts made in both these countries, smaller factories, in the case of what is considered "good work," still leave behind 60 to 65 per cent. of starch on the solid matter of the pulp, and the author has found pulps containing 75 per cent. The various mills brought out as improvements on the old rasping process have reduced the loss to 45 per cent., but at at the cost of a considerable amount of power. No great advance can be expected in apparatus for subdivision of the material, but fermenting the pulp for several months allows 15 to 20 per cent. of inferior starch to be recovered. The fermentation having altered some of the starch grains, this secondary starch is scarcely fit for anything except the manufacture of glucose; and the offensive smell communicated to the waste water gives trouble. Woelker attempted to cultivate ferments which would attack only the cellulose and leave the starch untouched; rasping is thus totally suppressed. The author adopted Woelker's process, and in three days obtained a pulp containing 18 per cent. of starch; the starch obtained, however, deposited incompletely and was always greyish and difficult to dry, but no lesion of the grains could be detected under the microscope. A large French factory, after trying a similar process, abandoned it in favour of rasping.

Few French factories are prepared to admit that they lose 15 to 20 per cent. of starch from defective methods. Antiseptics are little used, whilst in Germany the use of sulphurous acid and calcium bisulphite is general.

The determination of starch in potatoes by saccharification with acids is far from exact from the point of view of the starch manufacturer, as a portion of the cellulose is said to be saccharified, and the sugars, glucoses, carbohydrates, and soluble starches present, are counted as starch. The error from this cause may amount to 2 per cent. of the total dry starch. Maercker and Saare found in potatoes 0·58 to 6·07 per cent. of soluble carbohydrates calculated on the starch, and one of them found 35 per cent. in immature tubers. Aimé Girard showed by photomicrography that when potatoes sprout, starch is rendered soluble and passes into the expanding portion of the tuber. Baudry's method of estimation is equally inexact, for, in addition to the bodies above mentioned, the dextrins produced, affect the rotatory power of the starch rendered soluble by the salicylic acid. He has proposed a correction for pectin which has passed into solution, of 0·2 when the acid is used alone, and 0·35 when zinc chloride is also added. The solution is boiled for 50 minutes. As tubers of the same variety may show differences of 10 to 12 per cent. in starch, it is doubtful if the other constituents are more fixed.

After maturity the tubers rapidly change, starch becoming soluble and nitrogenous matters being transformed partially into solanine, the proportion of 44 mgrms. of solanine per kilo. for healthy tubers rising rapidly to 90 and 112 mgrms. The eyes contain 2,700 mgrms. per kilo. Baudry's method is rapid, the figures obtained being constantly higher than those given by saccharification and titration with Violette's solution.

For the determination of starch by density, preference should be given to an apparatus operating on 5 to 10 kilos., and the tubers should be cut to avoid air bubbles. The tables established in Germany by Behrend, Holdefleiss, Maercker, and Heidepriem (the latter being most highly approved) are far from being universally applicable. The author is of opinion that in each district observations should be made over several years and special tables prepared for each region. 170 samples of blue giant of the Nord gave by Heidepriem's table 2 per cent. less than by saccharification. 29 samples from the Oise showed 3 per cent. too high.

For control of the pulps and waste water, the author saccharifies by acids, defecates by acetate of lead, and, if necessary, filters through char. Maercker's method for the examination of pulps demands much time and manipulation, and Baudry's is slow and liable to considerable error.

Many starchworks have been closed for want of a practical method of dealing with the waste water, 10 to 13 litres of water being rejected per kilo. of potatoes worked.

—L. J. de W.

Carbohydrates, On the Thermal Phenomena attending the Change of Rotatory Power of Freshly Prepared Solutions of Certain; with some Remarks on the Cause of Multirotation. H. T. Brown and S. Pickering. Proc. Chem. Soc. 1897, [181], 129—130.

During an investigation of the thermal changes attending hydrolysis under enzyme action, the results of which are described in the next abstract, it became necessary to inquire whether the change in the multi-rotation of certain sugars is attended with any heat disturbance, as it is now well known that, at any rate, dextrose and maltose are liberated by hydrolysis in the "birotatory" state. The authors find that the changes of rotation experienced by dextrose, levulose, and milk-sugar in passing from the optically unstable α- to the optically stable β-form, are accompanied by distinct thermal effects, which, although taking place slowly in the solutions under ordinary conditions, can be produced, like optical stability, almost instantaneously by the addition of traces of an alkali. A full account is given of the apparatus employed, of the method of experiment, and the nature of the corrections to be applied. In the cases of dextrose and milk sugar, there is a liberation of heat accompanying the change of rotatory power; with levulose there is a very decided absorption; and with maltose no thermal disturbance is recognisable. The following are the values obtained :—

——	Per Grm. of Sugar.	Per Grm.-Molecule.
	Cal.	Cal.
Dextrose	+ 0·588	+ 106
Levulose	− 4·64	− 835
Milk-sugar	+ 0·19	+ 34
Maltose	0	0

The authors discuss the various explanations which have been given from time to time to account for multi-rotation, and consider that their experiments favour the view that it is conditioned by chemical rather than physical causes, and that Fischer is probably correct in his suggestion that dextrose, for instance, in passing from the optically unstable to the optically stable modification in solution, passes from the aldehyde, $C_6H_{12}O_6$, to the heptahydric alcohol, $C_6H_{14}O_7$. They believe, however, that the analogy which Fischer suggested, of the change of a lactone into its acid, is less close than that afforded by the gradual change of acetic aldehyde, in contact with water, into ethylidene glycol, where the CHO group becomes $CH(HO)_2$.

Carbohydrate Hydrolysis, On the Thermo-Chemistry of. (I.) The Hydrolysis of Starch by Vegetable and Animal Diastase. (II.) The Hydrolysis of Cane Sugar by Invertase. H. T. Brown and S. Pickering. Proc. Chem. Soc. 1897, [181], 130—131.

The attempts made to determine the thermal effects of hydrolysis have hitherto been confined to indirect methods, based on the heats of combustion of the hydrolysable substance and its products. Such methods, it is shown, cannot give results of any real value, as the thermal changes to be measured, are considerably within the experimental errors of the combustion values.

The paper describes the results obtained by direct measurement of the heats of hydrolysis of starch and of cane sugar. Lintner's soluble starch was for the most part used, as there are certain mechanical difficulties in employing starch paste in the calorimeter, owing to its viscidity. The hydrolytic agents used for starch were: (1) malt diastase, (2) pancreatic diastase, (3) Taka diastase, and (4) saliva. With malt diastase, the heat of hydrolysis was found to be +2·60 calories per grm. of amylin converted into maltose. The amount of heat is proportional to the water fixed, and is independent of the molecular complexity of the amylin attacked. The breaking down of the starch molecule prior to hydrolysis does not appear to be attended with any thermal disturbance.

With pancreatic diastase the heat liberated per grm. of amylin hydrolysed amounts to +1·8 cal., a value sensibly less than that deduced from the action of the malt diastase. With Taka diastase the heat disturbance is still less than with the other two agents. The possible causes of these differences are discussed.

Cane sugar was hydrolysed with invertase, and was found to give a thermal effect of +11·21 cal. per grm. of cane sugar inverted when the products were in the optically stable β-form, and 13·34 cal. per grm. at the moment of liberation of the products, *i.e.*, when they are in their "birotatory" or optically unstable form. It is the larger number which correctly represents the heat of hydrolysis of cane sugar.

Glucose, A Study of the Direct Production of, from Raw Material. F. Lippmann. Chem. Zeit. 21, [46], 458—460.

The author reviews the different modern methods of producing glucose from raw material without previous production of starch. In Europe the potato only need be considered. In order that the preparation of glucose from potatoes without the previous manufacture of starch may be practical, the following conditions must be fulfilled :—

1. The final product must be of equal value to that prepared from pure starch.

2. A larger quantity of final product must be obtained from the same quantity of raw material than by preparing starch from it and then working this intermediate product.

3. The cost must be no greater than by the starch process.

These conditions are difficult when one considers that purified starch is almost a chemically pure body, whereas the potato has the following average composition :—

Water, 73—78 per cent.; protein, 2—2·5 per cent.; fat, 0·1—0·3 per cent.; starch, 18—22 per cent.; cellulose, 1—2 per cent.; ash, 1—1·5 per cent.

The author passes by processes previous to 1831, in which the finely divided potatoes directly underwent inversion. The action of the H_2SO_4 on the cellulose and colouring matter of the potatoes, produced a bad-coloured and bad-smelling product. He then discusses the processes of Anthon (Dingler's Polyt. J. 1859, **154**, 69). Cords-Verneisel, Boudonneau (La Féculerie, 1893, **2**, 188), Pieper (Chem. Zeit. 1894, **18**, 1956, and Ger. Pat. 77,158, Aug. 22, 1891), Lassmann (Wagner, Die Stärkefab., 2 Aufl., 583), Colas, and Davoine (Dist. Franç. 1890, 474).

Boudonneau's method gives a product of better quality than the others, though Pieper's is very worthy of notice. The principle of Boudonneau's method consists in washing out the albuminous bodies with pure water from the very finely divided material; this is then pressed and the pulp inverted with dilute acid under pressure, but below the boiling point; the object being to prevent the decomposition of the cellulose.

From 1,000 kilos. of pressed pulp (75 per cent. H_2O), Boudonneau obtains 3,000 litres of glucose syrup of sp. gr. 1·030.—J. T. W.

PATENTS.

Refining of Sugar [Barium Peroxide], Process for the Complete. G. Ranson, Phalempin, France. Eng. Pat. 11,790, May 30, 1896.

"This is a process to obtain colourless syrups, derived from raw beetroot or cane sugars, by taking from them the chief part of the coloured molecules, by oxidising those that are poor in oxygen or that do not contain any, and by reducing those which are rich in oxygen." Barium dioxide, capable of being regenerated several times according to the quantity of syrup to be treated, is used either alone or by preparing a special solution of it with the addition of a very small quantity of bone-black. From 2 to 5 per cent. of the solution at 20° to 25° B. is added to the syrup "at a temperature of triple effect," mixed, and left in contact until the whole of the gas has been given off, and then carbonated. Or, for refining raw products sufficiently pure—say of the commercial type known as No. 3—the sugar is not dissolved, but broken into grains of the size of the refined product. A clairce is also prepared with No. 3, at 36° B., which is treated cold with about 3 per cent. of barium dioxide in the form of oxidised water, and ½ per cent. of fine or impalpable bone-black.

The barium carbonate is attacked with nitric acid to give carbonic acid and barium nitrate, which will be easily decomposed by heat and yield pure baryta, and the nitric acid be recovered.

For making tablets, the decolorised and refined syrup is run into a mould, or some modified forms thereof, in which wedges and vertical blades or partitions and reglets, with perforated sheet-iron plates placed horizontally, form a series of rectangular partitions, on which a space is reserved for brushing off and adding the quantity of clearc determined upon. Under the mould is situated a liquoring table, with which, by means of a vacuum, the operations of liquoring, sucking out, and the like are carried on when the syrup is partly solidified. There is a modification to suppress the horizontal perforated iron sheets, and an apparatus to be placed on the mould to convey damp air to facilitate the rapid detachment of the tablets, and a lifting table with an arrangement of screws to remove the vertical plates. The metal blades are loosened from the tablet block by a lever press. There are six sheets of drawings.—L. J. de W.

Molasses, Impt. in Extracting Sugar from, by means of Barium Hydroxysulphide, and Recovering the latter. W. Feld, Hönningen a Rhein, Germany. Eng. Pat. 16,441, July 24, 1896.

In extracting sugar from molasses by means of barium hydroxysulphide alone, there is this disadvantage, that only half the barium introduced is utilised in producing barium saccharate, whilst the other half passes into the residual liquid, from which it has to be recovered.

In the present invention this disadvantage is obviated by the use of a mixture of barium hydroxysulphide with an equivalent quantity of alkali hydroxide, or by treating the molasses with these reagents successively.

Sulphuretted hydrogen is separated from the residual liquor containing alkaline sulphhydrate by means of carbonic acid and heat, and the carbonised residue is again converted into alkali hydroxide by the action of caustic lime.

—L. J. de W.

Adhesive Compounds, Impts. in or relating to. A. J. Boult, London. From C. M. Higgins, Brooklyn, U.S.A. Eng. Pat. 9108, April 9, 1897.

The object of this invention is to produce an adhesive compound in the form of a dry or nearly dry powder, put up in bulk or in small packages or masses, which may be used for the production of sizes, pastes, or mucilages when dissolved in hot water, and will give the effect of manufactured dextrin, or a superior effect, at much less cost than dextrin, and in a manner which will enable the desired quality of the resulting paste or size to be more readily controlled and determined by the user or consumer than is possible with manufactured dextrin.

To this end a new article of manufacture is produced, consisting essentially of raw or unconverted starch or flour in a dry or powdered state, impregnated with some digestive or converting acid, or agent adapted in the presence of heat and moisture for the conversion of the starch or flour into a soluble or dextrin state, so that when such impregnated starch is dissolved in hot or boiling water it will produce a solution of actual dextrin, or more or less approaching actual dextrin, according to the temperature and time employed in making such solution, which is under the control of the user. This acidulated starch powder is put up in suitable receptacles or packages, and included in the same package a separate mass of neutralising alkaline powder duly proportioned to the contained mass of acidulated starch, but enclosed in a separate receptacle or envelope. After a quantity of the impregnated starch has been dissolved in hot water, a due proportion of the alkaline powder may be added to neutralise the acid and perfect the product. It is preferred to impregnate the starch with sulphuric acid and boric acid, and to form the neutralising powder of caustic soda or borax in a powdered form. The use of a solid dry digesting acid, such as oxalic acid in a powdered form, is also claimed, and the addition of an essential oil perfume or flavouring substance to the neutralising powder.

—L. J. de W.

XVII.—BREWING, WINES, SPIRITS, Etc.

Kilned Malt, Storage of. L. Aubry. Zeits. f. d. ges. Brauw. **20**, [25], 265—267.

Comparative experiments were made on the influence of temperature during storage, by filling three adjacent bins, each of 12 cb. m. capacity, with malt, Nos. 1 and 2 (the latter jacketed to prevent radiation) being filled with malt fresh from the cleaning heap, three days after unloading from the kiln, at a temperature of 119·7° F., and the former left open for 40 days, the temperature then registering 70·25° F. Bin No. 3 was filled from the same heap, 16 days after the others, and also left open, No. 2 being closed all the time. The temperature in 1 and 3 remained below 70° F. during the six months' duration of the experiment, whereas that in No. 2 only fell to 99·25° F. at the end of three weeks, and remained above 77° F. the rest of the time.

When analysed, the warm-stored sample revealed a rather lower water-content than the others, and the percentages of extract and maltose were also lower, but that of nitrogen higher; these conditions being, however, reversed in the wort after mashing. In the matured beer, the attenuation and percentage of alcohol were lowest in this sample, and conversely, the content of residual extract and maltose was greatest. The actual attenuation of the three

beers, in numerical order, was 50·0°, 48·0°, and 51·3° respectively, but there was no apparent difference in their flavour, brilliancy, gas content, &c.

The conclusions drawn by the author are to the effect that the filling of malt warm into silos of not excessive dimensions will not lead to any deterioration of flavour or production of rancidity, provided the conditions permit of gradual cooling and subsequent storage at normal temperature. If, however, the temperature be allowed to remain at over 100° F. for long, bad results may ensue, particularly with malt containing much moisture, since oxidation and other reactions, resulting in alterations of flavour, are thereby facilitated. The free circulation of air is also a factor of some weight, and accounts for the rancidity of malt stored warm in sacks. Exposure to air, as in the case of sample No. 3, favourably influences the rendering soluble of the extract and the clarification of the wort.—C. S.

Brewing Utensils, Disinfecting Power of Sodium Carbonate Solution in Cleaning. L. Steuber. Zeits. f. d. ges. Brauw. **20**, [19], 253—255.

From experiments made on the action of 5 per cent. and 10 per cent. solutions of pure calcined sodium carbonate on culture- and wild-yeasts, it appears that cells of both kinds of yeast are able to withstand 15 minutes' exposure to the 5 per cent. solution at 45° C., but are all killed at 55° to 60° C., whilst the 10 per cent. solution accomplishes the same result at 5° lower. Hot water used alone, with an exposure of half an hour, was without effect at 50° C., but above 60° C. destroyed the vitality of the cells.

Similar tests were applied to yeasts containing spores, the resisting powers of which were found to be greater, some even withstanding a short exposure at 75° C. In its influence on this form of growth, hot water is almost superior to the soda solution, and the action of the two strengths of the latter is nearly identical. Since fission fungi appear equally able to resist the action of soda (a germ of this order having been found to grow 14 days in a 5 per cent. solution in the laboratory), the disinfecting properties are presumably low, and it is therefore deemed advisable, when employing soda in brewery practice for cleaning out pipes and utensils, to make the minimum exposure 30 minutes and the minimum temperature 80° C., and to supplement its more mechanical property of loosening adherent matter by the sterilising influence of steam, with or without the conjunction of calcium sulphite, bleaching powder, &c.—C. S.

Amylomyces Rouxii, and other Starch-fermenting Moulds. J. Sanguinetti. Ann. Inst. Pasteur, March 1897; La Bière, **5**, [4], 49—52. (See also this Journal, 1897, 348 and 454.)

From the results of comparative experiments made with *Aspergillus orizæ*, Gayon's *mucor alternans*, and *Amylomyces Rouxii*, the author finds that the energetic saccharifying power possessed by all three is greatest in *A. orizæ*, the *Amylo. R.* taking second place. On the other hand, the latter leaves behind in a given time a larger amount of unconverted carbohydrates than either of the others—a circumstance which, combined with its greater fermentative power, renders it alone, out of the three, suitable for industrial employment, whether for the direct fermentation of starchy bodies or for utilising distillery residues.—C. S.

New Vinegar Bacterium. Zeidler. Centralbl. für Bakt. und Parasitenkunde, 1896, No. 23—24.

This bacterium has been named *Termobacterium aceti*; it converts alcohol into acetic acid, and subsequently converts a portion of the acetic acid into carbonic acid and water. If sugar is also present in the solution, a fixed acid—lactic acid—is also produced. In appearance it greatly resembles Kohn's *bacterium termo*. In old cultures, spirillum forms are frequently found. When grown in wort gelatin, glistening transparent patches are formed and the gelatin is not liquefied; as it is ærobic, it only grows on the surface.

The most suitable temperatures are between 70° and 85° F. It will not grow below 40° or above 120° F.

As this bacterium cannot grow without a free supply of air, there is little risk of its causing any trouble in beer.
—A. L. S.

Laccase: Its Oxidising Power, Influence of Manganese upon. G. Bertrand. Comptes Rend. 1897, **124**, 1032—1035.

In his researches (this Journal, 1895, 374 and 1074; 1896, 561) on laccase, the author has already remarked that this oxidising ferment is relatively rich in manganese (0·1 per cent. Mn). Continuing his investigations, he has made the interesting discovery that the oxidising power of the ferment varies directly as the percentage of manganese present. The coincidence is not merely accidental, but is shown to be due to a specific accelerating influence of manganese upon laccase. Comparative experiments were made with a specimen of laccase (derived from lucerne grass), containing only a minute trace of manganese, the volume of oxygen absorbed by a solution of hydroquinone (this Journal, 1895, 1074) under the influence of the ferment, being measured.

	Volume of O absorbed.
1. Manganese alone (blank experiment)	0·3 c.c.
2. Laccase alone	0·2 c.c.
3. Laccase + 1 mgrm. of manganese as sulphate	6·3 c.c.

The manganese cannot be replaced by other metals. A number were tried, but in no case was any accelerating action observed. This fact enormously increases the physiological importance to be attached to manganese, especially when it is considered that traces of this metal exist in many plants.—H. T. P.

Rum, A Bacterium in. V. H. Veley, M.A., F.R.S., and L. J. (Mrs. V. H.) Veley. (Communicated by the Authors.)

It is well known that considerable pecuniary loss has resulted to the manufacturers of rum in Demerara owing to "faultiness" in certain samples. In these, assessed as at 42 o.p. (equivalent to 74·6 per cent. alcohol by weight), when examined with a magnification of 1,200 diameters, cocci surrounded by gelatinous envelopes were found, not only in deposits, but also disseminated through the liquid. These cocci multiplied by division, and increased with greater or less rapidity, when transferred to suitable media. So far as the study of the life-history has proceeded, it appears that the bacterium belongs to the group of Coccacea, adopting the classification of Zopf. The organism has been found in 19 samples obtained from various sources.

Previous writers on "faulty rums" have observed in them flocculent precipitates, which have hitherto been considered as of a chemical nature; they, however, consist of aggregates of cocci in various stages of development. As these have been found invariably in "faulty" but never in "sound" rum, it seems reasonable to conclude that they form one of the principal causes of faultinesss.

The authors hope shortly to publish a full account of the micro-organism based upon the investigations of its life-history, as exhibited by cultures in various media.
—V. H. V.

Alcohol, Denaturing of. E. Barillot. Comptes Rend. **124**, [21], 1163.

The Swiss revenue authorities employ, for rendering alcohol non-potable, the "oils of acetone" resulting from the destructive distillation of the acetates and other organic salts of the alkaline earths. The author shows that this method is useless, since by treating the alcohol so denatured with bisulphite of sodium, the added oils are almost wholly precipitated and may be removed by simple filtration. This filtrate, on fractional distillation, gives 70 per cent. by volume of potable spirit. The minute trace of acetone which this distillate may still retain is readily removed by treatment with iodine or chlorine in alkaline solution, after which the recovered spirit is undistinguishable from duty-paid alcohol.—J. O. B.

Wines, Loss of Colour in (Casse des Vins), Properties of the Ferment Producing. P. Cazeneuve. Comptes Rend. 1897, **124**, 781—782.

In a preceding note (this Journal, 1897, 347), the author has shown that the disease known as "casse des vins" (attended by loss of colour and bouquet) is due to a soluble ferment—œnoxydase—under the influence of which, the colouring matter of wine is oxidised and precipitated, and the ethers, &c. similarly changed or destroyed. The ferment, although greatly weakened by contact with strong alcohol, retains its activity unimpaired for months in dilute alcoholic solution (9 per cent.) or in wine. Like other ferments, however, its activity appears to diminish in proportion to the amount of work done by it. Œnoxydase is powerfully affected by sulphurous acid: a mere trace (0·008 grm. per litre) of the latter added to wine entirely prevents the development of the "casse," the action of the acid being a directly destructive, and not merely a preservative one. On the contrary, formaldehyde, in comparatively large doses (0·08 grm. per litre), has no restrictive influence whatever. The colouring matter of the wine, although precipitated by the formalin, is, nevertheless, oxidised and rendered insoluble in dilute sulphuric acid, in which reagent the precipitate, produced by formalin in a sterile wine, is readily soluble. The activity of œnoxydase is neither increased, nor diminished, by blowing a current of oxygen or ozone through its solution.—H. T. P.

Maltone Wines [Malt Wines], Preparation of. J. Sauer. Zeits. f. Spiritusind. 1897, **20**, 156—157.

The so-called Maltone wines do not in the least resemble a mixture of wine and beer, as is frequently alleged. They owe their peculiar character to the method of preparation, which differs essentially from that adopted in the production of beer, and especially in the following points:—(1.) In the lactic fermentation of the malt wort. (2.) In the selection of the yeast and the degree of attenuation brought about by the latter. (3.) In the method of storage. The wort is prepared by the infusion method from the best barley malt, and under conditions yielding the highest proportion of maltose. A pure culture of the lactic acid bacillus is added to the wort, and the lactic fermentation allowed to proceed for 18—24 hours at 50°—51°; when the acidity has reached 0·6—0·8 per cent., the product is sterilised by heating it to 88°—94°. It is subsequently cooled and a pure culture of a specially selected wine yeast is introduced. Fermentation proceeds vigorously and the fermentable sugars soon disappear; more maltose in the form of malt extract is then added, and at a later stage pure cane sugar is introduced. By carefully conducting these fermentations, it has been possible to obtain over 19 per cent. by volume of alcohol. When at the end of the fermentation the yeast has settled and the wine is clear, the latter will still possess a decided malt aroma, which has yet to be removed, and this is effected in 3—4 weeks by warm storage and free contact with air.—A. K. M.

Active Diastase, Uncertainty of the Guaiacum Reaction for. B. Pawlewski. Ber. 1897, [10], 1313.

See under XXIII., *page* 639.

Alcohol, The Estimation of, by the Vaporimeter. F. Rothenbach. Wochenschr. f. Brau. 1897, **14**, 189.

See under XXIII., *page* 642.

PATENTS.

Pasteurising Liquids, New or Improved Apparatus for. J. H. W. Ortmann and C. W. C. Herbst, Hamburg, Germany. Eng. Pat. 21,203, Sept. 28, 1896.

The object is to pasteurise beer and other liquids containing gas whilst the liquid is passing from the storage or other vessel, and in such a manner that the gas is not lost. This is effected by causing the liquid to pass through a series of horizontal, undulating, ascending, and descending pipes contained in heating and cooling tanks. The gas, which separates during the heating process, is carried forward by the current of liquid, and becomes re-absorbed during the cooling operation.—A. K. M.

Mashing and Brewing, Impts. in the Process of and Apparatus for. L. Procházka, Turnau, Bohemia, and E. Frischauer, Vienna. Eng. Pat. 5582, March 2, 1897.

The object aimed at is a shortening of the time required for carrying out the process previously described by one of the patentees in Eng. Pat. 17,246, 1896 (this Journal, 1896, 915). The process consists in straining the mash after having brought it to a temperature of 50°—70°, boiling the grains with water to gelatinise the starch, and then reuniting the grains with the diastatic wort, in order to saccharify the remaining starch. The whole mash is finally boiled, to destroy the diastase, and the wort strained from the grains.—A. K. M.

Drying Substances [containing Aromatic Oils], Improved Process for. H. Humbser, Fürth, Bavaria. Eng. Pat. 6065, March 8, 1897.

This process is more particularly intended for the treatment of substances, such as hops, which contain aromatic oils readily volatile at comparatively low temperatures. The drying is effected by means of a current of air, which is first passed through a cold chamber, in which it deposits its moisture, then through a warming chamber, from which it passes through the substance to be dried. After taking up moisture from the latter, the air is again dried in the cold chamber, and the process continued as long as desired. The temperature to which the air is raised should be below that at which volatilisation of any aromatic constituents would occur.—A. K. M.

Non-Intoxicating Drinks, New or Improved Compounds for Making. J. H. Brodrick, Northfield, Worcestershire. Eng. Pat. 15,004, April 7, 1897.

A fermented, non-intoxicating drink, in imitation of stout, is made from the following constituents:—Hops (2 oz.), roasted malt (8 oz.), caramel (¼ oz.), ground ginger (1 tablespoonful), which are well mixed and compressed into a solid cake, the quantities given being sufficient to make two gallons of beverage. The cake is boiled with the water, sugar (1½ lb.) added, the product is allowed to cool, and is fermented and bottled in the usual way. For imitation ale, the caramel is omitted, and crystallised, or brown or amber malt is used in the place of the roasted malt.—A. K. M.

Mead, New or Improved Process for the Preparation of. M. van Look, Cologne, Germany. Eng. Pat. 10,180, April 23, 1897.

Pure honey is dissolved in water, boiled, and skimmed; lemon juice is added, the boiling and skimming being continued, whilst, if desired, spices, fruit syrups, &c. are introduced and stirred in. The liquor is allowed to cool, poured off from the sediment, and yeast is then added to it. After fermentation, it is poured into a fresh cask, in which it is allowed to remain for 3—4 months or longer, after which time it is ready for bottling.—A. K. M.

Barley, Impts. in Treating, for Alimentary Purposes, including Brewing and Distilling. J. Dunn, Kelso. Eng. Pat. 16,671, April 27, 1897.

See under XVIII. A., *page* 628.

XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

(A.)—FOODS.

Cows' Milk, On the Albuminoids of. K. Storch. Monatsh. f. Chem. 1897, **18**, [4], 244—281.

The author's experiments confirm Hammarsten's conclusion that only one kind of casein is present in cows' milk. When the milk is saturated with sodium sulphate, magnesium sulphate, or sodium chloride, the precipitate is not this unaltered casein, but one of two decomposition products, each of which contains phosphorus and is of similar composition to casein. Acetic acid precipitates milk casein

in an unchanged form, and it is possible that simultaneous saturation with two of the three above-named salts might also cause it to separate without decomposition.—C. A. M.

Milk, The Cause of the Coagulation of Heated. B. Bardach. Monatsh. f. Chem. 1897, **18**, [4], 199—216.

From the results of his investigation, the author came to the conclusion that the view of Cazeneuve and Haddon, that the coagulation of milk was caused solely by the action of acids produced by the oxidation of the lactose present in milk, was untenable. Preliminary experiments showed that the length of time required for complete coagulation of fresh milk was: at about 100°, about 12 hours; at 110°, five hours; at 120°, $1\frac{1}{2}$ hour; at 130°, one hour; at 140°, 20 minutes; and at 150°, three minutes. The partial separation of albuminoid substances which occurred on heating fresh milk for many hours at a little below 100°, was regarded as quite distinct from coagulation proper. Throughout the subsequent experiments, the time and temperature adopted were one hour and 130° respectively, the milk being heated in an autoclave to avoid frothing.

Fresh milk coagulated by heating at this standard temperature, and the clear serum distilled, yielded, as the result of many determinations, not more than 0·003 grm. of formic acid per 100 c.c. of milk. On adding 3 c.c. of 20 per cent. sulphuric acid to the filtered liquid before distillation, the amount of formic acid in 100 c.c. increased to 0·03 grm. A subsequent determination of volatile acid by distilling fresh milk, before coagulation, with the same proportion of sulphuric acid, gave 0·004 grm., the difference between this result and the preceding one being ascribed to the volatile acid produced in a state of combination by heating the milk to 130°. This amount of acid was too small to produce coagulation in the cold, which first occurred when the acidity, expressed as acetic acid, exceeded 0·13 per cent. (Hammarsten, 0·075 to 0·1 per cent.). With regard to the combined action of heat and acid, fresh milk boiled with gradually increasing quantities of acetic acid, first showed signs of coagulation when the percentage reached 0·055; but coagulation was not complete until the milk contained 0·07 per cent.—an amount considerably in excess of that of the volatile acids produced by heating the milk by itself at 130°. This result excluded the possibility of the volatile acids being the sole cause of coagulation.

Moreover, on mixing the acid liquid formed by heating a 10 per cent. aqueous solution of lactose containing 1 grm. of hydrogen disodium phosphate for an hour at 100° or 130° with an equal volume of fresh milk, no alteration, beyond the discoloration, appeared in the latter, either in the cold, or on heating on the water-bath, or on once boiling. Hence the author concluded that neither the volatile acids nor the total acids produced from lactose were the exclusive cause.

The behaviour of pure casein was next studied. A 10 per cent. aqueous solution containing 1 per cent. of hydrogen disodium phosphate heated for an hour at 130° partially coagulated, but a further deposit could be obtained from the filtrate by the addition of acetic acid.

The further experiments described in the paper, proved, in the author's opinion, that the casein was so altered by heating that it could be completely coagulated by the small amount of acid formed on heating lactose solution containing an alkali at 130°, and he considered that the coagulation of milk was dependent on the simultaneous action of both these factors.—C. A. M.

Meat Extracts, Analysis and Composition of. J. Bruylants. J. Pharm. Chim. 1897, **5**, 515.

See under XXIII., *page* 640.

Fats: Differentiation by the Volume of the contained Fatty Acids. R. Zaloziecki. Chem. Rev. Fett- u. Harz-Ind. **4**, [9], 119.

See under XXIII., *page* 639.

Horseflesh, Detection of, in Sausages and Meat Preparations. H. Bremer. Forschungs-Ber. 1897, **4**, 1.

See under XXIII., *page* 641.

Butter and Margarine, Discrimination between. A. v. Asbóth. Chem. Zeit. 1897, **21**, [32], 312.

See under XXIII., *page* 640.

Theobromine, Determination of, in Cocoa and Chocolate. L. Maupy. J. Pharm. Chim. 1897, [6], **5**, 329.

See under XXIII., *page* 641.

PATENTS.

Mineral Waters, Impts. in or relating to Artificial. J. K. Anderson, Waukesha, Wisconsin, U.S.A. Eng. Pat. 8779, April 6, 1897.

Any good drinking water is charged with carbon dioxide. Lithium carbonate and boric acid are then dissolved in separate quantities of pure water, the solutions are mixed, filtered if necessary, and the carbonated water is added in such proportion that the resulting mixture shall contain about 35 grains of lithium carbonate and 15 grains of boric acid to the gallon.—L. A.

Barley, Impts. in Treating, for Alimentary Purposes, including Brewing and Distilling. J. Dunn, Kelso. Eng. Pat. 16,671, April 27, 1897.

The barley is first treated as in the ordinary operations for forming "pot" barley, and the grains are then cut or broken by means of grooved rollers. The product is sifted, moistened with water, and passed between smooth and heated rollers, the flakes obtained being finally dried in the usual way.—A. K. M.

Milk and certain other Classes of Fresh Foods, An Improved Process and Apparatus for Preserving, by Sterilisation. A. J. Vause, Tempe Sydney, New South Wales, Australia. Eng. Pat. 11,637, May 22, 1897.

This specification describes an apparatus into which milk or other article of food may be introduced and sterilised by heating, and then, without being again brought into contact with unsterilised air, may be hermetically sealed up in vessels which have been sterilised in another part of the apparatus.—L. A.

(*B.*)—SANITATION; WATER PURIFICATION.

Arsenical Wallpapers, Cause of the Poisonous Action of. B. Gosio. Ber. **30**, 1024—1026.

A reply to Emmerling (Ber. **29**, 2728; this Journal, 1897, 158). No doubt there are many moulds and bacilli which can flourish in an arsenical culture-solution without giving off gaseous arsenic compounds; but some, though it may be few, certainly do evolve such compounds. *Mucor mucedo* and *Aspergillus glaucus* are among these, and Emmerling's failure to obtain evidence of such gaseous products must be ascribed to his having used unfit or unacclimatised varieties. Much more active than either of these is *Penicillium brevicaule*; this microbe works so energetically on all arsenical compounds that it can be used for the qualitative detection of arsenic. The author has not yet been able to identify the gas given off by the action of this microbe, but it is highly poisonous—a mouse shut up with a culture of the mould in an arsenical solution died in a very short time. *Penicillium brevicaule* develops with great rapidity on wallpapers; and this fact, coupled with the extremely poisonous character of the product, demonstrated as above stated, leaves no room for doubt that poisoning may occur through volatile arsenic compounds. Of course, this does not exclude the possibility of poisoning by arsenical dust.—J. T. D.

Arsenical Wallpapers, Cause of the Poisonous Action of. O. Emmerling. Ber. **30**, 1026.

A rejoinder to the above. The volatile compound of arsenic which one would expect is arsine, but in none of the author's experiments could a trace of this be found. Gosio has failed to identify any volatile product given off; and that the mouse in question referred to, died from arsenical poisoning has not been proved.—J. T. D.

Lead Chromate in Coloured Wrappers, Rapid Method of Detecting. J. Wolff. Annal. Chim. Analyt. 1897, 2, 105.

See under XXIII., *page* 635.

Vapour Condensers and Apparatus for Destroying Noxious Fumes, Recovering By-Products from Distillation, and like Purposes.

See under I., *page* 602.

PATENTS.

Water-softening Apparatus, Impts. in. H. L. Doulton and W. A. Manger, London. Eng. Pat. 11,559, May 27, 1896.

The hard water entering the apparatus is used to drive a wheel or motor suitably connected with stirring apparatus in the reagent tank, this tank being fitted also with floats and overflow pipes for removing the clear reagent. After leaving the wheel, the water flows through a pipe into a receiver and funnel, where it mixes with the reagent. The supplies of hard water and reagent are controlled by ball-valves and floats, which are also so arranged that when from any cause the supply of hard water ceases, the valve on the reagent supply is automatically shut.—O. H.

Impure Water and other Liquids, Impts. in the Manufacture of Materials for Use in the Filtration and Purification of. A. Angell, Southampton, and F. Candy, London. Eng. Pat. 14,125, June 25, 1896.

A highly porous filtering material is produced by mixing the following ingredients with sufficient water to make them cohere, then stoving and baking the material and afterwards granulating it. The proportions may be varied, and instead of tar and water a solution of treacle may be used:—

Dust or powder of the porous substances described in Eng. Pats. 13,818 of 1884 (this Journal, 1886, 38) and 15,948 of 1885 (this Journal, 1886, 671), 100 parts; powdered or granulated manganese, 5 parts; dried and powdered clay, 100 parts; tar, 2 parts.—L. A.

Sewage and other Polluted Water, Improved Preparations for Use as Precipitants in the Treatment of. A. Angell and F. Candy, Southampton and London. Eng. Pat. 14,124, June 25, 1896.

The essential feature of this invention is the use of nitre cake as an economical source of sulphuric acid. By grinding up bauxite with a solution of nitre cake, with or without the addition of more sulphuric acid according to the proportion of nitre cake used, a mixture of sulphate of alumina with sulphate of soda or potash is produced. This may be mixed with a solution of ferric sulphate, made by heating together ferrous sulphate, nitre cake, common salt, and manganese peroxide; or the ferric solution may be used alone.—L. A.

(C.)—DISINFECTANTS.

Corrosive Sublimate, Disinfecting Properties of. Borchow. Pharm. Zeit. Russland. 36, 162; Chem. Centralbl. 1897, 1099—1100.

The varying results obtained by different workers on the disinfecting powers of corrosive sublimate are explained by the different conditions of the experiments. The author found that the cultures mentioned were destroyed by different solutions in the times stated, the conditions being those which would obtain in the disinfection of living-rooms.

——	0·1 Per Cent.	0·2 Per Cent.
B. subtilis spores	6 days	5 days
Anthrax spores	9—10 hours	3—5 hours
Staphylococcus aureus	$2\frac{1}{2}$—5 „	$3\frac{1}{2}$ „
Typhus bacilli	50—90 minutes	30—80 minutes
Diphtheria bacilli.........	70—80 „	60—70 „

——	0·5 Per Cent.	1·0 Per Cent.
B. subtilis spores	22 hours	12 hours
Anthrax spores	70—80 minutes	60—70 minutes
Staphylococcus aureus	60—70 „	50—80 „
Typhus bacilli	20—50 „	15—25 „
Diphtheria bacilli.........	25—30 „	15—20 „

Cholera bacilli and anthrax cultures free from spores were killed in 15 seconds. The addition of 1 per cent. of sodium chloride, 0·5 per cent. of tartaric acid, or 0·5 per cent. of carbolic acid, and also the action of light, decreased the sterilising power of the sublimate solution.—A. C. W.

Arthriticine: A New Disinfectant. Pharm. Zeit. 1897, 42, 118.

Arthriticine is a new proprietary bactericide and disinfectant. Its composition is represented by the formula—

$$C_6H_4(OC_2H)N(CH_2NH_2CO)N(CH_2)_4NH,$$

and it is therefore monohydrophenolethyldiethylenediamine-amido-acetonitrile. It is prepared in tablets, each containing 25 centigrms. It is almost insoluble in cold, but is readily dissolved in boiling water. A 1 per cent. solution prevents the development of bacteria, but the spores of anthrax bacillus retained their vitality unimpaired after contact for several weeks with a concentrated solution of arthriticine. It is therefore only a weak antiseptic.
—J. O. B.

Formaldehyde on Urea, The Action of. [*Disinfectant.*] C. Goldschmidt. Chem. Zeit. XXI., 46, 460.

When formaldehyde is allowed to act on urea, various condensation products are formed, according as the solution is acid, neutral, or alkaline.

In acid solution the body $C_5H_{10}N_4O_3$ is formed (Ber. 29, 2438). When urea is allowed to stand for 24 hours with an excess of 40 per cent. formaldehyde solution, which has been rendered alkaline, a white precipitate is formed, soluble in hot water, from which it is precipitated as a jelly by alcohol. Mineral acids, on long boiling with water, separate formaldehyde; the latter is also slowly given off when the body is exposed to the air, so that it could be used as a disinfectant.

In neutral solution a mixture of a soluble and an insoluble body was formed. The latter appears to have the composition $C_5N_4H_{12}O_4$, the former to be the body above mentioned. The results of the analyses show that methylene urea is not formed from formaldehyde and urea, as Tollens and his co-workers suppose.—J. T. W.

Heavy Tar Oil Miscible with Water. Jégou. J. de Pharm. Chim. 1897, [6], 5, 328.

By admixture with an alkaline resin soap, the author renders heavy oil of tar readily miscible with water, so that when diluted, an emulsion is formed in which the disinfectant is evenly distributed. The soap for this purpose is prepared from: resin, broken small, 100 grms.; soap makers' lye, 95 grms.; distilled water, 200 grms.; commercial oleic acid, 40 grms. Dissolve the resin by boiling in the lye mixed with the water, and evaporate to produce 200 grms. of resin soap, cool, and add the oleic acid. Soft soap, 80 grms., may be substituted for oleic acid, in which case, 10 grms. less of lye is taken, and the evaporation is carried only to 240 grms. Of this oleo-resin soap, 240 grms. are dissolved in sufficient heavy oil of tar to produce 1 kilo.; the soap is first warmed and intimately mixed with 400 grms. of the tar oil; the temperature is then raised, but not to boiling, until complete solution is effected; the rest of the oil is then added, and the whole covered during cooling, to avoid too great loss by evaporation. When cold the product is filtered, or strained through a cloth.
—J. O. B.

XIX.—PAPER, PASTEBOARD, Etc.

Paper, Specific Weight of. Papier Zeit. 1897, **22**, [31], 1098.

The difference in weight between a square metre of papers of equal thickness may be due to the different treatment of the fibres in grinding, pressing, drying, &c., but is most probably due to the difference in the specific gravity of the fibres of the raw materials, and we are therefore entitled to speak of a "specifically light paper." A great difference may be observed between soda- and sulphite-cellulose fibres, soda-cellulose fibres being probably the lightest in use for paper-making. In sulphite pulp the encrusting matter is not entirely removed from the fibres, thus rendering them specifically heavier, and it has also the appearance of being sized, rendering it unsuitable for copying- or blotting-papers. The fibres treated with soda-lye swell, and thus appear lighter. The different kinds of wood have not much effect on the weight, though soda cellulose from hard-wood trees was found lighter than soda cellulose from pine wood. The cleaner the pulp, the lighter it will be; and considerable care should be taken when a heavy and light pulp are worked together, the latter tending to float, and sometimes it may be recognised in the paper by the fibres being on the surface, and in the direction of the running of the wire on the machine.

The editor of the *Papier Zeitung* remarks that:—

The specific gravity of cellulose is 1·6, the specific gravity of pine wood being 0·4 to 0·6. As the wood fibres are freed from encrusting matters, they approach nearer to the purer cellulose, and therefore become specifically heavier. Consequently, the soda-cellulose fibres are specifically heavier than the sulphite, the paper made from soda cellulose being erroneously termed "specifically light"—a term which should be avoided, and the paper marked "light" or "thin."—S. P. E.

Soda Cellulose, Silk Gloss of. Papier Zeit. 1897, **22**, [29], 1020.

It was observed by Mercer in 1844 that on treating cotton with soda lye, the fibres shrink, and their structure is altered from a ribbon-like form to a cylindrical one. The internal canals also disappear almost entirely—due to the thickening of the parenchymous cells. The mercerised cotton is also stronger and more elastic, and absorbs colouring matters much better than the ordinary material. Thomas and Prevost (Ger. Pat. 85,564) have found that when mercerised cotton is stretched to its original length, a rich silk-like gloss appears, which is unaffected by the drying processes. The fibres are converted into a cylindrical form like silk, and the transparency becomes increased. The gloss, colour absorption, and blotting qualities of soda cellulose are already highly appreciated; and the question whether a paper with a rich silk gloss could be manufactured by the Thomas and Prevost method is worthy of experimental trial.—S. P. E.

Soda Cellulose, Formation of a Calcium Soap during the boiling of. Papier Zeit. 1897, **22**, [29], 1020.

At a North American soda-cellulose works, A. Zimmermann noticed the formation of black lumps of a wax-like substance, about the size of hazel nuts, which appeared in the lye, and which was insoluble in alkalis or alcohol. On analysis this substance was found to consist of raw fibres and a resinous calcium soap. The water used contained very little CaO (0·3 in 100,000); the lye did not contain a trace of CaO, the wood being therefore almost the only source of lime. The appearance was observed only when fresh pitch-pine was treated, old dry wood or fir not causing the formation. The formation was not affected by altering the strength of the lye.—S. P. E.

Sulphite Lye, Utilisation of, as a Waterproofing Solution for Paper. P. Remy. Papier Zeit. 1897, **22**, [43], 426.

Neutral or acid waste sulphite lye is treated with iron or lead salts, in quantities equivalent to the calcium salts present, when a waterproofing solution is obtained. As lead salts are poisonous and also discolour the paper, the iron salts are the more suitable. These solutions contain salts of other metals, according to the salt added, such as calcium chloride, if ferric chloride has been used. These may be removed before treatment, either by adding sulphuric acid, a soluble sulphate, or by treating with soda or ammonia, filtering, and then adding the iron salts. As the lye is generally dark-coloured, the paper is discoloured, but this may be reduced by washing with water, until the colouring matter—a combination of iron with a substance resembling tannin—is removed, the wash water being no longer coloured. The paper is then dried. The solution of lye may be evaporated before treatment, to remove some of the free sulphurous acid.

(1.) 100 c.c. of the unevaporated lye, containing about 0·83 grm. of CaO, are treated with a solution of $2\frac{1}{2}$ grms. of 66 per cent. ferric chloride or $4\frac{1}{2}$ grms. of crystallised ferrous sulphate, and neutralised with a little lye, soda, ammonia, milk of lime, &c. For concentrated solutions the lye is evaporated down, the alkali added, the whole filtered, and then the iron salt added.

(2.) 100 c.c. of the lye are mixed with sulphuric acid and placed in contact with the precipitate obtained by treating 3—4 grms. of ferric chloride or 5—6 grms. of ferrous sulphate with an alkali until the sour smell disappears.

(3.) 100 parts of the lye, after evaporating down to 34°—35° B., containing about 53 per cent. of solid matter and 4·4 per cent. of CaO, are treated with sufficient water to enable the lime (precipitated by adding 9 parts of soda ash) to precipitate easily, decanting, and adding to the clear liquid a solution of 30 parts of crystallised sugar of lead.

Paper treated with any of these solutions, after being subjected to a dry heat, or 70°—130° C., is rendered waterproof (Ger. Pat. 90,798).—S. P. E.

PATENT.

Paper Pulp from the Broom Plant, Impts. in the Manufacture of. H. H. Lake, London. From E. Berland and J. Girard, France. Eng. Pat. 210, Jan. 3, 1896.

The thick wood is first separated from the small stems, and the separated materials cut up in the usual way. The material is then digested in an open vessel with weak caustic soda, and afterwards, under pressure, with strong caustic liquors. The digested material is washed first while hot, and afterwards in the cold, and bleached in the ordinary manner.

The undecorticated broom may also be treated—after being cut, as before, into small pieces—in a disintegrator provided with an exhaust fan, which draws the light fibres of the bark from the machine and discharges them into a dust- or settling-chamber.

The crushed material, fine and coarse, is boiled with dilute caustic soda solution (1°—2° B.) for one or two hours, and passed through crushing rollers, provided with rotary cleaning brushes, after which it is further digested at a low pressure with alkaline liquor. The pulp is then treated in the usual manner

The treatment of the decorticated broom is similar.
—O. H.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

Ethylsulphuric Acid, Preparation of, from Gases charged with Ethylene. P. Fritzsche. Zeits. Anal. Chem. 1897, **36**, 303—306.

Having freed the gas from tar, ammonia, benzene, and hydrogen sulphide by treating it at 70°—80° C. with sulphuric acid containing not over 80 per cent. of pure acid (whereby homologues of ethylene, such as propylene, butylene, &c., if present, are simultaneously removed), the purified gas is heated to 110°—120°, by passing it through chambers maintained at this temperature. It is then conveyed to absorption apparatus, which consists of a group of scrubbers filled with acid-resisting material, over which strong sulphuric acid, heated to 110°—120°, flows in a

continuous stream. Fresh acid is introduced at the exit of the last scrubber of the series, where the gas leaves the apparatus. The acid collected at the bottom of the last scrubber is pumped into the last but one scrubber, and so on, until it reaches the first scrubber, into which the gas is introduced. By this arrangement an acid containing from 50 to 60 per cent. of ethylsulphuric acid can be obtained.
—D. B.

Ether, Free from Alcohol; Preparation of. P. Fritzsche. Zeits. Anal. Chem. 1897, **36**, 298—302.

Since on treating gas containing ethylene with strong sulphuric acid, ethylsulphuric acid is obtained, the direct preparation of ether therefrom is of importance. The author has devised a method which admits of the conversion of ethylsulphuric acid into ether and sulphuric acid according to the equation—

$$2(C_2H_5OSO_2OH) + H_2O = (C_2H_5)_2O + 2H_2SO_4.$$

Four distilling vessels, A, B, C, and D, are connected in such a manner that the gas evolved in A passes into the bottom of the vessel B, thence into the bottom of C, and, finally, into the bottom of D. On leaving the latter, the gas is conducted through a suitable condenser, and eventually the condensed product is collected. Each vessel is charged with the same quantity of ethylsulphuric acid. A is diluted with about 35 per cent. of water, B with about 22 per cent., and C with about 10 per cent., whilst D is not diluted. The distillation is then proceeded with in the following manner:—The contents of A are heated strongly, whilst the liquid in B is only slightly warmed. The gas liberated in A is composed of alcohol, ether, and steam, and is taken up by the liquid in B. The latter, after a short interval, begins to boil. Meantime the contents of C and D are heated slightly, so that the gas from B, which is now more highly charged with ether, raises the liquid in C to the boiling point and forces the gas into D. If the acid originally employed is of high percentage—that is to say, moderately free from uncombined sulphuric acid—the ether leaving the vessel C will, from the commencement, pass unaltered through the liquid in D, whilst any alcohol, which may be carried over mechanically, is acted upon by the acid in D, and converted into ether. If, however, the acid is highly charged with free sulphuric acid, the ether and alcohol passing from C to D at the early stages of the distillation are taken up by the free acid and converted into ethylsulphuric acid, until the point of equilibrium has been reached, at which ether passes unchanged through the liquid. If the distillation has been conducted in a proper manner, the volume of liquid in D should not have been altered; B should, however, show a marked and C a moderate increase. When the acid in A has been exhausted and concentrated to such an extent that the distillation in B can no longer be effected without the aid of external heat, the operation is stopped. The acid in A is run out, the liquid in B transferred to A, that in C to B, and in D to C, after which D is charged with a further quantity of undiluted acid. The strength of the liquid in A, B, and C is then readjusted as before, and the distillation continued until A has again been exhausted. Instead of removing the charge in A after each operation, the process can be made to work continuously by using an apparatus in which the strong acid passes in a constant stream into the vessel, water being introduced gradually into the apparatus to maintain the correct degree of dilution, the exhausted acid also leaving the apparatus in a continuous stream.—D. B.

Ether, Contribution to the Study of the Preparation of. L. Prunier. J. Pharm. Chim. 1897, **5**, 513—514.

Sufficient attention has not been given to the sulphonic acids and their derivatives formed in the manufacture of ordinary ether. The author makes the following statements:—1. These compounds are found in ordinary commercial ether, from which they may be extracted by washing with water, and in larger quantities in the residue of the ether manufacture. 2. They are contained in the oils formed in the rectification of the crude product. 3. The production of these compounds from ethyl sulphuric acid, especially towards the end of the process, may be experimentally shown. If ethyl sulphuric acid be heated to 140° C. with sulphuric acid diluted with 3 or 4 mols. of water, and then alcohol added, a little ether is formed together with several sulphonic derivatives, some of which are sufficiently volatile to distil with the ether. 4. The sulphonic acids are produced in greater quantity when the temperature exceeds 140° C., and when the acid is not diluted. Thus, in the preparation of ethylene, when the liquid begins to darken (165° to 175° C.), it may be shown that it contains sulphonic acids in addition to ethylsulphuric acid, neutral ethyl sulphate, polyethylene hydrocarbons, and sulphurous acid. 5. Such a liquid, after dilution and continued boiling to decompose the esters and drive off sulphur dioxide, saturation with baryta, and filtering, leaves on evaporation a residue containing sulphonic derivatives. These may be oxidised by permanganate or nitric acid, and the presence of sulphuric acid shown in the ordinary way. 6. This production of sulphonic acids appears to be not without interest to the theory of the production of ordinary ether and mixed ethers.—A. C. W.

Perfume of Flowers, A New Method for Extracting. J. Passy. Comptes Rend. 1897, **124**, 783—784.

In this process, the flowers are completely immersed in cold water, or preferably in a solution of salt, the liquid being occasionally (when charged with perfume) drawn off and replaced by fresh. From the aqueous solution, the perfume is extracted by means of ether or other volatile solvent. The process has given satisfactory results in the case of certain flowers—notably the lily of the valley—the perfume of which has, hitherto, not been isolated.—H. T. P.

o-Brom-p-ethoxyphenylsuccinimide (Brompyrantin). A. Piutti. Ber. 1897, [10], 1170—1174.

Bromine acts upon *p*-ethoxyphenylsuccinimide at 100°, producing a derivative analogous in constitution to the bromphenacetin described by Hodurek (this Journal, 1897, 352).
—A. C. W.

Coffee, A New Alkaloid from. Forster and Riechelmann. Ph. Zeits. f. offentl. Chemie; through Pharm. Zeit. **42**, 309.

When an aqueous decoction of coffee has been extracted with chloroform and the caffeine thus removed, a body remains which does not give the murexide reaction, but which is precipitated by phosphomolybdic acid. This precipitate is mixed with milk of lime, and dried by the addition of plaster of Paris, in order to avoid the action of heat in the presence of an alkaline liquid. The dry powder is then extracted with alcohol, and the alcoholic solution evaporated to dryness. A brown oily residue results, which crystallises with difficulty, and is stated to be an alkaloid. It differs from caffeine in giving a precipitate with picric acid and in being insoluble in chloroform; also in not giving the murexide reaction.—J. O. B.

Cosaprine: A New Antipyretic. P. Schwarz. Pharm. Zeit. 1897, **42**, 131.

By treating parasulphanilate of sodium with acetic anhydride, the author has obtained a body to which he gives the name cosaprine; it occurs in well-formed white crystals having the constitution $C_6H_4(SO_3Na)NH.COCH_3$. It is readily soluble in water, and is stated to be less toxic than acetanilide, and at the same time an active antipyretic.
—J. O. B.

Eucaine B: A New Cocaine Substitute. P. Silex. Pharm. Centralb. **38**, 103.

This new cocaine substitute is closely allied to eucaine, for which Silex now proposes to retain the name Eucaine A, while he calls his newly produced compound Eucaine B. Eucaine B is benzoylvinylacetone-alkamine.

Though closely related to eucaine, and also to cocaine and tropacocaine, it is much less toxic than the last two. The hydrochloride of Eucaine B is soluble in about 20 parts of water; the solution is not decomposed by boiling, and may therefore be sterilised by heat. (See also this Journal, 1896, 679 and 745.)—J. O. B.

Ecgonine, Solubility of. Oechsner de Coninck. Comptes Rend. **124**, [21], 1159.

The pure alkaloid is insoluble at ordinary temperatures in ether, acetone, petroleum spirit, chloroform, bromoform, benzene, toluene, isobutylic alcohol, ethylene bromide, carbon bisulphide, and turpentine. It dissolves in the following menstrua :—

In distilled water at 17° C.	1 in	4·6	fluid parts.
„ alcohol, 95 per cent., at 17° C.	„	67·0	„
„ acetic ether at 20·6° C.	„	77·0	„
„ rectified methylic alcohol at 19·2° C.	„	18·5	„
„ ethylic alcohol, 66 per cent., at 19·6° C.	„	17·0	„
„ „ 71 „ 19·8° C.	„	21·3	„
„ pure paraldehyde at 17·7° C.	„	133·4	„

—J. O. B.

Iodothyroidine, Method of Preparation of. M. Catillon. Nouv. Remèdes, 1897, **13**, 129.

In a communication to the Société de Thérapeutique the author suggests the name iodothyroidine for a standardised preparation of the active principle of thyroid gland, which he obtains as follows :—The fresh glands are submitted to pancreatic digestion with pancreatine and water ; the residue, after washing with petroleum ether, is dissolved in dilute soda solution and filtered, the filtrate being treated with a slight excess of dilute sulphuric acid, when the active principle precipitates ; this is collected and washed, the iodine content in a portion determined, and sugar of milk added to the rest in sufficient quantity to reduce the amount of iodine present to 0·0003 grm. for each grm. of iodothyroidine. This name is suggested as being more accurate and less confusing than thyroidine, which has been applied loosely to the active principle itself and to its commercial dilutions.

—J. O. B.

Antiarthrine. Thoms. Apoth. Zeit. 1897, **12**, 152 ; through J. Pharm. Chim. [6], **5**, 337.

A product under this name, introduced as a much-advertised secret remedy in Munich, has been found to be in great part composed of salicin, coloured by the addition of an extract or of a vegetable powder, possibly derived from torrefied horse-chestnuts.—J. O. B.

Peronine : A New Sedative. Pharm. Zeit. 1897, **42**, 35.

Peronine, prepared by Merck, of Darmstadt, is the hydrochloride of the benzylic ester of morphine, in which the hydrogen atom of the hydroxyl molecule of morphine is replaced by the alcohol radicle $C_6H_5CH_2$. It is represented by the formula $C_{17}H_{18}NO_2O.C_6H_5CH_2HCl$.

It forms a light white powder, soluble in water and in dilute alcohol, insoluble in ether and in chloroform.

—J. O. B.

"Geosote": Guaiacol Valerianate. Rieck. Pharm. Centralh. 1897, **38**, 56.

The valerianic ester of guaiacol—a yellowish oily liquid boiling between 245° and 265°, and having a specific gravity of 1·037—has been introduced into medicine under the name of "geosote."—J. O. B.

Guaiacol Phosphite and Phosphatol, Preparation of. Ballard. Repert. de Pharm. 1897, [3], **9**, 104.

About 50 grms. of caustic soda, dissolved in 90 per cent. alcohol, are added to 124 grms. of crystallised guaiacol, so that a clear yellowish solution is produced. Into this solution phosphorus trichloride is slowly introduced, by means of a tapped tubular funnel, until the solution is no longer alkaline to phenolphthalein ; the sodium salts formed during the process are filtered off, the filtrate is distilled to remove the alcohol, and the residue extracted with absolute alcohol, which only removes the guaiacol phosphite ; this alcoholic extract is evaporated on the water-bath and the phosphite crystallised out, purified by recrystallisation, drained on filter-paper, and dried over sulphuric acid. It forms a white, crystalline powder, with a sharp, piquant, non-caustic taste, and slight odour. It melts at 77·5°. It is somewhat soluble in water, very soluble in strong alcohol, ether, chloroform, acetone, &c. ; also in fatty oils.

By treating creosote by this method a mixture of the phosphorous esters of the various phenols present is obtained, which the author has termed "phosphatol." It is a thick reddish-yellow liquid, with a slight odour of creosote and a warm taste ; it boils at about 14°, but is not of definite composition.—J. O. B.

Holocaine : A Cocaine Substitute. E. Tauber. J. Pharm. Chim. 1897, **5**, [7], 336. (See also this Journal, 1897, 555.)

This body, obtained by combining molecular proportions of phenacetin and paraphenetidine, is introduced as a substitute for cocaine for producing local anæsthesia. It is para-diethoxy-ethenyldiphenylamidine.

It forms crystals melting at 121°, acting as a strong base, and forming with acids crystalline sparingly soluble salts. The hydrochloride crystallises in needles, soluble to the extent of 2 per cent. in cold water. A one per cent. solution is employed for therapeutic use.—J. O. B.

Red Acaroid Resin of Xanthorrhœa Australis. A. Tschirch and K. Hildebrand. Schweiz. Woch. für Chem. und Pharm. 1897, **35**, 138.

The crude resin consisted of small reddish-brown dusty pieces with a bright fracture, and contained about 10 per cent. of foreign matter, from which it was freed by solution in alcohol. It was then found to be soluble in ether, in ethyl, methyl, and amyl alcohols, in glacial acetic acid, potassium hydrate solution, acetone, and in phenol ; while it was only sparingly soluble in benzene, toluene, chloroform, and carbon bisulphide. The resin was found to contain : (*a*) free paracumaric acid, 1 per cent. ; (*b*) as tannol esters—paracumaric acid, 2 per cent., and benzoic acid, a trace ; (*c*) an aldehyde—para-oxybenzaldehyde—0·6 per cent. ; (*d*) erythroresinotannol, chiefly as paracumaric ester, 85 per cent. Neither cinnamic acid in the free state nor its tannol ester, both of which the authors have found in yellow acaroid resin, were detected in this variety. The formula $C_{40}H_{40}O_{10}$ was determined for erythroresinotannol.—J. O. B.

Yellow Acaroid Resin of Xanthorrhœa Hastilis. A. Tschirch and K. Hildebrand. Schweiz. Woch. für Chem. und Pharm. 1897, **35**, 121.

An examination of this resin has shown the following constituents :—(*a*) Paracumaric acid, 4 per cent., and cinnamic acid, 0·5 per cent. in the free state ; (*b*) as tannol esters—paracumaric acid, 7 per cent. ; cinnamic acid, 0·6 per cent. ; (*c*) also styracin and phenylpropyl-cinnamate (?), 1 per cent. ; para-oxybenzaldehyde and vanillin (?), 0·6 per cent. ; lastly (*d*) xanthoresinotannol, 80 per cent. This last, combined with the paracumaric acid in the form of ester, constitutes the bulk of the resin. The formula $C_{43}H_{45}O_{10}$ is established for this body, which is therefore the homologue of erythroresinotannol, $C_{40}H_{40}O_{10}$.—J. O. B.

Essence of Petit-Grain. J. Passy. Bull. Soc. Chim. 1897, 519.

Tiemann and Semmler (Ber. **25**, 1180 ; this Journal, 1892, 706), and Bertram and Wahlbaum (J. Prakt. Chem. **49**, 590), have pointed out that essence of petit-grain (*Citrus Bigaradia*) consists mainly of linalol, acetate of linalyl and a high-boiling sesquiterpene, but that these constituents do not sufficiently explain its odour. The author finds that it also contains geraniol, partly as the free alcohol and partly as its acetic ether. These contribute to a certain extent to the smell of oil of petit-grain, but there are, in addition, other hydroxy products which have not yet been identified.

—T. A. L.

Castor Oil, Chemistry of. H. Meyer. Pharm. Zeit. **42**, 326.

According to the author, the active constituents of castor oil are ricinoleic and ricinelaidic acids. When pure castor

oil is treated with nitric acid, ricinelaidic acid and ricinelaidin are formed, which, by themselves, are inactive. In solution or in the form of emulsion, they have, however, a marked aperient action. Dioxystearic acid, first discovered by Juillard, is also inert. By heating ricinoleic acid with glycerin in a current of carbonic acid gas to a temperature of 280°—300° C., an almost pure triglyceride is formed, which, in viscosity, taste, and aperient action, is exactly similar to the natural oil. It dissolves in an equal volume of alcohol and has a specific gravity between 0·959 and 0·984. This pure triglyceride may be regarded as synthetic castor oil.—J. O. B.

Alcohol, Denaturing of. E. Barillot. Comptes Rend. **124**, [21], 1163.

See under XVII., *page* 626.

Alkaloids, Action of Tannin and Gallic Acid on certain. O. de Coninck. Comptes Rend. 1897, **124**, 773.

See under XXIII., *page* 639.

Production of Camphenol from Camphor. J. E. Marsh and J. A. Gardner. Proc. Chem. Soc. 1897, [181], 137.

The authors have described (Trans. 1897, **71**, 285) the production of an isomeride of camphor—camphenol. This substance was obtained by the action of strong sulphuric acid on chlorocamphene, $C_{10}H_{15}Cl$. Camphenol is produced by the action of the same reagent on camphene dichloride, $C_{10}H_{16}Cl_2$, which is the immediate product of the action of phosphorus pentachloride on camphor. The same camphenol is apparently produced from both the isomerides of the formula $C_{10}H_{16}Cl_2$, obtained from ordinary camphor, and a satisfactory yield is obtained in both cases. The action of strong sulphuric acid on other chloro-derivatives of terpenes has been examined. In particular, turpentine dihydrochloride behaves in a manner very similar to the camphor derivative, but the nature of the product of the reaction has not yet been determined.

Essential Oils, A New Product for Reducing. Extract from the British Pharmaceutical Journal, through U.S. Cons. Reps., June 1897, 213.

This is a volatile oil offered for sale on the London market. It has a "fancy" name, is stated to be of vegetable origin, and to be specially manufactured for the purpose of reducing essential oils. It is also said to combine perfectly with both Sicilian and French essential oils. With the view of ascertaining its properties, a pint sample of the oil was obtained and submitted to analysis. The following results were arrived at: Specific gravity at 15·5° C., 0·869; optical rotatory power in tube at 200 mm., 59°; flashing point (Abel's method), 100° F.; solubility in three volumes of alcohol of specific gravity 0·82°, fairly soluble; residue on evaporation, trace.

Fractional Distillation. — Percentage yielded between 155° and 160° F., 3·5; 160° and 165° F., 55; 165° and 170° F., 21; 170° and 180° F., 9; above 180°, 8·5.

There was no aldehyde present, and only a trace of the presence of ester could be discovered when the oil was subjected to saponification.

From the results obtained, this oil appears to have the characters of a levopinene, such as is obtained from *Pinus sylvestris, Abies excelsa, Pinus maritima,* &c. The oil has a delicate odour not unlike that of some of the commercial varieties of pine oils, and experiment shows that considerable proportions of it may be mixed with oils of lemon and bergamot without the presence of the adulterant being detectable to smell or taste. Such detection would also be difficult by any of the ordinary physical tests (specific gravity, rotation, &c.) which are applied to essential oils; and the importance of having standards, such as the percentage of citral in lemon oil and of linalool acetate in bergamot, is emphasised by a consideration of the impunity with which adulteration can otherwise be conducted.

PATENTS.

Vanilline, Process for the Manufacture of, from Isoeugenol and Eugenol. H. Imray, London. From Haarmann and Reimer, Holzminden, Germany. Eng. Pat. 14,928, July 6, 1896.

Vanillin is prepared from isoeugenol by treating the boiling alkaline solution with peroxides, such as sodium peroxide, or other bodies giving off oxygen in the presence of excess of alkali; on cooling and acidulating, vanillin may be washed out with ether. Vanillin may be prepared direct from eugenol by this method by first converting it into isoeugenol, according to De Laire's Eng. Pat. 17,547 of 1890 (this Journal, 1891, 854), and then oxidising the latter as described above.—J. O. B.

Thyroid Gland, Process for the Production of the Active Constituents of. O. Imray, London. Eng. Pat. 16,137, July 21, 1896.

Thyroid glands are reduced to a finely-divided state by chopping, and extracted with a solution of sodium chloride (0·75 per cent.), in which menstruum the active principles are soluble; after separation from the inert matter by mechanical means, the solution is treated with a 10 per cent. solution of tannin, which completely precipitates the active principles. The precipitate is then dried and used as a medicament. It contains about 0·4 per cent. of iodine.—J. O. B.

Vanillin, Impts. in the Manufacture of. G. B. Ellis, London. From La Société Chimique des Usines du Rhone, anciennement G. P. Monnet et Cartier, Lyons. Eng. Pat. 16,239, July 22, 1896.

The process consists, first, in the preparation of carboxylic esters, especially the methyl carboxyl ester of protocatechuic acid, by the action of the methyl or other ester of chloroformic acid upon the potassium or sodium salt of protocatechuic aldehyde. The ester thus produced, which melts at 98°—99° C., is then methylated by means of alcoholic potash and dimethyl sulphate at ordinary temperatures, and finally hydrolysed by means of dilute mineral acids. Upon distilling off the alcohol, vanillin is left in the residue, from which it is extracted with ether. By this method, the simultaneous formation of a large amount of isovanillin is avoided.—J. O. B.

Drying Substances [containing Aromatic Oils], Improved Process for. H. Humbser, Fürth, Bavaria. Eng. Pat. 6065, March, 8, 1897.

See under XVII., *page* 627.

XXI.—PHOTOGRAPHY.

PATENT.

Photographs with Coloured Effects, Impts. in, and in Methods of Producing the Same. G. J. Sershall, Birmingham. Eng. Pat. 3121, Feb. 5, 1897.

The photograph with coloured effects consists of a transparent front, preferably of celluloid, carrying a photographic film to form the keystone of the coloured picture, and a colour-printed backing of paper, cardboard, or the like, developed by any well-known mechanical process of colour printing; the two being fixed permanently together with the film side of the transparent front and the coloured surface of the backing adjacent to each other. A number of single photographs may be thus arranged in a sheet, which may be registered and cut out.

In practice, a negative is first taken of any number of photographs. From this a sheet of carbon tissue is taken, which is developed upon a sheet of transparent celluloid, ivory, gelatin, or talc, the result being a number of pictures on one side of the transparent sheet. A sheet of transparent gelatin is then fixed to this sheet with registering pins, and a tracing taken with a sharp-pointed instrument of the outlines of the pictures and of those parts to be developed by colouring matter. The tracing is removed, treated with powdered blacklead, and rubbings

down, or impressions are taken from it upon stones sufficient to comprehend all the colours required in the backings. The traced parts of the stones are then drawn and coloured, and the backings are printed from them in the ordinary way. The transparent films and the coloured backings are then pressed together with any transparent adhesive material, and thus form the coloured photograph. —J. O. B.

XXII.—EXPLOSIVES, MATCHES, Etc.

Sodium Acetylide and Carbide, Thermal Properties of. C. Matignon. Comptes Rend. 1897, **124**, 1026—1028.

The heats of formation of the above compounds are as under:—

	Cal.
C_2 (diamond) + H + Na (solid) = C_2HNa (solid) .	− 29·2
C_2 (diamond) + Na_2 (solid) = C_2Na_2	− 8·8

Also—

C_2H_2 (gaseous) + Na = C_2HNa (solid) + H	+ 28·9
C_2H_2 (gaseous) + Na_2 = C_2Na_2 (solid) + H_2	+ 49·3

Sodium acetylide and carbide are therefore endothermic compounds. They are decomposed, with separation of carbon, when incautiously heated; and furnish, in combination with oxidising agents, highly explosive mixtures, most of which are very sensitive to friction.—H. T. P.

Sodium Acetylide and Carbide, The Preparation of: C. Matignon. Comptes Rend. 1897, **124**, 775—777.

The following processes are based on the observation that when fused sodium acts on acetylene, the sole product at temperatures below 190° C. is sodium acetylide, whilst only sodium carbide is formed at temperatures exceeding 210°—220° C.:—

Sodium Acetylide, $NaHC_2$.—Pure, dry acetylene is passed into a large flask containing metallic sodium, heat being applied by means of an oil-bath maintained at 180° C. The contents of the flask must be frequently agitated, in order to break up the crust of acetylide and to expose a fresh surface of sodium to the gas; and the action should be allowed to continue for some time after the mass has lost all metallic appearance, in order that every trace of sodium may be converted into acetylide.

Sodium Carbide.—The method is, in general, the same as above; but the oil-bath must be rapidly heated to 220° C., and maintained at 220°—230° C. Any acetylide formed below 200° C. is again decomposed at the higher temperature, either under the influence of heat alone, or by the action of the excess of sodium, into carbide. A white, very pure carbide is obtained by this process.—H. T. P.

PATENTS.

Fuse or Quick-match, An Improved Safety. C. Lamm, Blackburn. Eng. Pat. 14,320, June 29, 1896.

Two kinds of fuse are described: one consisting of a twist or core of nitrocellulose, or mixed nitrocellulose and cellulose, which has been impregnated with a solution of nitrate and chlorate of potassium, containing, in addition, either yellow prussiate, picrate, or carbonate of potassium. This core is then immersed in a bath of ether-alcohol, nitrobenzene, or other suitable solvent, until the nitrocellulose is gelatinised. After drying, the twist may be employed directly as a fuse, or can be insulated. The other consists of a core of cellulose, which, after boiling in a solution of chlorate and ferrocyanide of potassium, and drying, is surrounded with jute yarn and made waterproof. —W. M.

Explosives, Impts. in or relating to. M. Van Look, Cologne. Eng. Pat. 6461, March 11, 1897.

The inventor proposes to make an explosive by charging a cartridge or shell with calcium carbide and water held in separate receptacles. The explosion is brought about by causing the fracture of the water vessel, when the reaction between the water and carbide generates gas.—W. M.

Explosive Compounds, Impts. in the Manufacture of. G. B. Ellis, London. From La Société Chimique des Usines du Rhone, Lyons, France. Eng. Pat. 15,022, July 7, 1896.

Finely ground coke is treated with desulphurised tar which has been neutralised by the addition of magnesium carbonate and then dried and ground so as to form a pulverulent carbonaceous material, which is then mixed by sifting with finely ground chlorate or perchlorate of potassium. A still safer explosive can be made by previously treating the chlorate with a dilute solution of shellac or purified tar. Suitable proportions are: "80 parts of chlorate or perchlorate of potassium, $5\frac{1}{2}$—$7\frac{1}{2}$ parts of tar, 12—14 parts of charcoal or coke, $\frac{1}{2}$—1 part of carbonate of magnesia or lime."—W. M.

XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

Polariscope, Yellow Sodium Light for the. F. Dupont. Bull. Assoc. Chim. **14**, 1041.

Difficulties are often encountered in using yellow light for polarimetric observations. The author finds that a mixture of sodium chloride and tribasic sodium phosphate, melted together in approximately molecular proportions, answers perfectly. This mixture melts more easily than common salt, never decrepitates, and imparts to the flame a brilliant yellow colour, so that observations thus become extremely easy and precise.—L. J. de W.

Gas-Analysis Apparatus. O. Bleier. Ber. 1897, [10], 1210—1211.

In order to obviate the inclusion of air in connecting the author's pipettes (this Journal, 1896, 616) with the burette figured in this Journal, 1896, 294, which see, it is proposed to replace the tap *a* (Fig. 1, *loc. cit.*) by a two-way cock, which will connect a horizontal capillary on the tap with the interior of the burette or with a cup above the tap. The pipette is connected to this horizontal capillary, and the absorbent is made to flow into the tap before connection is made between burette and pipette; after the absorption, the liquid is again led into the tap; this may be accomplished by means of a screw clip on the rubber connection. If mercury be used, the three-way cock *b* (*loc. cit.*) may be omitted and the capacity of the burette reduced to 25 c.c. (the length remaining the same) without loss of accuracy. The size of the pipettes may be diminished in the same degree.

In exact gas analysis the use of such a two-way tap to effect connection between burette and pipette without the introduction of air, considerably simplifies the apparatus. In the Hempel apparatus the mercury bath may be dispensed with, the measuring bulb fixed, and solidly connected with the water jacket and pressure tube.—A. C. W.

PATENT.

A Photometer for Automatically Measuring and Registering the Intensity of the Light of a Gas Flame, and Apparatus therefor. W. and B. Cowan, Ltd., Westminster. From C. V. L. Martenson, Rio de Janeiro. Eng. Pat. 9041, April 9, 1897.

The principle on which this invention is based is that if a gas flame under constant pressure be allowed to impinge upon an inclined surface, given horizontal movement in such a direction that a line upon it, perpendicular to a line of greater incline, is maintained at a certain level during the motion on or over a vertical line passing up through the centre of the flame, the smoke deposited by the flame will produce a characteristic impress. The lower part of the impress will rise or fall according to the greater or less illuminating power of the gas, the width of the impress varying in direct proportion to the illuminating power.

The apparatus is constructed within a case, to shelter the flame from all draughts. The flame impinges upon a cone of incombustible material mounted on a vertical axis, to which a uniform rotatory motion is imparted by suitable mechanism. The rate may be one revolution in 24 hours,

or one per week, the cone having its surface marked off into hours. There is thus automatically obtained a diagram recording the variations in the quality of the gas. The sides of the cone may be either straight, slightly concave, or slightly convex; or it may be replaced by a cylinder mounted on an oblique axis. The angle of the oblique axis is controllable by means of a sliding block, which can be operated by a screw outside the apparatus.

The four claims embrace the general method of automatically testing the gas, and various details of the constructions shown.—H. B.

INORGANIC CHEMISTRY.—QUALITATIVE.

Lead Chromate in Coloured Wrappers, Rapid Method of Detecting. J. Wolff. Annal. Chim. Analyt. 1897, **2**, 105.

Lead chromate is frequently employed on the Continent as a pigment for the yellow paper which is used as wrappers for chocolate and other sweetmeats. Its use for this purpose is obviously objectionable.

The presence of the lead pigment is readily shown as follows:—A square of about 5 cm. of the suspected paper is placed, without folding, in a small flat-bottomed capsule, and moistened with alcohol, 90 per cent.; when it is thoroughly saturated, the excess of alcohol is drained off, and a few drops of nitric acid are allow to flow over the surface of the paper. If chromate be present, the characteristic odour of aldehyde will be given off, and the paper will be coloured green. The nitrate of lead formed during the reaction is dissolved in 10 or 15 c.c. of water, and if too great an excess of acid has not been used, the characteristic precipitate of lead iodide may be obtained at once from the decanted solution. Green papers, coloured with a mixture of lead chromate and Prussian blue, may be tested in a similar manner. The author regards zinc chromate as inoffensive as a pigment for these articles.
—J. O. B.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Illuminating Gas, Determination of Oxygen in. O. Pfeiffer. J. für Gasbeleucht. 1897, **40**, 354—357.

In gas analyses carried out over water, the error of observation is not less than 0·2 per cent, and therefore the ordinary determinations of oxygen in coal-gas are of doubtful value. When oxide of iron is continuously revivified *in situ*, it becomes necessary to have a better method of ascertaining the amount of oxygen or air in the gas. Several likely schemes having been tried and found worthless, a colorimetric method was finally found satisfactory.

100 c.c. of the gas to be examined are taken in a Bunte burette, and the water within is then drawn out by suction until it only fills the capillary of the lower cock. 5 c.c. of solution of caustic potash (1 to 2) is then passed up through this cock. The funnel at the top of the burette having been emptied, 0·2 grm. of pyrogallol is put in it and 2 c.c. of water added. The solution thus formed is admitted to the burette, with the exception of one drop, which seals the capillary. The burette is agitated for 5 minutes, and water is then admitted through the lower cock until it reaches a predetermined mark—*e.g.*, the zero—on the burette. After the lapse of two minutes, the colour of the liquid in the burette is compared with that produced in the same volume of water in a wide test glass by the addition, drop by drop, of a caramel solution of known strength. The number of drops required to bring the water to the same colour as the liquid in the burette, corresponds to a certain amount of air. This relation is ascertained empirically.

The caramel solution is made by dissolving 30 grms. of cane sugar in a litre of boiling water, adding thereto 30 c.c. of sulphuric acid (1 to 3), and boiling for $\frac{1}{4}$ hour. Then 85 c.c. of solution of caustic soda (sp. gr. 1·30) are added gradually, and boiling is continued for another $\frac{1}{4}$ hour. If kept in the dark, the solution thus made does not change in colour. It corresponds in tone to the gallotlavin produced by the pyrogallol reaction in the burette. Samples of gas containing known percentages of air are then treated precisely as just described, and the number of drops of the caramel solution which give the requisite colour in each case is ascertained. From values so found, a curve is drawn, from which may be deduced the percentage of air corresponding to any number of drops of the caramel solution.

The oxygen in the small quantity of water admitted to the burette may generally be ignored, as its amount is small and nearly constant for all the tests. In some cases—such as when the presence of oxygen merely is in question—it is, however, desirable to use water free from oxygen. The small quantity of water in a test glass may be deoxygenated by boiling, but even prolonged boiling of larger quantities fails to remove entirely the oxygen. Phosphorus removes the oxygen, but imparts its odour to the water. The water for the purpose is best deoxygenated by contact with zinc, of which the activity has been enhanced by immersion in water containing a few drops of solution of copper sulphate. After washing, the zinc is put in a Woulff's bottle, which is filled with water saturated with coal-gas. After bubbles of gas and air have been liberated, the water will in a few hours be free from oxygen, and may be drawn off for use through the longer limb, while more water is admitted to the bottle through the shorter limb. A continuous supply of water free from oxygen may be thus obtained.—J. A. B.

Chlorine and Bromine, Separation and Estimation of. H. Baubigny and P. Rivals. Comptes Rend. **124**, 859—862.

Potassium permanganate decomposes cupric bromide in the cold with liberation of bromine, while it does not act on cupric chloride. To apply this in analysis, the mixed alkali chloride and bromide (0·2 to 0·4 grm.) are carefully weighed, dissolved in water with 4—8 grms. of copper sulphate crystals, and to the cold solution is added 0·35 to 0·40 grm. of potassium permanganate. After solution, the whole is evaporated to dryness in a wide, shallow vessel, *in vacuo*, over caustic potash at the ordinary temperature. After 24 hours the residue is taken up with water, sulphurous acid added to reduce remaining permanganate or manganese dioxide, and the chlorine precipitated by silver nitrate containing excess of nitric acid. The mode of calculation is obvious.

Results of test experiments with known quantities of alkali chlorides and bromides are given, proving the accuracy of the method. Where chloride alone was used, the evaporation with permanganate was twice repeated, to exaggerate the possibility of decomposition of the chloride.

Substituting aluminium sulphate for copper sulphate in the method as described, a considerable loss of chlorine always takes place.—J. T. D.

Cyanogen, Notes on the Estimation of, by Silver Nitrate. [*Potassium Iodide and Ammonia as Indicators.*] W. J. Sharwood. J. Amer. Chem. Soc. 1897, **19**, [5], 400—434.

5 c.c. of ordinary ammonia (Liq. Ammon.) and 2 c.c. of a 5 per cent. potassium iodide solution are added to 25—100 c.c. of the solution in which cyanogen is to be determined, and the liquid is titrated with standard silver nitrate (1·305 grms. of cryst. $AgNO_3$ in 100 c.c. of water) till a faint permanent cloud forms. For rapid approximate determinations, 10 c.c. of the sample should be taken and 1 c.c. each of ammonia and iodide solution. If sufficient ammonia be already present in the solution, no more should be added; if a large amount be present, a larger quantity of iodide is added to reduce the error. A larger addition of iodide will also partly remove the error due to the presence of thiosulphates. If sulphides be present in small amount, the potassium iodide is replaced by 5 or 10 c.c. of a solution of 0·5 grm. of iodine and 2—3 grms. of potassium iodide in 100 c.c. of water, the mixture allowed to stand five minutes, and, after addition of ammonia, titrated. The result is corrected by adding the difference between two parallel tests (1) on some pure dilute potassium cyanide solution, and (2) on an equal amount of potassium cyanide *plus* 5 or 10 c.c. of the iodine solution. If the amount of sulphide be large, or if other reducing agents be present, twice the usual volume of solution is taken, soda and then sodium plumbite in very

slight excess added, the mixture well shaken, made up to a definite volume, filtered, and one-half of the clear filtrate used for titration, rejecting the first few c.c. filtered. In presence of zinc, a considerable excess of caustic soda or potash is added before titration and the final precipitate tested with more alkali; if it dissolves, more alkali is added and the titration continued; if insoluble, the cyanogen found represents that contained in both potassium zinc cyanide and free potassium cyanide. The free potassium cyanide may be rapidly and roughly determined by adding a few drops of a 5 per cent. solution of potassium ferrocyanide and titrating with silver nitrate until a faint flocculent precipitate forms (using a c.c.), then adding ammonia, potassium iodide, and soda, and titrating for total cyanogen (using b c.c. altogether). Then, according to Bettel, $a - 0{\cdot}086\ (b - a)$ represents the cyanogen as free cyanide, and $(b - a) \div 0{\cdot}921$ gives the cyanogen present as double cyanide, if free alkali were originally absent; the error and correction is increased if free alkali were originally present, and varies slightly with the time occupied in titrating. The free cyanide may be more accurately determined by acidulating a portion of the solution with hydrochloric acid, evaporating off the hydrocyanic acid, estimating zinc by standard ferrocyanide, and subtracting four equivalents of potassium cyanide for each atom of zinc found.

In presence of copper, the cyanogen present as double copper cyanide is obtained by estimating the copper colorimetrically with ammonia, after expelling hydrocyanic acid with nitric or sulphuric acid, and allowing 3—4 equivalents of cyanogen for each atom of copper. In presence of calcium, magnesium, or manganese, ammonium chloride is added before titration, and in presence of aluminium or lead, caustic soda. The quantities of gold, silver, and other interfering substances present, are usually too small to appreciably affect the results.

With impure solutions, the results obtained by this method are more accurate than those given by the ordinary Liebig method; with pure solutions, the results are practically identical, but the end reaction is sharper and an estimation can be more rapidly carried out. 170 grms. of $AgNO_3$ correspond to 52 grms. of cyanogen.—A. S.

[*Alkali Chlorides in Presence of Iodides, &c.*] *Quantitative Analysis by Means of the Telephone, A Simple Method for.* H. Erdmann. Ber. 1897, [10], 1175—1182.

Normal solutions of very similar electrolytes have approximately the same conductivity. Hence, in solutions of equal percentage strength, the resistance is nearly proportional to the molecular weight of the dissolved substance. For example, Bouty found the resistances of 5 per cent. solutions of potassium chloride, bromide, and iodide to be in the ratio 1:1·47:2·13, whilst the ratio of the molecular weights of these salts is 1:1·60:2·23. In consequence of the great differences in the conductivities of such solutions, the amount of potassium chloride contained in a mixture with the bromide or iodide may be quantitatively determined. For purposes in which absolute measures of conductivity are not required, but only comparative values, the physical method of determining conductivities may be greatly simplified for the analytical process. The apparatus consists of two cylindrical vessels 9 cm. high and 4 cm. in diameter, in which are contained the electrodes—platinum plates covered with platinum black (see "Physico-chemical Measurements," Ostwald; translated by McGowan), placed about 2 cm. apart. The two cells are contained in a covered vessel full of water and provided with a stirrer. The current is obtained from a small induction coil worked through a resistance of 0·6—0·7 ohm by a Bunsen's or Grove's cell. The ends of a Wheatstone's bridge are connected to the cells and to the induction coil; the telephone is placed on the wire connecting the junction of the two cells with the sliding contact on the bridge wire.

One per cent. solutions of salts or mixtures are employed. These should be made up in graduated flasks of Jena glass which have been well boiled with water. The water employed for the solutions need not be distilled through a condenser with a tin tube. For the details of the process, the analysis of a mixture of potassium chloride and bromide is given. 10 grms. of the pure chloride are dissolved in water and the solution made up to 1 litre; the two cells and electrodes are washed with this solution, 50 c.c. is then placed in each cell, the circuit closed, and the sliding contact adjusted until the minimum of sound is perceptible in the telephone. After a few minutes, to allow equalisation of temperature, the position is constant; successive readings will not differ by more than 0·2 mm.; the mean of two or three readings is taken. One cell is now emptied, washed out with 1 per cent. potassium bromide solution, filled with 50 c.c. of this solution, and readings taken as before. In the same way this cell is filled with 1 per cent. solutions of unknown composition and readings taken. If A be the reading in millimetres on the scale 1 m. long, r and r' the resistances in the two cells, then the ratio of these resistances—

$$v = \frac{r}{r'} = \frac{A}{1000 - A}$$

The following results were obtained with solutions of known composition:—

KCl : KBr.	A.	v.	KBr taken.	KBr found.
			Per Cent.	Per Cent.
100 : 0	651·02	1·86518	0·00	..
99 : 1	650·60	1·86180	1·00	0·53
1 : 1	606·96	1·54472	50·00	50·31
2 : 3	597·32	1·48324	60·00	59·96
1 : 9	563·27	1·28962	90·00	90·38
0 : 100	551·24	1·22820	100·00	..

The results of determinations of potassium iodide in mixtures with the chloride were more exact. This method of analysis might with advantage be used where a large number of determinations have to be carried out in a short time.—A. C. W.

Magnesium Sulphate, An Adulteration of. P. A. Lamanna. Boll. Chim. Farm. **36**, 198; Chem. Central-Bl., 1897, 1100.

The author has observed a case of adulteration of magnesium sulphate with zinc sulphate; this may be detected by the addition to an aqueous solution of the salt, of one drop of potassium ferricyanide solution, which causes a brownish-yellow precipitate.—A. C. W.

Silicic and Tungstic Acids, Note on the Separation of. J. S. de Benneville. J. Amer. Chem. Soc. 1897, **19**, 377—379.

This note calls further attention to the fact already pointed out by several observers, that in the method of determining tungsten by obtaining a mixed residue of tungstic acid and silica and dissolving the tungstic acid out of this by ammonia, a part of the silica is also dissolved by the ammonia. On evaporating the ammoniacal solution to dryness, igniting, and digesting with hydrofluoric acid, with or without the addition of a few drops of sulphuric acid, the silica can be expelled and the tungstic acid obtained pure. In determining silicon in tungsten alloys, it is also important to bear in mind the fact that a part of the silica is dissolved by ammonia. A table is given which shows that in a ferro-manganese containing nearly 7 per cent. of tungsten, several evaporations with hydrofluoric acid were sometimes necessary to remove the whole of the silica from the mixed oxides of silicon and tungsten.—H. S. P.

Silica, Determination of, in Blast-Furnace Slag. G. H. Meeker. J. Amer. Chem. Soc, 1897, **19**, 370—374.

The author advocates the use of sulphuric acid for decomposing blast-furnace slags in the determination of the silica contained in them, in preference to hydrochloric acid, because he finds that slags generally, and especially some special kinds of slag containing spinel (magnesium aluminate) and certain complex titanium compounds, are more perfectly and rapidly decomposed, and further, that the silica is more quickly dehydrated and rendered insoluble by sulphuric acid than by hydrochloric acid.

His method of procedure is as follows:—"one-half grm. of finely ground slag is placed in a four-inch evaporating basin, covered with cold water (about 3 c.c.), and stirred to break up lumps. To the contents of the basin 10 c.c. of concentrated hydrochloric acid are then added, stirring vigorously to prevent sticking to the bottom.

"As soon as the slag seems dissolved as much as it will, and before it has set to a jelly, pour in 40 c.c. of sulphuric acid (one volume concentrated acid to one volume water), stir well, wash off stirring rod, and then cover solution with a funnel. The funnel should have fused edges and be of such size as to rest upon the sloping inside of the evaporating basin and set down above the solution.

"Place the evaporating basin on a wire gauze and boil rapidly until fumes of sulphur trioxide begin to come from under the edges of the funnel. Cool, dilute somewhat, and clean off the funnel. Add about 10 c.c. of hydrochloric acid and dilute until the basin is as full as it can be conveniently. Cover with watch-glass and boil gently for one minute. Filter at the vacuum pump. Wash five times with hot hydrochloric acid (sp. gr. 1·10) and five times with hot water. Burn and weigh. The silica is remarkably pure. On 'chilled' slags the impurities are from 0·05 to 0·20 per cent."

A table of results is given comparing this method with a method consisting of twice evaporating the slag to dryness with hydrochloric acid at a temperature not exceeding 120° C., and finally purifying the silica with hydrofluoric acid, which shows a very good agreement between the two methods for ordinary slags; but in the case of a slag containing spinel and other compounds not decomposed by hydrochloric acid, the determination of the silica by the hydrochloric acid method was about $1\frac{1}{2}$ per cent. too high.

The author also refers to the known fact that slags containing spinel are only decomposed with great difficulty by fusion with sodium carbonate, and points out that his method, owing to the readiness with which it decomposes such slags, has therefore a distinct advantage.—H. S. P.

Lead, Volumetric Determination of. J. H. Wainwright. J. Amer. Chem. Soc. 1897, **19**, 389—393.

This is a modification of the method of volumetric determination of lead by a standard solution of potassium bichromate, using as an indicator spots of silver nitrate. The method must be carried out exactly under the conditions specified, which is described in the case of litharge as follows:—1·00 to 1·25 grms. is dissolved in 10 to 15 c.c. of nitric acid (sp. gr. 1·20), the solution neutralised with ammonia in excess, and a considerable excess of acetic acid added. It is then boiled, and the standard solution of potassium bichromate run into the boiling liquid in sufficient quantity to precipitate all the lead. The liquid is then again brought to the boil, and the addition of bichromate solution then continued at the rate of about half a c.c. at a time, stirring well between each addition, until the lead chromate settles suddenly to the bottom, leaving the liquid clear. This occurs usually about 1 c.c. from the final quantity of bichromate required. The addition is then continued drop by drop, testing the clear supernatant liquid between each addition by mixing a few drops of it on a white porcelain plate with a drop or two of silver nitrate until a distinct red colour of silver chromate appears.

The chief points to be observed are:

1. The solution of the lead salt should be as concentrated as possible, and decidedly acid with acetic acid.

2. It should be free from other metals, especially such as may exist in the lower forms of oxidation. Antimony and tin must be oxidised by repeated evaporations with fuming nitric acid. Bismuth must also be absent.

3. The solution of lead must be kept as near the boiling point as possible.

4. The strength of the bichromate solution should be such that 1 c.c. approximately equals 0·01 grm. of lead. It may be standardised with pure lead or a pure "white lead," in which the lead has been accurately determined gravimetrically.

5. The solution of silver nitrate should be dilute—not more than 2 to 3 per cent.

Results by this method agree to within 0·2 to 0·3 per cent. with the gravimetric, in which the lead is weighed as lead sulphate.—H. S. P.

Lead, Determination of, in Lead Ores. R. K. Meade. J. Amer. Chem. Soc. 1897, **19**, 374—377.

One grm. of the ore, if rich—more, if poor—is treated in a platinum dish covered by a watch-glass, with 10 to 50 c.c. of a mixture of one part of sulphuric acid (1·84 sp. gr.) and three parts of nitric acid (1·42 sp. gr.). Heat is applied on a water-bath or hot-plate until the action of the acids on the ore has apparently ceased. The watch-glass is rinsed and removed, and 10 to 15 c.c. of hydrofluoric acid are added, and the solution evaporated until fumes of sulphuric acid come off. The liquid is then cooled, diluted to about 100 c.c. with water, digested until all the soluble salts are dissolved, the precipitate of lead sulphate filtered and washed, first with a 2 per cent. solution of sulphuric acid and then with alcohol, after which it is dried, separated from the paper, ignited in the usual way, and weighed.

The method is, of course, not applicable in presence of barium, strontium, and calcium, or of metals that are converted into insoluble oxides by nitric acid.—H. S. P.

Lead, Estimation of, in Slags and other By-Products. D. J. Williams. Chem. and Metall. Soc. of S. Africa, Proc. of Meeting, May 15, 1897, 11—14.

Three volumetric methods for the rapid estimation of lead in slags are compared with a standard gravimetric method. In every case the ore was finely crushed in an agate mortar. In the first case, 1 grm. of the sample was boiled with 5 c.c. of HCl and 5 c.c. of HNO_3, cooled, and then evaporated with 15 c.c. of H_2SO_4. When sulphuric acid fumes were evolved, the liquid was diluted, filtered, and washed as usual. The precipitate was then transferred to a beaker and dissolved in ammonium chloride, the residue being extracted with 200 c.c. of strong caustic soda, and the last traces of lead so removed, precipitated with sulphuric acid, and transferred to the beaker containing the bulk of the lead. A few c.c. of nitric acid were now added, then a few drops of phenolphthalein and an excess of caustic soda, whereupon the lead was precipitated with pure oxalic acid. After filtering and washing with cold water, the precipitate was rinsed into a flask containing 70 c.c. of hot water; a few c.c. of strong sulphuric acid were added, and the liberated oxalic acid was determined by standard permanganate solution. Each determination required 45 minutes, and the mean of 10 tests showed an error of 0·4 per cent.: bismuth and antimony are interfering metals. In the second process, the lead was obtained as sulphate, as before, and this was then transferred to a beaker and mixed with 50 c.c. of a saturated ammonium carbonate solution. After boiling for a few minutes, filtering, and washing with hot water, the precipitate was dissolved in acetic acid, and titrated with standard ferrocyanide solution. Such a determination may be made in an hour, and may be correct to within 0·7 per cent. The third process is that described by Alexander (see this Journal, 1893, **12**, 376); it requires only 25 minutes, and should be correct to within 0·15 per cent. The details of the method as used by the author are as follow:—A weighed quantity is heated on the hot plate with 20 c.c. of H_2SO_4 until decomposed. After cooling, 20—30 c.c. of HNO_3 are added; the mixture is now heated until fumes of sulphuric acid are evolved. After cooling, it is diluted to about 75 c.c. with cold water, and boiled to dissolve any sulphates. The liquid is filtered, leaving as much as possible of the precipitate in the beaker, and washed with dilute sulphuric acid. The precipitate is then dissolved in ammonium acetate solution, and made up to $\frac{1}{4}$ or $\frac{1}{2}$ litre, and titrated with standard ammonium molybdate solution, with a freshly made solution of tannin as an indicator, using drops of the liquids on a porcelain plate for the test. The solution need not contain free acetic acid, and ammonium chloride may be substituted for the acetate. Although arsenic and antimony do not interfere with this method, large quantities of lime may prove prejudicial.—W. G. M.

Nickel from Cobalt and Iron, Cobalt from Aluminium; Separation of. E. Piñerúa. Comptes Rend. **124**, 862—863.

Nickel chloride and aluminium chloride are insoluble in ether saturated at a low temperature with hydrochloric acid, while the chlorides of cobalt and of iron dissolve in it readily.

The mixed chlorides of nickel, cobalt, and iron are dissolved in as little water as possible, a mixture (about 20 c.c. to 0·3 grm. of the chlorides) of equal volumes of ether and fuming hydrochloric acid is added, and the whole, in a bath of salt and ice, saturated with hydrochloric acid gas. The nickel separates as a heavy yellow crystalline precipitate, which is washed, first by decantation, and then on the filter, with ether saturated with hydrochloric acid, and then converted into one of the ordinary forms in which nickel is weighed. Cobalt remaining in solution gives a deep blue colour to the liquid, iron a green; small quantities of iron perceptibly change the blue of the cobalt solution; hence this method affords a ready qualitative test for the other metals in reputedly pure salts of cobalt or of nickel.

Where much iron is associated with nickel, the precipitate retains traces of iron, and should therefore, after washing, be redissolved and reprecipitated.

Exactly the same method is pursued to separate cobalt from aluminium. In all these cases the method gives very exact results.—J. T. D.

Phosphor Bronze, Phosphor Copper, Phosphor Tin, &c.; Analysis of. M. Wickhorst. J. Amer. Chem. Soc. 1897, **19**, [5], 396—398.

Estimation of Phosphorus alone.—1 grm. of the sample is treated with 20 c.c. of aqua regia (15 of HNO_3 to 5 of HCl) and the mixture warmed. When the reaction is complete, water is added, then excess of ammonia, and the whole made up to 200 c.c. The copper, lead, &c. are precipitated by sulphuretted hydrogen, the sulphides filtered off, and the phosphorus precipitated in 100 c.c. of the filtrate by adding 10 c.c. of magnesia mixture and ammonia. After standing a few hours, the precipitate is filtered off into a Gooch crucible, washed with dilute ammonia containing a little ammonium sulphide, ignited, and weighed as magnesium pyrophosphate. If a very accurate determination be required, the precipitate is dissolved in dilute hydrochloric acid and reprecipitated with magnesia mixture and ammonia.

Complete Analysis.—0·5 grm. of the sample is treated with 5 c.c. of strong nitric acid, and the mixture heated until the reaction is complete. The residue is well rubbed with a glass rod, and after adding a little water, the insoluble matter is filtered off on to a small filter and washed with water containing a little nitric acid. The moist filter is placed in a weighed porcelain crucible, and the whole heated, gently at first, and finally with the blowpipe. The residue consists of stannic oxide and nearly all the phosphorus as pentoxide. The latter is estimated by fusing with 0·5 grm. of Na_2CO_3 and 1 grm. of S, keeping the lid on the crucible. Heat is applied until the excess of sulphur is driven off, after which the mass is allowed to cool, dissolved in water, excess of ammonia and 1 grm. of ammonium chloride added, and the phosphorus precipitated with magnesia mixture. The precipitate is collected on a Gooch filter, washed with dilute ammonia containing a little ammonium sulphide, dissolved in dilute hydrochloric acid, reprecipitated with ammonia and magnesia mixture, filtered, ignited, and weighed. The difference between the weights of phosphorus (calculated to pentoxide) and of the total insoluble residue gives the amount of stannic oxide, from which the percentage of tin can be calculated. A small amount of phosphorus is dissolved by the nitric acid treatment, and this is determined by weighing out a duplicate sample, treating in the same way as above, precipitating the filtrate by molybdate solution, and determining the phosphorus by one of the usual methods.

The filtrate from the insoluble tin oxide and phosphorus pentoxide is just neutralised with ammonia, 5 c.c. of nitric acid added, and the mixture made up to about 150 c.c. with water in a large platinum dish. A piece of platinum foil is suspended in the solution and an electric current passed through, so as to precipitate the lead as dioxide on the dish and the copper as metal on the foil. When all the copper is precipitated—ascertained by testing a drop of the solution with ammonia—the liquid is siphoned off from the precipitates, fresh water being poured in, until the liquid is nearly free from acid. The precipitates are finally well washed with water, dried, and weighed. Iron and zinc are estimated in the liquid by warming and precipitating with excess of ammonia and ammonium sulphide. The precipitate, after washing, is ignited, very cautiously at first, and finally with the blowpipe, and then weighed. The mixed oxides of iron and zinc are then dissolved in hydrochloric acid, and the iron is precipitated with ammonia. The amount of zinc may thus be obtained by difference.

—A. S.

Zinc Sulphide, Precipitation of, in Gravimetric Determination of the Metal. J. Meunier. Comptes Rend. **124**, [21], 1151.

The difficulty experienced in collecting zinc sulphide for gravimetric determination is overcome by the author in the following simple manner:—The zinc solution, preferably slightly warmed, is precipitated with ammonia, just sufficient of the precipitant being cautiously added to redissolve the hydrate at first formed. A slow current of sulphuretted hydrogen is then passed through the solution, until a drop of it, on a white tile, gives a blackish coloration with another drop of a solution of ferrous sulphate. When this occurs the whole of the zinc will have been precipitated, and is in a suitable condition for collection. The passage of the gas is immediately stopped, and the zinc sulphide collected, washed, and dried in the usual way. It will be found that the filtrate will at once run perfectly clear, and that the precipitate may be rapidly washed, especially if warm solutions be employed. The presence of large quantities of other salts does not affect the ease and rapidity with which the process may be conducted.

—J. O. B.

Nitrous Acid, Colorimetric Estimation of Small Quantities of. E. Riegler. Zeits. Anal. Chem. 1897, **36**, 306.

In furtherance of the author's communication on the detection of nitrous acid by means of naphthionic acid (this Journal, 1897, 261), the following colorimetric method is recommended for the estimation of small quantities of nitrous acid:—Dissolve 0·406 grm. of pure silver nitrite in hot distilled water, treat with sodium chloride in slight excess, allow to cool, and dilute to 1 litre. After settling the precipitate, dilute 100 c.c. of the clear solution with water to 1 litre. 100 c.c. of this solution, corresponding to 0·001 grm. of N_2O_3, are transferred to a flask, about 0·05 grm. of crystallised naphthionic acid and 5 or 6 drops of strong hydrochloric acid are added, and, after thoroughly shaking the mixture, 30 drops of ammonia are added. A rose-coloured solution is obtained, the coloration of which serves as an index of the above degree of concentration. For the estimation of nitrous acid in water, 100 c.c. are placed in a flask, about 0·05 grm. of naphthionic acid and 5 drops of strong hydrochloric acid are added, well shaken and treated with 30 drops of strong ammonia. The intensity of colour is then compared with a colorimeter, against that of the standard. In this manner it is possible to determine 0·00001 grm. of N_2O_3 in 100 c.c. of water.—D. B.

ORGANIC CHEMISTRY.—QUALITATIVE.

α-Naphthol from β-Naphthol, A Reaction permitting the easy Distinction of. E. Leger. J. Pharm. Chim. 1897, **5**, 527—529.

A cold saturated aqueous solution of the naphthols made by trituration in a mortar, gives a distinctive coloration with sodium hypobromite. This reagent is obtained by mixing 30 c.c. of caustic soda of 36° B. with 100 c.c. of water and shaking with 5 c.c. of bromine. To 10 c.c. of the naphthol solution add 2 drops of the hypobromite, α-naphthol gives a dirty violet coloration or precipitate, the coloration is even given by a saturated solution diluted with 9 volumes of water. β-naphthol gives a yellow coloration,

becoming greenish and returning to yellow. This reaction is less sensitive, the saturated solution diluted with 2 volumes of water giving a coloration which at once disappears on agitation. The reaction permits of the recognition of 1 part of α-napthol in 100 parts of β-naphthol.—A. C. W.

Stearin in Spermaceti, A Simple Test for Detecting. E. Hirschsohn. Pharm. Centralh. **38**, 283.

One grm. of the spermaceti is dissolved in 10 c.c. of petroleum ether. If the solution be turbid the sample is probably adulterated. It is then shaken with an equal volume of a 1 per mille cupric acetate solution. If only 2 per cent. of stearin be present, the petroleum ether layer acquires a green colour.—J. O. B.

Potato Starch and Potato Flour. Saare. Papier Zeit. 1897, **30**, 1059.

See under XVI., *page* 623.

Active Diastase, Uncertainty of the Guaiacum Reaction for. B. Pawlewski. Ber. 1897, [10], 1313—1314.

According to Lintner, the addition of a solution of active diastase to an alcoholic solution of guaiacum resin containing a few drops of hydrogen peroxide, produces a blue coloration either instantly or within a few minutes, whilst invertin, pepsin, and other similar ferments do not give this action. The reaction has been inserted without comment in several subsequent works.

When the coloration is immediate, it may be taken as proof of the presence of active diatase, if there be no other substance in the solution capable of producing a blue coloration. But when the coloration appears in a few minutes, the evidence is not conclusive that diastase is present, because peptone, gelatin, albumin, &c. produce similar colorations, and guaiacum tincture is even coloured blue by hydrogen peroxide without the presence of other substances; in a weak freshly prepared solution the coloration being immediate, and in a strong dark solution appearing after 3—5 minutes.

The reaction may be used as a test for the presence of N_2O_3 and HNO_2, a deep blue coloration being instantly produced by 0·00005 grm. of sodium or amyl nitrite. Other oxidising agents—ozone, chlorine, bromine, iodine, nitric acid, nitric and chromic acids, permanganate, ferricyanide, &c.—also give blue colorations; thus the reaction loses its value as a test for diastase.—A. C. W.

Alkaloids, Action of Tannin and Gallic Acid on certain. O. de Coninck. Comptes Rend. 1897, **124**, 773—775.

The author has investigated the action of tannin and gallic acid on nicotine and cicutine, under varying conditions. The following reactions are of value:—Two solutions of tannin, A and B, containing respectively 1·434 grms. and 0·0223 grm. of tannin dissolved in 19 c.c. of water, were employed.

0·1 c.c. of *nicotine* dissolved in 150 c.c. of water yields, with 0·1 to 0·5 c.c. of solution B, a yellow coloration. After a time the liquid becomes opalescent and slightly fluorescent. 0·2—0·3 c.c. of nicotine dissolved in water is precipitated on addition of a drop of solution A, the limit of precipitation being reached at a dilution of about 2 litres. A solution of 0·1 c.c. of *cicutine* in water yields a precipitate with one drop of solution A, so long as the dilution does not exceed 175 c.c. The opalescent liquid, which at first exhibits a bluish-white fluorescence, gradually acquires a yellow colour, and develops an intense green fluorescence, persistent for at least 12 hours. (See this Journal, 1897, 470.)—H. T. P.

ORGANIC CHEMISTRY.—QUANTITATIVE.

Nitrogen, The Combustion of. O. Bleier. Ber. 1897, [10], 1269.

The nitrogen is mixed with oxygen in the proper proportion, brought into a suitable vessel (an enamelled autoclave or a very strong-walled explosion pipette) containing dilute alkali, and a large quantity of electrolytic gas is pumped in. The mixture is exploded and shaken up, to promote the absorption of oxides of nitrogen. The operation must be repeated several times. (Compare this Journal, 1897, 466—467.)—A. C. W.

Xylidines, Commercial; Estimation of. W. Vaubel. Zeits. Anal. Chem. 1897, **36**, 285—289.

The following bases are present in commercial xylidines:—

I.—$CH_3.CH_3.NH_2$ 1.2.4 = α-amido-orthoxylene.
II.—$CH_3.CH_3.NH_2$ 1.2.3 = β-amido-orthoxylene.
III.—$CH_3.CH_3.NH_2$ 1.3.4 = α-amido-metaxylene.
IV.—$CH_3.CH_3.NH_2$ 1.3.2 = β-amido-metaxylene.
V.—$CH_3.CH_3.NH_2$ 1.4.2 = amido-paraxylene.

The chief constituents are α-amido-metaxylene or asymmetric metaxylidine and amido-paraxylene or paraxylidine. The percentage of the former is usually estimated by converting the base into the corresponding hydrochloride or acetate, both compounds being insoluble in water. When glacial acetic acid is used as precipitant, equal quantities of acid and base give the best results. More accurate results are, however, obtained by converting the acetate into the bromide. Asymmetric metaxylidine takes up one atom of bromine; and although β-amido-metaxylene behaves in a like manner, its salts, especially the hydrochloride, are readily soluble. The other bases form dibromides with bromine.—D. B.

Sumach, Adulterations of, and Methods for their Detection. M. Spica. Chem. Central-Bl. 1897, 1101—1102.

Sumach is adulterated with the leaves of *Tamarix Africana* (Bruca) and *Pistacia lentiscus* (Stinco). These adulterations may be detected in several ways. By a nitrogen determination, ash determination, or analysis of ash, as the following figures show:—

——	Sumach.	Bruca.	Stinco.
Nitrogen per cent. ...	0·87—0·98	1·48—1·99	1·47—2·01
Ash „ ...	6·6	12·4	5·4
	Per Cent. of Ash.		
SiO_2 and insoluble	24·050	37·100	6·200
CaO	29·950	8·530	25·300
SO_3	4·670	20·139	5·218
MgO..................	6·250	9·368	5·760
$Al_2O_3 + Fe_2O_3$	7·150	7·400	7·410
CO_2	12·600	1·130	13·750
P_2O_5	3·344	1·113	4·021
Cl	3·101	4·405	5·321
K_2O	6·305	7·950	14·604
Na_2O	2·004	2·630	12·176
Loss	0·576	0·235	0·240
	100·000	100·000	100·000

Adulterations may also be determined by a colorimetric method, since sumach gives a colour reaction not given by the adulterants. A solution of 0·15 grm. of safranine in 1 litre of water serves as the standard of comparison. 5 grms. of material are boiled for ½ hour with ½ litre of water, the liquid cooled, made up to ½ litre, and filtered. To 25 c.c. of the filtrate in a beaker 5 grms. of a solution of basic lead acetate (specific gravity at 15° = 1·184) and 15 c.c. of caustic potash (specific gravity at 15° = 1·155) are added, and the whole concentrated to 15 c.c. The solution should now be reddish-brown and almost clear. If a noticeable precipitate has formed, or if the liquid is only yellow, the sumach is adulterated. The 15 c.c. are diluted to 250 c.c., and, if necessary, filtered; a solution prepared from pure sumach will now show the tint of the safranine solution. If a filtered sumach extract be warmed with excess of potash and a few drops of ammonium molybdate be added, a chocolate-brown precipitate is obtained; in the presence of Bruca this has a greenish, in presence of Stinco a yellowish-brown tinge.—A. C. W.

Fats, Differentiation by the Volume of the contained Fatty Acids. R. Zaloziecki. Chem. Rev. Fett- u. Harz-Ind. **4**, [9], 119—121.

The differences in volume of the fatty acids in fats being, by reason of their different specific gravities, greater than

is expressed by their weight, the author has sought to apply this as a means of differentiation, particularly in the case of butter and its adjuncts.

The apparatus employed is a 200 c.c. flask with a long neck, graduated to 15 c.c. in divisions of 0·1 c.c., resembling a specific gravity instrument. 10 grms. of fat, filtered, and dried at 100° C., are carefully weighed and saponified on the water-bath, with 4 grms. of purified KHO and 50 c.c. of alcohol of 70°. When the alcohol is all evaporated, the soap is dissolved in hot water and transferred into the measuring vessel, rinsed down with hot water, and treated with 25 c.c. of sulphuric acid to separate the fatty acids, sufficient hot water being added to cause the fatty acid layer to rise into the graduated part of the neck. To ensure rapid clarification of the fatty acid, the water should be hot, and the bottle immersed in a water-bath to maintain the contents at the reading temperature, 80° C. Another plan is to proceed as before with half the quantity of fat, dissolving the fatty acid layer with a known volume of benzene, and reading off the volume of the resulting solution, at room temperature; the first method is, however, considered preferable.

The following results have been obtained :—

	Vol. of Fatty Acids at 80° C.
	C.c.
Table butter, A	10·75 / 10·70
„ B	10·80 / 10·80
Market butter A	10·85
„ B	10·80
Old rancid butter	10·95
Old beef tallow	11·70
Fresh „	11·50
Mutton tallow	11·60
Fresh hog's lard	11·50
Margarin	11·60
„ lard	11·40
Oleomargarin	11·35
Cocoanut oil	10·95 / 10·90
Olive oil	11·25
Rape oil	11·55
Linseed oil	11·50
Castor oil	11·30

	Vol. of Fatty Acid in Benzene Solution at 20° C.	
	C.c.	
Table butter A	5·10	(from 5 grms. substance)
„ B	5·20	„ „
Beef tallow	5·45	„ „
Margarin	5·40	„ „
Cocoanut oil	5·25	„ „

The average figure for butter, 10·8 c.c. per 10 grms., is therefore appreciably lower than the figures for the fats used as adjuncts thereto; even if a margin of 0·2 c.c. be allowed on both sides, the difference between the maximum for butter (11·0) and the minimum for butter substitutes of animal origin, amounts to 0·3 c.c. per 10 grms.—sufficient to differentiate between them.—C. S.

Butter and Margarine, Discrimination between. A. v. Asbóth. Chem. Zeit. 1897, **21**, [32], 312—314.

The author asserts that the lead salts of the soluble fatty acids in butter are as soluble in water as are the acids themselves, and on this he bases the following process for arriving more rapidly at the Hehner and Reichert values :— The saponification value is determined on 2·5 grms. of the fat. The resulting soap is decomposed with a solution of lead acetate, and the lead soap filtered and washed in a linen cloth, which is squeezed in the hands to remove water. The insoluble lead salts are decomposed by being shaken in a Muter tube with ether and hydrochloric acid, the ethereal solution washed with water, its volume read off, an aliquot portion run into a weighed flask, the ether evaporated, and the fatty acids dried and weighed. From the result the total weight of insoluble fatty acids can be calculated and the Hehner number obtained. The weighed fatty acids are then titrated with standard alkali and the result calculated on 2·5 grms. The difference between the number of c.c. of decinormal alkali required by the total fatty acids and those used by the insoluble fatty acids gives the Reichert value.
—C. A. M.

Hübl's Iodine Solution, The Behaviour of Tannic Acids and Tannin Extracts towards. C. Boettinger. Chem. Zeit. **21**, [46], 460.

The author refers to his previous communications on the behaviour of gallic acid, tannin, and the acetyl derivatives of these bodies towards solutions of iodine and mercuric chloride (Chem. Zeit. 1896, **20**, 984; 1897, **21**, 57). He has now examined the acetyl compounds of the tannins of oak bark, oak wood, and pine bark; these compounds give practically no iodine number. The pure tannins were then prepared from the acetyl compounds by the author's method (this Journal, 1884, 526; and 1887, 550. Also Ann. 1887, **238**, 370). They gave iodine numbers varying according to the quantity of iodine and the concentration.

The tannic acid of oak bark gave iodine numbers from	120·4—134·5
The tannic acid of oak wood gave iodine numbers from	39·2— 51·4
The tannic acid of pine bark gave iodine numbers from	46·4— 55·9

The use of chloroform as a solvent of the tannic acids is quite unnecessary.

The same tests were applied to the extracts of different tanning materials, with the following results :—

	Mean Iodine Number.
1. Oak-wood extract	29
2. Pine bark	99
3. Oak bark	106
4. Algarobilla	114
5. Canaigre	119
6. Valonia	153
7. Pegu catechu	164
8. Sumach	165
9. Quebracho wood	188
10. Terra japonica	202

These figures are the mean of 6—8 experiments, the mean numbers for the pure tannins being: oak bark, 130·1; oak wood, 45·8; pine bark, 50·3.—J. T. W.

Starch, Control of the Manufacture of. Hébert. Bull. Assoc. Chim. 1897, **14**, 1003.

See under XVI., *page* 623.

Meat Extracts, Analysis and Composition of. J. Bruylants. J. Pharm. Chim. 1897, **5**, 515—521.

Compare this Journal, 1895, 897; 1896, 130, 149, 897. The quantitative method of analysis was as follows:— Water, ash, sodium chloride, total nitrogen, nitrogen as ammonium salts, matters insoluble in water, and nitrogen in this insoluble part were determined according to ordinary methods. Then gelatin, albumoses, peptone, non-albuminoid nitrogenous substances soluble and insoluble in alcohol, were separated by successive treatment with alcohol of 40, 80, and 93—94 per cent. Gelatin is thrown down by 40 per cent., albumoses by 80 per cent., and peptones by 93—94 per cent. alcohol. These precipitates are far from pure. After purification, the nitrogen in each was determined; this, multiplied by 6·25, gives the amount of gelatin, albumose, or peptone.

10 grms. of dry extract or 20 grms. of liquid extract were dissolved in water, the insoluble part filtered off and washed. After drying, nitrogen was determined in this residue by Kjeldahl's method. The solution was heated to boiling and filtered hot; the precipitate gives the coagulable albumin. The filtrate was evaporated to the consistence of a solid extract, and several times extracted by 80 per cent. alcohol. The solution, A, contains peptones, non-albuminoid nitrogenous substances, mineral salts, glucose, sarcolactic acid, &c. The residue, A, contains gelatin, albumoses, some non-

albuminoid nitrogenous matters, mineral salts, &c. This residue, washed with 80 per cent. alcohol and freed from alcohol on the water-bath, was dissolved in 60 c.c. of distilled water, 40 c.c. of absolute alcohol were added, and the mixture left for 24 hours. The solution, B, contains albumoses, non-albuminoid nitrogenous matters, and mineral salts; the residue B, mineral salts and gelatin. This residue, after washing with 40 per cent. alcohol, was used for the gelatin determination.

The solution B, after addition of the wash liquors of residue B, was evaporated to 10 c.c., poured into 30 c.c. of saturated ammonium sulphate solution, a slight excess of the same salt added, the mixture heated to boiling, and filtered. The precipitate of albumoses thus obtained was washed with ammonium sulphate solution, dissolved in water, and ammonia driven off by barium carbonate; the nitrogen determination now gave the amount of albumoses. The ammonium sulphate solution, freed from ammonia, was used for a nitrogen determination, half of which belongs to non-albuminous matters and half to peptones. The solution A was evaporated to the consistence of extract, dissolved in the least possible quantity of 80 per cent. alcohol, and absolute alcohol added to bring up the percentage to 93—94. After 24 hours the solution was decanted and the residue again treated in the same way. The precipitate was dissolved in 100 c.c. of water, a slight excess of basic acetate of lead added; after 24 hours the mixture was filtered and the precipitate taken up in 100 c.c. of water. The nitrogen found in the precipitate belongs to the non-albuminoids. The lead acetate liquors, freed from lead by sulphuretted hydrogen and evaporated, give the peptones.

The solution in 93—94 per cent. alcohol, after evaporation, also gives non-albuminoids. The nitrogen in this residue is given in the table as "nitrogen soluble in strong alcohol."

——	Liebig's Extract.	Solid Bovril.	Bovril "for Invalids."	Liquid Bovril.
Water	16·75	19·20	22·35	43·25
Sodium chloride	2·95	4·50	4·00	9·75
Other mineral salts	18·24	16·20	17·05	6·25
Insoluble in water	..	..	7·10	8·19
Organic matters	62·06	60·10	54·50	32·06
Total nitrogen	9·30	8·85	9·12	4·85
Nitrogen in part insoluble in water	..	..	1·09	1·19
Nitrogen, ammoniacal, uric acid, &c.	0·60 }	0·50 }	0·15 }	0·30 }
" from lead precipitate	0·65 } 5·09	0·57 } 4·56	0·45 } 4·48	0·27 } 2·67
" non-albuminoid, from 80 per cent. alcohol	0·15 }	0·20 }	0·18 }	0·05 }
" soluble in strong alcohol	3·69 }	3·29 }	3·40 }	1·05 }
Nitrogen, from gelatin	0·19 }	0·25 }	0·12 }	0·05 }
" from albumoses	0·80 } 3·93	0·95 } 3·78	0·75 } 3·57	0·45 } 1·83
" from peptones	2·94 }	2·58 }	2·70 }	1·33 }
Total soluble proteids	24·56	23·62	22·40	11·43
Insoluble albumin	..	..	6·81	7·43

The substance insoluble in water is fine meat powder.

—A. C. W.

Horseflesh, Detection and Estimation of, in Sausages and Meat Preparations. H. Bremer. Forschungs-Ber. 1897, **4**, 1—8.

The various processes proposed for the detection of horseflesh are based either on the glycogen reaction or on an examination of the fat. To the latter class belongs the author's method, which consists in extracting and examining the intra-muscular fat. The finely minced sample, from which all visible fat has been removed, is heated with water on the water-bath for an hour. The fat rising to the surface is poured away, and the flesh washed several times with hot water, well dried at 100°, and extracted for several hours with a low-boiling petroleum spirit. Part of the intra-muscular fat thus obtained is used for the determination of the refractive index, iodine number, and Reichert-Meissl number. The remainder is saponified and converted into zinc salts, which are extracted with ether. The fatty acids are liberated from the soluble portion, and their iodine number determined.

In this way the author obtained the subjoined results:—

——	Iodine No. of Intra-Muscular Fat.	Iodine No. of Liquid Fatty Acids.
1. Horseflesh sausage, without pork	75·8	108·1
2. Horseflesh sausage, with about 6 per cent. of pork.	74·0	104·1
3. Horseflesh cervelat, with about 22 per cent. of pork (well smoked).	53·7	92·4
4. Horseflesh cervelat, with about 25 per cent. of pork.	71·1	102·1
5. Ordinary sausage	57·6	94·2
6. Thuringian cervelat, with about 65 per cent. of lard.	64·3	95·8
7. Mixture of 1 and 5 in equal parts	66·4	103·1
8. " 4 and 6 " "	65·2	99·5

It is stated that when horseflesh is present the petroleum spirit extract and the liquid fatty acids have a more or less reddish colour. On the other hand, the flesh of bulls gives a similar coloration; so that this must only be regarded as confirmatory evidence. When, however, the red colour is present, and when at the same time glycogen is detected, while the iodine number of the intra-muscular fat exceeds 65, and that of the liquid fatty acids is considerably over 95, there can, in the author's opinion, be but little doubt as to the presence of horseflesh.—C. A. M.

Theobromine, Determination of, in Cocoa and Chocolate. L. Maupy. J. Pharm. Chim. 1897, [6], **5**, 329.

The quantity of the sample taken for the following process is generally 5 grms. for cocoa of good quality; if it is desired to increase the amount of alkaloid available for final weighing, more of the sample may be used.

The finely crushed cocoa is placed in a flask with 60 grms. of petroleum ether or ligroin; the flask is well corked and the mixture shaken occasionally for a day. The powder and liquid are then thrown upon a plain filter, drained, and, after washing with a little more of the solvent, the cocoa is dried. The dry cocoa free from fat, thus obtained is now triturated with 2 grms. of distilled water, the moist mass introduced into a small flask with 20 grms. of a mixture of pure crystalline phenol, 15 parts; chloroform, 85 parts, and boiled under a reflux condenser for one hour; the mixture is then filtered, preferably by the aid of a filter pump, and the residue again extracted twice with further portions of the phenolchloroform solvent, boiling for 30 minutes on each occasion. The mixed chloroformic extracts are distilled, the last traces of chloroform being carefully driven off; to the cold residue 40 grms. of anhydrous ether are added, and, after well shaking, the mixture is left for six hours. Theobromine is precipitated, while caffeine, colouring matter, and traces of fat remain in solution. The precipitated alkaloid is thrown on a counterpoised filter, washed with a few c.c. of ether to remove

the last traces of phenol, dried, and weighed. The alkaloid should be white or scarcely tinted, even in the case of roasted cocoas or chocolate.

With chocolate 10 grms. are taken, the fat is removed with petroleum ether, and the powder then mixed with 4 grms. of 70 per cent. alcohol, and the assay continued as described above for 5 grms. of cocoa. Should the amount of sugar present exceed 50 per cent., the amount of phenol-chloroform solvent is reduced so as to employ about 60 centigrms. of phenol for every grm. of cocoa considered present as chocolate, the volume of the liquid being made up by more chloroform. The following percentages of theobromine were obtained by the author with this method:—

	Per Cent.
Roasted Trinidad	1·44
„ Caracas	1·38
„ Grenada	1·60
„ Para	1·28
„ Martinique	1·52
Chocolate (60 per cent. of sugar)	0·54

—J. O. B.

Alcohol, The Estimation of, by the Vaporimeter. F. Rothenbach. Wochenschr. f. Brau. 1897, **14**, 189.

The author considers the vaporimeter to be an utterly untrustworthy instrument, especially when employed for the estimation of alcohol in impure liquids, such as vinegar mashes, beer, &c., which contain substances (including dissolved CO_2 and air) able to influence the alcoholic vapour tension. He recommends for the examination of the materials referred to, the well-known distillation process.

—H. T. P.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Platinum Chloride, Recovery of Waste. H. W. Wiley. J. Amer. Chem. Soc. 1897, **19**, 258—261.

The author mentions that the following method has been worked out by K. P. McElroy. The waste platinum from potash determinations is collected, dissolved in water, and aluminium in the form of clippings or turnings added to the hot solution. In a few minutes a platinum-aluminium couple is formed, and reduction proceeds vigorously. The addition of hydrochloric acid is advisable to facilitate the deposition of the platinum, and when the reduction is complete, more hydrochloric acid is added to dissolve the excess of aluminium. The spongy platinum is well washed by decantation, and treated with nitric acid to remove any traces of copper; it is again washed, and dissolved in aqua regia (5 parts of hydrochloric to 1 part of nitric acid). The solution thus obtained is evaporated, dilute hydrochloric acid added, and re-evaporated. Should nitrous fumes be evolved on the addition of water, the solution is evaporated, and this treatment continued until the nitric acid is removed. The resulting solution of platinum chloride is then diluted and filtered.—J. L. B.

Phosphorus, A New Oxide of. A. Besson. Comptes Rend. 1897, **124**, 763—765.

The new oxide, to which the author assigns the formula P_2O, is obtained by the action, at 50° C., of hydrogen phosphide on a mixture of phosphorus oxychloride with hydrobromic acid, or a bromo-oxy compound of phosphorus; or, preferably, by heating to 50° C. a mixture of phosphonium bromide and phosphorus oxychloride. The oxide separates in the solid state, and, when pure, forms a reddish-yellow voluminous powder, which is stable at ordinary temperatures and at 100° C. When heated to 135° C. *in vacuo*, it loses oxygen until the percentage of phosphorus in the residue attains 90—95. The oxide is combustible, and reacts in the same way as phosphorus with nitric acid. It burns vividly when projected into chlorine. When the reaction is moderated by operating with a carbon tetrachloride solution of the gas, the product formed is either PCl_5 or PCl_3, according as the chlorine or oxide is present in excess. Under the same conditions, bromine forms $POBr_3$, and either PBr_5 or PBr_3; whilst with iodine the sole product is PI_3.

P_2O appears to be a neutral body. It is insoluble in water and dilute alkaline solutions, and even when heated to 150° C. with water, is only slightly attacked, yielding traces of phosphorous acid and hydrogen phosphide.

Phosphonium iodide acts quite differently on phosphorus oxychloride, the products being P_2I_4, an intimate mixture of red phosphorus and iodine, and metaphosphoryl chloride (PO_2Cl).—H. T. P.

A Vegetable Lipase, extracted from Penicillium Glaucum. E. Gérard. J. Pharm. Chim. 1897, **5**, 529—530.

A solution of 2 grms. of monobutyrin in 100 c.c. of water to which 0·3 grm. of the soluble ferments of *Penicillium glaucum* was added, showed a continual increase in acidity, due to saponification of the ester. Sigmund (Monatsh. für Chem. **13**, 567) states that ferments which decompose glucosides also saponify fats. The ferments of *Penicillium* contain emulsin, but the author did not observe any appreciable saponification of monobutyrin by that ferment.

—A. C. W.

Strontium Sulphide, A Method for preparing highly Phosphorescent. J. R. Mourelo. Comptes Rend. 1897, **124**, 1024—1026.

The author has experimented with several methods—described in detail—for the preparation of strontium sulphide, and finds that by far the best results, so far as phosphorescent power of the product is concerned, are obtained as follows:—

An intimate mixture of 285 grms. of commercial strontium carbonate, 62 grms. of flowers of sulphur, 4 grms. of crystallised sodium carbonate, 2·5 grms. of salt, and 0·4 grm. of bismuth subnitrate, is placed in a crucible, covered with a layer of coarsely powdered starch, and submitted for five hours to a bright red heat, and subsequently allowed to cool very slowly (10—12 hours). A white, friable mass is thus obtained, which develops a fine greenish-blue fluorescence when exposed for about one second to daylight, and so intense as to be plainly visible in a shaded situation. The author remarks, however, in conclusion, that the greater number of his preparations became perfectly inert when powdered, although in some instances the phosphorescent power of the sulphide was restored by re-ignition in contact with starch.—H. T. P.

Phosphoresence of Strontium Sulphide. J. R. Mourelo. Comptes Rend. 1897, **124**, [25], 1521—1523.

While the phosphorescence of calcium sulphide varies considerably in colour, with even slight variations in the conditions of its preparation, that of strontium sulphide is remarkably constant, being a more or less greenish-blue. The tint varies slightly, and the intensity to a greater degree, with the source of the sulphide: the most intense, and the greenest, being that of the sulphide prepared from a mixture of strontium and sodium carbonates, sodium chloride, and bismuth subnitrate, while the least intense is the bluer phosphorescence of the sulphide reduced from the sulphate by lamp-black.

The author has made experiments on the sulphides prepared as above, and also by the action of hydrogen sulphide and of sulphur on strontia, and by Verneuil's method, in which the samples, prepared in the first instance at different temperatures and after different periods of heating, were afterwards exposed for varying periods to further heating, sometimes at temperatures above those of preparation. In no case (provided care had been taken to avoid any oxidation) was there any change produced in the colour or intensity of the phosphorescence of the sample: so that temperature seems to have but little influence on the phosphorescence.—J. T. D.

Chromium, The Atomic Weight of. Eng. and Mining J., June 26, 1897, 655.

Investigations in pure science are often unintelligible to the practical man, and generally appear useless; yet sometimes their bearing upon technology and industry is

exceedingly direct. An instance of this kind is pointed out by Mr. F. W. Clarke in a recent number of the *Journal* of the American Chemical Society. The question is often asked, Why are new determinations important? Are not those now accepted good enough for practical purposes? To this question Mr. Clarke makes the following answer:—

There are two rival values for the atomic weight of chromium. The one, 52·5, based on the old work of Berlin, is used by European analysts; the other, 52·1, based on later researches, is used in the United States. The Baltimore Chrome Ore Works imparts chromic iron in cargoes of about 2,500 tons, which are assayed in Glasgow and Baltimore. The chemists of the former city based their percentage calculation on the atomic weight of chromium, 52·5, and those of the latter place on 52·1. The variation in percentage resulting from this amounts to a difference of 367·50 dols. in the value of a cargo.

Obituary.

PAUL SCHÜTZENBERGER.

Schützenberger was born Dec. 23rd, 1827, at Strasburg. He first gave himself to the study of medicine, but later on he became devoted to chemical science, which then and throughout the remainder of his life, absorbed his whole attention. From 1851 to 1861 he was a lecturer in the Realschule, and still later he became lecturer in the High School at Mulhouse. In 1865 he was assistant in the Collège de France, Paris, and prepared the experiments, &c. for Ballard's lectures; and in 1868 he was advanced to the position of second director of the chemical laboratory at the Sorbonne, in 1876 becoming Professor in the Collège de France. On the foundation of the School of Chemical Industry and Physics, in the year 1882, Schützenberger was chosen as director of that institute. As the successor of Dumas, he was elected a member of the Academy of Medicine, and, as the successor of Debray, as a member of the Academy of Sciences; and finally, he obtained the rank of an Officer of the Legion of Honour for distinguished services to his country.

Schützenberger was the first to point out the law of the substitution of the metals in salts by electro-negative elements. The highest interest also attaches to the chloracetate $C_2H_3O_2Cl$, first prepared by him, in which chlorine plays the part of a metal. In Schützenberger's researches he displayed marvellous versatility, attacking with uniform success problems in the fields of physiological, organic, and inorganic chemistry. His researches on the natural dyestuffs must also be recorded: on madder, cochineal, woad, and indigo. Likewise, his very important investigations on the respiration and changes of beer yeast; also on the constitution of the albuminoids, with the aid of baryta water under pressure, according to which these albuminoids must be regarded as crystalline substances. In Germany he probably won most renown by his work, published in 1866, on Dyestuffs and Colouring Matters, as well as by the discovery, made conjointly with Lalande, of the "Hydrosulphite Vat."

But he also published a remarkable work on Physiological Chemistry, a monograph on The Phenomena of Fermentation, and his great text-book on General Chemistry.

In the latter years of his life he entirely devoted himself, with several of his students, to the study of the rare earths. He was the originator of the communications on "Lucium"—a hypothetical element supposed to belong to the Yttrium group of metals. In Paul Schützenberger, France has lost one of the most gifted and original of her chemists. He died in Paris, on the 26th of June 1897.—W. S.

Trade Report.

TARIFF CHANGES AND CUSTOMS REGULATIONS.

(*From the Board of Trade Journal.*)

France.

Customs Decisions.

The following decisions as to the classification of articles in the French Customs tariff have been communicated by the Director of French Customs:—

Mastic for cycles, composed of caoutchouc or gutta-percha and vegetable rosin, with or without the addition of mineral matter, is to be classed as vegetable rosin (No. 115).

Or brillant or *glanz gold* is classified as a "chemical product, not denominated, of a base other than alcohol" (No. 282). *Platine brillant*" and "*Argent brillant*" will be classed under the same category.

Netherlands.

Removal of Duty on Aniline Oil.

U.S. Cons. Reps., July 1897, 464.

The United States Minister at The Hague reports, on May 24, 1897:—

By royal order of May 8, 1897, appearing in the *Netherlands Gazette* of to-day, aniline oil is declared to be exempt from import duty.

Portugal—East Africa.

Classification of Sulphuric Acid.

A despatch, dated 16th June last, has been received at the Foreign Office from Sir H. G. Macdonell, Her Majesty's Minister at Lisbon, stating that, by a ministerial decree of June 15 last, it is provided that, as in the Imports Customs tariff for the province of Mozambique no mention is made of chemical products, sulphuric acid must be classified, for the payment of the duty leviable thereon, under Article 42 of the Mozambique Customs Tariff of December 29, 1892.

United States.

Customs Decisions.

The following decisions relating to the classification of articles in the Customs tariff, and the application of the Customs laws of the United States, have recently been given by the authorities in that country:—

Alizarin black, a coal-tar colour or dye, is liable to duty at 35 per cent. *ad valorem* under paragraph 18 of the Act of 1890.

Nucoa-nut oil, or "nucoa butter," or "solidified cocoa-nut oil" is classified as "cocoa butterine," at the rate of $3\frac{1}{2}$ cents per lb. under paragraph 230 of the Act of 1894.

"Fibrite," artificial sulphate of lime, is classified for duty at 25 per cent. *ad valorem* under paragraph 39 of Act of August 28, 1894.

Potato starch or potato flour is dutiable at $1\frac{1}{2}$ cents per lb., as provided in paragraph 232 of the Act of 1894, for starch.

Carbolineum in Nicaragua.

U.S. Cons. Reps., June 1897, 269.

On the 9th of March 1897, the Government of Nicaragua issued a decree by which the duties on the importation of carbolineum are abolished.

Carbolineum, or crude carbolic acid, is used in this country to a great extent for the killing of insects, and will find a ready market here at this time of the year, when the dry season is giving place to the rainy one, and obnoxious insects are abundant.

Spirit Drawback in Canada.

Chem. and Druggist, July 10, 1897, 41.

The Canadian Government have, in response to an agitation in the drug trade there, permitted druggists to obtain a drawback of 75 c. per proof gallon on spirit used for the production of preparations in the manufacture of which alcohol is destroyed for potable purposes. The same rate has hitherto been allowed to perfumers only.

OFFICIAL NOTICES.

Board of Trade Committee on Consular Reports.

Standard, July 21, 1897.

The Board of Trade have appointed a Committee consisting of the following gentlemen, *viz.*, Sir Courtenay Boyle (Chairman), Sir H. Stafford Northcote, M.P., Sir Henry Bergne, Mr. S. E. Spring Rice, and Mr. A. E. Bateman, to consider and advise as to the best means of bringing to the knowledge of the home trades the information furnished by Consuls and Commercial *Attachés*, and by Agents General and other representatives of Colonial Governments and of India, as to the supply and demand and other conditions of the markets in their respective districts; and as to the opportunities which they afford for the introduction and development of British trade; also to report whether it is, in their opinion, desirable that Consuls and Agents should be instructed to remit home from time to time samples of goods most in demand in such markets; and, if they shall be of that opinion, to advise what (if any) arrangements should be made for the exhibition of such samples in London, or for their circulation, with the aid of Chambers of Commerce, to the provincial trade centres, with information as to price, and other conditions under which they are supplied. Mr. G. J. Stanley, of the Board of Trade, will act as secretary to the Committee.

Carbide of Calcium.

Since the Order in Council of the 26th February 1897, in virtue of which certain parts of the Petroleum Acts, 1871 to 1881, were applied to carbide of calcium, the question of the expediency of exempting small quantities of this substance from the operation of the Order has occupied the attention of the Home Office, and the Secretary of State having been advised that such exemption might be safely extended to quantities of carbide of calcium not exceeding 5 lb., when kept in separate substantial hermetically-closed metal vessels containing not more than 1 lb. each, an Order in Council was made on the 7th of July, authorising the keeping of not more than 5 lb. of carbide of calcinm in vessels as above described without a licence, and the original Order of the 26th Febrnary has been amended accordingly.

The amending Order appeared in the *London Gazette* of the 9th July. It is to be observed that where the carbide of calcium is not kept in vessels as above described, no quantity may be kept without a licence.

Proposed National Physical Laboratory.

Standard, Aug. 4, 1897.

The Treasury have appointed a committee to consider and report upon the desirability of establishing a National Physical Laboratory for the testing and verification of instruments for physical investigation, for the construction and preservation of standards of measurement, and for the systematic determination of physical constants and numerical data useful for scientific and industrial purposes, and to report whether the work of such an institution, if established, could be associated with any testing or standardising work already performed, wholly or partly, at the public cost. The following will be the members of the committee:—Lord Rayleigh (Chairman), Sir Courtenay Boyle, Sir Andrew Noble, Sir John Wolfe Barry, Mr. W. C. Roberts-Austen, Mr. Robert Chalmers (of the Treasury), Mr. A. W. Rücker, Mr. Alexander Siemens, and Dr. T. E. Thorpe.

EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

American Drug Preparations in France.

U.S. Cons. Reps., June 1897, 215.

Inquiries having been made at the U.S. Consulate at Limoges by certain leading druggists of Central France concerning certain American preparations of drugs, a few samples were shown to them, with which they expressed themselves very pleased. It seemed to these gentlemen that a good opportunity for introducing these goods presents itself.

The following list of drugs are those most usually prescribed in this country: Sulphate of quinine, opium, nux vomica, Dover's powders, lithia, chlorate of potash, bromide of potassium, bromide of sodium, salicylate of soda, iodide of potassium, and others that enter into the daily use of a physician having a general practice. A certain quantity of proprietary medicines might find a ready sale if put upon the market in a wise manner, but it will be absolutely necessary to have the formulas printed on every bottle. Such common preparations as seidlitz powders and citrate of magnesia, and heavy drugs like glycerin, borax, potash, cod-liver oil, borate of soda, caustic soda, and alkalis are used in large quantities in this country. The French druggists have been deterred from using American drug preparations because the weights and measures employed by the exporters from the United States are not the same as those in use in France; the decimal system is the only one known here—grains, drachms, and ounces are unknown quantities to the laity, and the profession will not take the trouble to translate the weights into grams and milligrams so as to find out what dose to give. The French admit that the manner of putting up drugs in the United States is greatly superior to that of other countries, but the system of foreign weights, measures, and currency forms a barrier to their introduction into this Republic. All drugs should be put in neat, but not too large, packages; the matter of colour and taste are important items to take into consideration, because the masses are not accustomed to taking medicine without a doctor's prescription, as are the people of the United States.

The World's Aluminium Output.

U.S. Cons. Reps., June 1897, 322.

The United States Consul at Zürich reports as follows:—

The aluminium production in the different countries, together with the horse-power employed and the daily capacity of each plant, is given in the following table:—

Works.	Horse-Power.	Average Daily Production.	
United States.			
Pittsburg Reduction Company's Works:—		Kilos.	Lb.
At Kensington, Pa.	1,600	906	1,993
At Niagara Falls, N.Y.	1,600	1,000	2,200
Switzerland.			
Aluminium Ind. Gesellschaft, Neuhausen.	4,000	2,270	4,994
France.			
Société Electr. Mét. Franc., at Lapraz.	2,500	1,360	2,992
Société Ind. de l'Aluminium, at St. Michel.	2,000	1,134	2,495
Total	11,700	6,670	14,674

Or, in round numbers, 6,700 kilos. (14,740 lb.), giving at present an annual production of about 2,000 metric tons. The output during 1895 was about 1,200 metric tons. A considerable reduction in the price of aluminium, on account of the additions to works now in operation and their increase in horse-power capacity, as well as the contemplated erection of new plants, is imminent. In Great Britain, the British Aluminium Company has purchased the Irish bauxite pits, for the purpose of manufacturing aluminium at Larne Harbour, near Belfast. The same company has bought water rights at Foyers Falls, in Scotland, where 4,000 horse-power is at its disposition.

The new addition to the Neuhausen works, which is to be operated by the Héroult system, will, when in operation, add 2,700 kilos. (5,940 lb.) to its daily production. A German-American syndicate has planned the erection of works for the manufacture of aluminium at the waterfalls of Sarpfoss, in Norway. The water-power there, it is estimated, will be produce 10,000 horse-power, and the plant is to be ready for operation some time in 1898. If they utilise 5,000 horse-power, a daily production of 3,000

kilos. will result. Further, the Neuhausen company is building new works, with water-power to produce 10,000 horse-power, at Rheinfelden, Switzerland, and has also acquired water rights for similar purposes at Lend, near Gastein, in Austria.

These new additions (Rheinfelden, 6,000 horse-power; Foyers Falls, 3,000 horse-power; Sarpfoss Falls, 5,000 horse-power), as well as the increased horse-power capacities projected for the plants now in existence (the increase of 7,100 horse-power at Niagara Falls and 4,000 horse-power at St. Michel), will, in the year 1898, with an aggregate capacity of 33,000 horse-power, turn out an average of 19,300 kilos. (42,460 lb.) daily, or, annually, 5,790 metric tons. It becomes therefore evident that the price of aluminium will fall to about 3 frs. (60 cents) per kilo., or about 27 cents per lb.; so that soon but three commercial metals will be cheaper than aluminium, namely, iron, lead, and zinc.

Brimstone in Sicily.

U.S. Cons. Reps., June 1897, 203.

The U.S. Consul at Messina reports as follows:—

(1.) The exports of sulphur from Sicily in 1896 were 44,000 tons in excess of 1895, of which 31,000 tons were exported from January to July. This increase is to be attributed to the exceedingly low prices which prevailed towards the close of 1895 and beginning of 1896, at which figures the working of a majority of the mines was unprofitable. The increase of 13,000 tons in the last six months of 1896 is undoubtedly due to the sudden advance in prices, which induced consumers to lay in heavy stocks in anticipation of a further rise. These stocks, however, cost them relatively low prices.

(2.) Although prices steadily declined during those years, the production, as estimated by the mining bureau, frequently increased and never decreased. Stocks show a constant increase. These facts unquestionably prove that the cost of production has diminished, cheaper transportation has been gained by the construction of roads and railways, and improvements have been made in mining and smelting. It follows, therefore, that production at the lower prices has continued steadily because it was found profitable.

(3.) The increase of exports during these years is natural, because the large production had to be sold, and this was made possible by the fall in prices.

(4.) The Italian Parliament abolished the export duty of 8*s*. 6*d*. (2·66 dols.) per ton with the object of favouring exports, since this duty increased the cost to the foreign consumer. The artificial advance which the Anglo-Sicilian sulphur trust wishes to establish is therefore in opposition to all the above facts, although the latter are responsible both for the decline in price for several years and the abolition of the export duty.

(5.) It is stated that the production in 1896 shows an increase, and although the statistics are not yet published, there is the best authority for estimating it at about 385,000 tons. That there really was an increase is confirmed by the following facts:—There were shipped in 1896, 44,000 tons more than in 1895, but nevertheless the visible stock in the ports of Sicily on the 31st of December 1896 was only 18,000 tons less than on the same date in 1895. The difference of 26,000 tons must therefore come from an increase of production, which, in part at least, belongs to 1896, as some of this quantity may be sulphur stored in the mines in 1895 and not shipped promptly. Further, the quantity carried by rail from the stations in the interior to the ports was larger in the fiscal year 1895–96 than in 1894–95.

(6.) Everything points to an increased production for 1897, and if the present prices check the consumption, stocks must necessarily increase during the year.

(7.) Should Sicilian brimstone be partly replaced in the United States by pyrites for the manufacture of sulphuric acid, this will diminish their imports, which have been from 100,000 to 125,000 tons per annum, or 25 to 30 per cent. of the total production of Sicily.

(8.) For the above reasons, it seems more than doubtful if the Anglo-Sicilian sulphur trust can succeed in maintaining the prices at the present artificial point for any length of time. Its capital is 750,000*l*. (3,619,885 dols.), and it has purchased its brimstone at about 65*s*. (15·80 dols.) per ton free on board Sicily, plus all the other expenses.

Exportation of Sulphur from Italy.

U.S. Cons. Reps., June 1897, 204.

According to statistics published by the general direction of the revenue, the sulphur exported from Italy to foreign countries during the year 1896 reached 3,563,700 quintals (1 quintal = 220·16 lb.). The export in 1895 was 3,175,661 quintals; 1894, 2,990,896 quintals; 1893, 3,108,674 quintals; 1892, 2,910,307 quintals.

The exports according to countries, as compared with the preceding year, were as follows:—

Countries.	1895.	1896.
	Quintals.	Quintals.
Austria-Hungary	146,067	180,222
Belgium	60,182	71,328
France	755,291	750,758
Germany	174,895	183,896
Great Britain	263,516	248,932
Greece	114,500	130,410
Holland	42,380	44,759
Portugal	71,356	111,922
Russia	180,004	167,042
Spain	120,620	82,140
Sweden and Norway	47,992	115,761
Turkey	54,752	60,134
United States	999,733	1,304,482
South and Central American States	65,527	27,213
Other countries	75,846	82,910
Total	3,175,661	3,563,700

GENERAL TRADE NOTES.

German Chemical Profits.

Chem. and Druggist, July 31, 1897, 214.

Dr. Otto N. Witt, President of the German Society for the Protection of Chemical Interests, has published an interesting pamphlet on wages and profits of the German chemical industry, from which the following figures are taken. The average dividends paid between the years 1886 and 1895 by various chemical companies in Germany were as follows:—Industries of alkalis and acids, 7·4 per cent.; pharmaceutical, photographic, and chemico-technical preparations, 12·6 per cent.; coal-tar dyes, 19·2 per cent.; explosives, 16·5 per cent.; artificial manures, 7·5 per cent. The profits in the industry of acids and alkalis show the smallest annual fluctuations, while those on pharmaceutical preparations exhibited the largest difference. The average dividend of the coal-tar dye industry has increased without a break and with remarkable equality since 1886. In that year the average dividend was not quite 10 per cent. In 1891 it was over 20 per cent., and between 1892 and 1895 the average has been 23·45 per cent. Unfortunately there is no means of showing what proportion of this increased profit belongs to the coal-tar remedies, which are now cultivated by most of the factories with at least as much care as the manufacture of colours itself. Companies, however, only form a small portion of the chemical industry. With regard to private firms, there is reason for believing that their average profits amount to from 6 per cent. to 8 per cent. on the capital invested. As a set-off to this profitable condition of things, it must be taken into account that burdens on the industry, in consequence of enforced contributions towards insurance of workmen, &c., are increasing every year. Wages also have risen considerably. In 1891 the average wage of each working man employed in the chemical industry was only 836 m.; in 1894 it had risen to 885 m., and in 1896 to 907 m. In addition to the sick and invalid insurance, the compulsory insurance for accidents absorbs enormous amounts. As an instance, it is mentioned that one chemical factory, employing 1,247 insured persons, paid in 1896 40,849 m. towards accident insurance, 8,640 m. towards invalid insurance, and 11,629 m. towards the sick fund. In addition to this total of 61,180 m., the factory in the same year paid fully 12,000 m. in voluntary assistance to its workmen.

Production of Cellulose in Sweden.

Board of Trade J., July 1897, 91.

The production of cellulose in Sweden has, according to a statement in *Le Monde Economique*, rapidly increased since 1867. The following table shows the amount produced in quinquennial periods from 1867 to 1896:—

Periods of 5? Years.	Production by Mechanical Method.	Production by Chemical Method.
	Tons.	Tons.
1867—1871	61,700	..
1872—1876	97,800	5,350
1877—1881	140,000	18,240
1882—1886	194,000	110,000
1887—1891	296,000	202,400
1892—1896	455,000	416,200

Platinum in New South Wales.

Eng. and Mining J., July 17, 1897, 67.

Among the minerals produced in New South Wales during 1896, platinum occupies a position for the first time, the output being valued at 3,479*l.*, says the *Australian Mining Standard*. The production of platinum for many scientific purposes is assuming great importance, and it is to be hoped that the discoveries which have been made in New South Wales will lead to the establishment of a permanent and lucrative industry.

Works' Effluents in Sewers.

The Leeds Regulations.

Chem. Trade J., July 10, 1897, 19.

The Streets and Sewerage Committee of the Leeds Corporation have decided that the following resolutions of the Committee be extended to include the liquid trade refuse from all manufactories (if any) which the Medical Officer of Health is of opinion would unduly interfere with the purification of the sewage, or which would tend to produce an effluent injurious to agriculture if applied thereto, or which would create a nuisance in the sewers injurious to health, or would be calculated to damage the structure of the sewer:—

That the liquid trade refuse from tanneries and leather works be permitted to be discharged into the public sewers on the conditions:—1. That an examination shaft be built in each manufactory over the drain discharging into the sewer, of such dimensions and in a manner to enable the Medical Officer of Health to take samples from the passing liquid refuse whenever he may desire to do so. 2. That the exercise of this permission be only commenced and continued after the Medical Officer of Health shall have certified that such shaft has been built to his satisfaction, and that the works for preliminary depuration of the liquid refuse, if any, have been made, brought into operation, and be continued to his satisfaction. That no certificate be issued by the Medical Officer of Health in accordance with the previous resolution, without the sanction of the chairman or deputy-chairman of this Committee.

It was also decided that, in addition to the foregoing, the following conditions be imposed:—

That, wherever required by the Medical Officer of Health, works for the preliminary depuration of liquid refuse be carried out by the manufacturers. That the liquid refuse from all breweries, in addition to being subject to the foregoing requirements and to those of the Medical Officer of Health, shall be passed through a sieve of $\frac{1}{16}$ of an inch mesh, to be provided by the owner or occupier of the premises.

The Importation of Chemical Products into Portugal.

Pharm. J., July 3, 1897, 18.

The importation of chemical products into Portugal during 1895 as compared with the value in 1894 was as follows, the percentages showing the increase:—Pyroligneous acid, 1,000*l.* (107 per cent.); arsenious acid, 250*l.* (70 per cent.); muriatic acid, 2,800*l.* (170 per cent.); nitrate of potash, 10,800*l.* (23 per cent.); nitrate of soda, 5,100*l.* (40 per cent.); iodine, 580*l.* (15 per cent.); phosphorus, 1,300*l.* (97 per cent.); refined carbonate of potash, 1,580*l.* (76 per cent.); unrefined carbonate of potash, 484*l.* (53 per cent.); carbonate of soda, unrefined, 340*l.* (44 per cent.); chloride of sodium, 1,009*l.* (79 per cent.); sulphate of soda, 1,920*l.* (45 per cent.); sulphate of potash, 520*l.* (1,826 per cent.); sulphate of iron, 1,360*l.* (870 per cent.); sundries unspecified, 55,600*l.* (6 per cent.). Nitrate of silver, 620*l.*, is nearly the same as previous year, and the same in the case of the value of sulphate of copper imported, 36,920*l.*, although the quantity decreased by some 500 tons. The following show a decrease:—Sulphuric acid, 79*l.* (41 per cent.); borate of soda, 355*l.* (51 per cent.); carbonate of soda, dry, 11,900*l.* (9 per cent.); chloride of lime, 4,200*l.* (27 per cent.); salts of quinine, 1,700*l.* (27 per cent.).

Carbide of Calcium and the Petroleum Acts.

In view of the growing importance of the manufacture and the use of carbide of calcium for illuminating purposes, it was considered desirable by the State authorities to promulgate certain regulations with regard to its sale and storage, which were formally specified in an Order in Council, dated February of this year.

The regulations appear to be founded very largely upon the Petroleum Acts, 1871–81, but as the regulations referred to in this connection might be considered as not sufficiently explicit without further definition, the Acetylene Illuminating Company, manufacturers of carbide of calcium at the Falls of Foyers, have issued a pamphlet which contains an abstract of both the Petroleum Acts and the regulations of the London County Council applicable to the storage of carbide of calcium.

It is pointed out in the preliminary observations made by the Acetylene Illuminating Company that it is optional for all local authorities to modify the suggestions made by the London County Council in any manner they may think desirable.

The Acetylene Illuminating Company, for some months past, have been in correspondence with the Home Office. We have it upon their authority that the Home Office are anxious to avoid any measures which might result in an obstruction to the new industry, at the same time entertaining a careful and proper regard for the public safety.

BOARD OF TRADE RETURNS.

Summary of Imports.

Articles.	Month ending 30th June	
	1896.	1897.
	£	£
Metals	1,675,956	1,881,128
Chemicals and dyestuffs	497,063	401,507
Oils	614,539	6[illegible]0,226
Raw materials for non-textile industries.	4,499,017	5,025,197
Total value of all imports	35,229,255	36,321,809

Summary of Exports.

Articles.	Month ending 30th June	
	1896.	1897.
	£	£
Metals (other than machinery)	3,034,046	2,793,784
Chemicals and medicines	681,237	628,031
Miscellaneous articles	2,840,879	2,751,040
Total value of all exports	20,560,061	19,089,997

Imports of Metals for Month ending 30th June.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Copper:—				
Ore .... Tons	4,509	8,674	73,307	44,507
Regulus .... „	5,123	7,567	123,348	176,778
Unwrought ..... „	5,290	5,561	259,868	274,457
Iron:—				
Ore .... „	504,856	562,334	352,455	424,809
Bolt, bar, &c. ... „	7,712	6,761	58,388	51,662
Steel, unwrought .. „	2,351	1,344	16,906	28,300
Lead, pig and sheet „	12,808	12,620	138,898	149,972
Pyrites .... „	47,792	47,356	82,741	80,450
Quicksilver .... Lb.	40,427	978,960	3,393	89,481
Silver ore .... Value £	..	..	110,172	86,466
Tin .... Cwt.	45,170	47,768	135,856	145,357
Zinc .... Tons	6,987	6,436	114,584	111,366
Other articles ... Value £	..	..	205,950	215,513
Total value of metals	..	..	1,675,956	1,881,128

Imports of Oils for Month ending 30th June.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Cocoa-nut .... Cwt.	13,342	15,096	15,840	16,918
Olive .... Tuns	1,765	1,910	55,616	59,444
Palm .... Cwt.	107,062	126,324	107,355	125,311
Petroleum .... Gall.	13,191,061	14,431,962	248,624	261,917
Seed .... Tons	1,983	1,622	40,811	33,197
Train, &c. .. .. Tuns	1,173	1,141	21,178	19,318
Turpentine .... Cwt.	53,354	29,082	50,527	29,894
Other articles .. Value £	..	..	74,588	74,197
Total value of oils...	..	..	644,539	620,226

Imports of Raw Material for Non-Textile Industries for Month ending 30th June.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Bark, Peruvian .. Cwt.	1,204	1,847	2,195	3,186
Bristles .... Lb.	100,388	255,375	15,762	34,021
Caoutchouc ...... Cwt.	31,064	25,286	335,541	264,635
Gums:—				
Arabic .... „	10,069	6,864	23,111	10,363
Lac, &c. .... „	8,853	17,556	41,221	73,697
Gutta-percha .... „	5,267	3,471	46,813	34,777
Hides, raw:—				
Dry .... „	29,615	51,034	76,959	141,838
Wet .... „	51,966	60,888	115,842	125,794
Ivory .... „	781	1,002	33,652	39,704
Manure:—				
Guano .... Tons	6,669	1,075	27,249	3,650
Bones .... „	2,947	4,932	10,579	12,164
Nitrate of soda... „	6,295	8,948	48,089	67,398
Phosphate of lime „	22,188	28,051	31,915	42,578
Paraffin .... Cwt.	56,222	48,847	52,038	44,095
Linen rags .... Tons	1,866	1,984	17,012	19,890
Esparto .... „	17,952	18,268	74,805	70,671
Pulp of wood .... „	27,180	37,388	138,899	182,172
Rosin .... Cwt.	146,155	192,984	33,753	46,909
Tallow and stearin „	219,117	246,330	220,784	229,678
Tar .... Barrels	5,982	2,588	2,650	1,110
Wood:—				
Hewn .... Loads	256,291	238,732	518,224	498,908
Sawn .... „	789,886	898,272	1,700,236	2,077,011
Staves .... „	15,101	10,915	52,760	37,525
Mahogany ..... Tons	2,732	3,730	23,433	35,212
Other articles .... Value £	..	..	855,500	928,211
Total value	..	..	4,499,017	5,025,197

Besides the above, drugs to the value of 68,670*l.* were imported, as against 71,841*l.* in June 1896.

Imports of Chemicals and Dyestuffs for Month ending 30th June.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Alkali .... Cwt.	18,715	28,325	10,990	10,253
Bark (tanners', &c.) „	45,093	36,168	21,007	12,304
Brimstone .... „	12,858	11,100	2,832	9,587
Chemicals ...... Value £	..	..	131,671	120,123
Cochineal .... Cwt.	167	466	1,019	2,951
Cutch and gambier Tons	1,256	1,802	27,685	30,281
Dyes:—				
Alizarin .... Value £	..	..	21,215	16,391
Anilin and other „	..	..	39,125	32,544
Indigo .... Cwt.	1,680	1,708	53,790	26,674
Nitrate of potash . „	25,576	38,331	21,117	26,369
Valonia .... Tons	2,053	1,426	31,521	14,818
Other articles ... Value £	..	..	135,061	99,122
Total value of chemicals	..	..	497,033	401,507

Exports of Metals (other than Machinery) for Month ending 30th June.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Brass .... Cwt.	9,840	10,622	39,127	43,750
Copper:—				
Unwrought .... „	42,389	29,753	105,055	76,667
Wrought ...... „	23,358	23,442	69,893	71,656
Mixed metal ... „	7,814	13,126	19,095	31,581
Hardware ...... Value £	..	..	183,389	173,796
Implements ..... „	..	..	126,192	100,125
Iron and steel ... Tons	350,575	317,309	2,260,873	2,029,363
Lead .... „	5,141	3,260	62,221	42,431
Plated wares ... Value £	..	..	29,602	28,322
Telegraph wires . „	..	..	19,680	88,340
Tin .... Cwt.	9,274	8,609	30,067	27,901
Zinc .... „	17,850	15,773	12,782	11,686
Other articles .. Value £	..	..	75,770	68,163
Total value	..	..	3,034,046	2,793,784

Exports of Miscellaneous Articles for Month ending 30th June.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Gunpowder ...... Lb.	786,000	936,200	23,158	21,308
Military stores .. Value £	..	..	179,637	188,219
Candles .... Lb.	2,013,000	1,784,500	29,732	24,045
Caoutchouc ..... Value £	..	..	96,874	86,147
Cement .... Tons	36,324	32,138	60,772	52,633
Products of coal Value £	..	..	149,710	133,323
Earthenware ... „	..	..	142,286	169,365
Stoneware ...... „	..	..	18,848	12,444
Glass:—				
Plate .... Sq. Ft.	113,183	94,706	6,868	5,559
Flint .... Cwt.	9,036	6,149	19,389	13,305
Bottles .... „	61,758	65,333	29,241	31,168
Other kinds .... „	18,567	17,405	15,227	15,135
Leather:—				
Unwrought .... „	12,398	11,575	116,936	106,026
Wrought ..... Value £	..	..	27,009	34,503
Seed oil .... Tons	4,583	4,216	87,927	70,674
Floorcloth ..... Sq. Yds.	2,054,800	1,899,300	82,751	77,879
Painters' materials Val. £	..	..	156,544	129,656
Paper .... Cwt.	91,479	78,449	141,047	122,009
Rags .... Tons	4,015	4,288	22,300	21,479
Soap .... Cwt.	65,055	61,008	64,438	62,111
Total value	..	..	2,840,879	2,751,040

Exports of Drugs and Chemicals for Month ending 30th June.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Alkali............ Cwt.	368,448	440,186	94,983	112,532
Bleaching materials ..	102,372	95,958	34,299	29,998
Chemical manures Tons	29,117	18,902	121,894	103,849
Medicines....... Value £	..	..	89,422	92,015
Other articles... "	..	..	340,639	289,637
Total value	..	..	681,237	628,031

Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—PLANT, APPARATUS, and MACHINERY.

Applications.

14,387. F. C. Roberts. Improvements in furnace tuyères. June 14.

14,406. A. J. Boult.—From A. Vandenberghe, Belgium. Improvements in or relating to apparatus for the manufacture of acetylene, sulphuretted hydrogen, carbonic acid, and other gases.

14,408. A. J. Boult.—From F. Würtenberger, Italy. Improvements in or relating to regenerative furnaces. Complete Specification. June 14.

14,539. J. H. R. Dinsmore. Improvements in and connected with thermometers. June 16.

14,778. H. Mawhood. A new system of reverberatory furnace, with retorts for calcining divers products. June 18.

14,896. F. H. Addis. Improvements in or connected with apparatus for cooling air. June 19.

14,933. R. Kurka. Improvements in or relating to filters. June 19.

15,105. S. C. Davidson. Improvements in apparatus for drying, or partially drying, or otherwise treating tea leaf, tobacco leaf, coffee, grain, malt, wool, and other substances. June 23.

15,106. S. C. Davidson. Improvements in apparatus for the drying or other treatment of tea leaf, tobacco leaf, coffee, grain, malt, fruits, and other substances. June 23.

15,107. S. C. Davidson. Improvements in apparatus for applying aqueous vapour, or moist air, or gases to substances, especially applicable for softening, limping, or withering tea leaf, moistening or "ordering" tobacco leaf, or for similar treatment of other analogous substances. June 23.

15,117. J. H. Bowley. Improvements in apparatus for controlling atmospheric conditions. Complete Specification. June 24.

15,223. E. Pamphilon, H. J. R. Pamphilon, and E. Wright. Improvements in triturating apparatus. June 25.

15,231. A. Huillard. An improved mode of and apparatus for desiccating various substances. June 25. Date applied for Jan. 7, 1897, being date of application in France.

15,381. W. J. Baker. Improvements in and relating to apparatus for purifying water and steam, and for heating and softening water. June 28.

15,402. E. Sterné. *See* Class II.

15,529. G. C. Henning. Recording or indicating apparatus for testing materials. Complete Specification. June 29.

15,740. J. R. Green. Improvements in non-conducting coverings for boilers, pipes, and for other purposes. July 2.

15,917. J. R. Green. Improvements in non-conducting coverings for boilers, pipes, and for other purposes. July 5.

16,035. A. J. Boult.—From A. G. Waterhouse and A. W. Case, United States. Improvements in or relating to heating fluids and distilling. Complete Specification. July 6.

16,237. P. Gerlach. Improved cooling apparatus for beer and other liquids. Complete Specification. July 8.

16,398. R. Haddan.—From H. Colberg, A. Serret, and F. Amigo. An improved furnace for the production of carbide of calcium. Complete Specification. July 10.

16,454. R. D. Bailey and L. P. Ford. *See* Class XVII.

16,513. R. T. Hewlett. An improved apparatus for scalding, pasteurising, or sterilising milk or other fluids. July 13.

16,787. W. Lockwood. An improved method of generating steam and other vapours. July 15.

16,836. J. Johnson. Worm liqueur cooler and refrigerator. July 16.

Complete Specifications Accepted *

1896.

10,763. F. Svoboda. Improvements in and connected with Siemens' regenerative furnaces. June 30.

15,671. W. Waddell and D. Waddell. *See* Class XI.

15,721. W. H. Watkinson. Improvements in steam boilers and in vessels for heating and cooling fluids. June 23.

16,555. N. McG. Greaves. Improvements in appliances for the prevention of smoke from furnaces. July 14.

17,530. G. Helps. Improvements in and relating to regenerative furnaces for heating gas retorts and the like. June 30.

18,137. J. L. Dobell. Improvements in means or apparatus for condensing nitrous acid or other fumes or vapour. July 14.

18,377. C. W. Ramstedt. Improvements in apparatus for heating or cooling liquids. July 14.

19,297. W. R. Lake. From R. Spasciani. Improvements in carboys, demijohns, and the like. July 21.

19,800. T. Preece and A. Preece. Improvements in combined self-producing gas furnaces and muffles for use in enamelling metallic articles. July 14.

20,134. L. F. Haubtman. Improvements in apparatus for clarifying saccharine solutions. June 30.

27,477. J. Grossé. *See* Class XVII.

27,989. F. M. Ashley. Improvements in sterilising apparatus. July 7.

28,718. T. Tennessy. Improvements in and relating to gas generators. July 7.

29,858. F. Altmann. Improved method and apparatus for burning liquid fuel. June 23.

1897.

7888. E. Fournier. Improvements in or relating to apparatus for use in sterilising, disinfecting, and similar purposes. July 14.

8456. J. Muir, E. A. Morse, and F. O. Pierson. Refrigerating apparatus. June 23.

9582. S. H. Johnson. Filter presses. July 21.

10,867. A. Ehrhardt. Linoleum drying and oxidising apparatus. June 30.

11,700. E. Simoneton. Apparatus for filtering liquids. July 21.

14,226. L. Lewis. Composition for preventing incrustation in steam boilers. July 21.

II.—FUEL, GAS, AND LIGHT.

Applications.

14,368. E. E. Probert.—From A. C. Niermeyer, Holland. Improvements in incandescent gas lamps or lanterns principally intended for street use. Complete Specification. June 14.

14,401. G. F. Redfern.—From J. Martin, Victoria. Improved means for supplying vaporised liquid fuel at high temperatures to steam boiler and other furnaces. June 14.

14,406. A. J. Boult.—From A. Vandenberghe, Belgium. *See* Class I.

14,471. H. A. Jones. Improvements in gas apparatus. Complete Specification. June 15.

14,499. J. C. J. Steinbach. Improvements in and relating to mantles and the like for incandescent gas burners. Complete Specification. June 15.

14,505. H. H. Lake.—From the firm Piatti and Co., Italy. Improvements in the manufacture of illuminating gas. June 15.

14,535. T. Watkins. Improvements in oil or spirit vaporising apparatus. June 16.

14,555. C. C. Smith. Improvements in acetylene gas generators. June 16.

14,558. J. A. Murray and G. Davis. Improved candle wick. June 16.

14,594. J. B. I. Julhe. Improvements in the means or apparatus for supplying and burning petroleum or other inflammable liquids for illuminating purposes. June 16. Date applied for March 24, 1897, being date of application in France.

14,598. J. Heathcock. A new or improved manufacture of briquette fuel. Complete Specification. June 16.

14,689. N. Lejeune. Improvements in acetylene gas producers. June 17.

14,700. H. R. Bean and H. Ringwood. An improved purifier and enricher for acetylene gas. June 17.

14,742. T. R. Fowler. Improvements in and appertaining to apparatus for the manufacture of acetylene gas. June 13.

14,822. W. B. Smith. Improvements in refractious substances to be heated by gas, including oxyhydrogen, for illuminating. June 18.

14,835. G. W. Gaskell and R. F. Reeve. A new or improved acetylene gaslight apparatus applicable for use in photographic studios for photographing at night. June 18.

14,841. S. Pitt.—From The Compagnie Internationale des Procédés A. Seigle, France. Apparatus for heating by means of liquid hydrocarbons. Complete Specification. June 18.

14,890. W. Hutchinson. Improvements in and relating to incandescence gas burners. June 19.

14,905. H. Seftou-Jones.—From P. P. H. Macé, France. Improvements in or releating to the production of acetylene gas. June 19.

14,929. A. J. B. Légé and A. D. Pennellier. Improvements in or connected with the production and burning of acetylene gas. June 19.

15,060. C. Kaestner. Improvements in or relating to acetylene gas burners. June 23.

15,125. J. W. Searth. Improved apparatus for the generation of acetylene gas and the like purposes. June 24.

15,261. F. Altmann. Improvements in apparatus for generating a vaporous or gaseous mixture from hydrocarbon containing liquid and water, for the production of a blue flame. June 25.

15,317. M. Graetz. Improvements in oil-burners for the production of non-luminous flames, especially for heating incandescent bodies. June 26.

15,350. A. Zendroni. Improvements in the manufacture of artificial fuel or briquettes. June 26.

15,369. E. F. J. C. Bauweraerts. Improvements relating to the carburetting of illuminating gas, and to apparatus therefor. Complete Specification. June 26.

15,402. E. Sterné. Improvements in capsules or containers for containing compressed or liquefied gases. June 28.

15,483. B. Baron. *See* Class XXII.

15,509. L. V. Pratis and P. Marengo. Improvements in and connected with, and apparatus for the manufacture or production of, hydrogen gas. Complete Specification. June 29.

15,558. F. Crabb and E. W. Hampton. Improved incandescent lamp. June 30.

15,657. W. T. McGarry. Converting peat into coal. July 1.

15,754. F. H. Smith. Improvements in acetylene gas generators. July 2.

15,813. E. Andreoli. Improvements in and connected with the manufacture or production of ozone, and in apparatus therefor. July 2.

15,900. E. Soxhlet, F. Van den Berge, A. Borreman, and V. Borreman. An improved lighting and heating gas, and means for producing the same. July 3.

15,963. A. Kiesewalter. Improvements in incandescence mantles. Complete Specification. July 5.

15,966. A. Salomon and J. Jacquin. Improvements in apparatus for the generation of acetylene gas. July 5.

16,029. P. Pascal. Improvements in and apparatus for the manufacture or production of ozone. July 6.

16,132. F. Stadelmann and Co., Ltd.—From K. Thurnauer, Germany. Improvements in acetylene gas burners. July 7.

16,145. A. George. Improvements in the manufacture of briquette fuel. Date applied for Feb. 20, 1897, being date of application in France. Complete Specification. July 7.

16,344. J. Ackroyd and B. Ackroyd. Improvements in apparatus for making acetylene gas. July 10.

16,751. J. Stevenson. An improved washer for extracting ammonia liquid. July 15.

16,793. O. V. Sigurdsson. Improvements in means employed in the manufacture of acetylene gas. July 15.

16,890. R. H. Whiteley and W. Ellis. Improvements in the method of and apparatus for enriching coal gas. July 16.

16,975. G. C. Marks.—From A. F. Bowers, France. Improvements in apparatus for the generation of acetylene gas. Complete Specification. July 17.

Complete Specifications Accepted.

1896

15,671. W. Waddell and D. Waddell. Assisting combustion, preventing smoke, and economising fuel, and in the means employed therefor. June 30.

17,038. L. T. FitzGibbon. Improvements in and connected with apparatus for generating and storing acetylene gas. July 7.

17,198. B. Grau. Improvements in coking ovens. July 7.

17,605. W. H. Wheatley.—From C. Schmid. Improvements in the manufacture of incandescence bodies for illuminating purposes. July 14.

17,646. M. Duffield. An improved acetylene gas generator. July 21.

17,678. A. C. Fraser. Improvements in process and apparatus for generating and liquefying acetylene. Filed Aug. 10. Date applied for Feb. 21, 1896, being date of application in United States. June 23.

17,778. B. T. L. Thomson. Improved means for producing carbonic acid and hydrogen gases. June 23.

18,819. W. Clark.—From A. Meyenberg, M. Wendorf, and S. Henlein. Improvements in incandescent lamps for the combustion of petroleum. July 14.

18,992. E. Chesnay and L. Pillion. Improvements in apparatus for generating, storing, and delivering acetylene gas. Filed Aug. 27. Date applied for Jan. 31, 1896, being date of application in France. June 30.

19,059. W. P. Thompson.—From D. H. Chivert, France. Improvements in or appertaining to the production of acetylene gas, and apparatus therefor. July 7.

19,126. G. Voigt. Improvements in apparatus for producing acetylene gas. June 30.

19,953. G. Kremer. Improvements in the production, by the cold process, of gas for lighting and motive power, and apparatus therefor. July 21.

20,238. H. Turner and W. Allen. Improvements in or applicable to smoke-burning apparatus. July 14.

20,406. P. Trost. Improvements in acetylene gas lamps. July 7.

20,694. W. P. Thompson.—From V. I. Comucci. Improvements in apparatus for generating and storing acetylene gas. July 21.

22,628. E. A. Morten Brown and E. Mawndrell. Improved apparatus for production and storage of acetylene gas. July 7.

26,784. R. Moll and A. Palmer. A new or improved method of manufacturing incandescent mantles and caps for gas and other burners. July 7.

1897.

1398. W. R. Addicks. Improvements in the manufacture of carburetted water-gas and in apparatus therefor. July 14.

4870. E. J. Frest and B. Middleditch. Improvements in devices for automatically regulating the quality of gas. July 7.

8989. J. G. Kerr and C. Fry. Improved apparatus for the production and storage of acetylene gas. July 7.

10,371. A. A. Stevenson. Composition for making gas. June 23

11,130. A. C. Humphreys and A. G. Glasgow. Apparatus for making carburetted water-gas. July 21.

11,131. A. C. Humphreys and A. G. Glasgow. Apparatus for making carburetted water-gas. July 14.

11,252. F. van den Abeele. Automatic apparatus for supplying incandescent gas burners with gas under pressure. July 14.

12,096. L. J. Davies. Process for making coke from anthracite and similar coal. July 7.

12,556. R. Guatanneus-Moens and E. Carreer-Dilger. Improved automatic apparatus for the production of acetylene gas. June 30.

13,496. F. A. Mitchell. Acetylene gas generators. July 7.

14,015. F. Trendel. Apparatus for producing acetylene gas. July 21.

14,368. E. E. Probert.—From A. C. Mirmeyer. Improvements in incandescent gas lamps or lanterns principally intended for street use. July 21.

14,499. J. C. J. Steinbach. Mantles and the like for incandescent gas burners. July 21.

14,598. J. Heathcock. Manufacture of briquette fuel. July 21.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Application.

15,517. M. Ziegler. Improved process of and apparatus for carbonising peat, lignite, and the like, and the recovery of by-products therefrom. June 29.

Complete Specification Accepted.

1897.

10,577. G. C. Malks.—From La Compagnie pour la Fabrication des Compteurs et Matériels d'Usines à Gaz. Triple-action apparatus for the recovery of benzols and other products contained in certain gases.

IV.—COLOURING MATTERS and DYES.

Applications.

14,375. H. Imray.—From F. Hoffmann and La Roche and Co., Switzerland. Manufacture of phenetidine-sulphonic acid and its salts, as also of acetyl derivatives of the latter. Complete Specification. 14 June.

14,376. H. Imray.—From F. Hoffmann and La Roche and Co., Switzerland. Manufacture of sulphonic acids of guaiacol and of creosote, and of metallic salts of these sulphonic acids. June 14.

14,917. M. Otto. Improvements in the manufacture of colouring or carbinol matters. June 19.

14,918. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of black colouring matters of the anthraquinone series. June 19.

14,919. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of yellow colouring matters from benzoin. June 19.

14,920. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of asymmetric diamidophenyl acridines and their alkyl substitution products. June 19.

14,921. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of green mordant-dyeing colouring matters from halogen substituted fluoresceïnes. June 19.

15,009. R. Barge. Process for separating ortho-anhydro-sulphamine-benzoic acid from para-sulphamine-benzoic acid. Complete Specification. June 21.

15,290. W. McCowan. Improvements in the manufacture of colouring matter. June 26.

15,353. S. Pitt.—From L. Cassella and Co., Germany. Producing diazo salts in a durable form. June 26.

15,377. W. H. Claus, A. Rée, and L. Marchlewski. Production of new polyazo colouring matters. June 28.

15,478. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of dyestuffs. June 29.

15,479. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of colouring matters. June 29.

15,587. H. Koechlin. Improvements in treating paranitraniline reds and analogous colours, when applied on vegetable fibres or silk, to render them fast to light. June 30.

15,630. A. Ashworth. Improvements in the manufacture and production of colouring matters suitable for dyeing unmordanted cotton. June 30.

15,890. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of ortho- and para-nitrobenzylaniline sulphonic acids and their homologues. July 3.

16,306. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of methylphenmorpholine. July 9.

16,483. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of dyestuffs of the triphenylmethane series fast to alkali. July 12.

16,495. J. Y. Johnson. — From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of blue mordant-dyeing colouring matters from dinitroanthraquinones. July 12.

16,496. J. Y. Johnson. — From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of new colouring matters for animal fibre. July 12.

16,713. M. Prud'homme. Manufacture of colouring matters having acid action. July 14.

Complete Specifications Accepted.

1896.

13,093. H. R. Vidal. Manufacture of dyes. Date applied for June 3, 1896, being date of application in France. July 21.

14,931. G. B. Ellis.—From La Société Chimique des Usines du Rhone, anciennement G. P. Monnet et Cartier. Manufacture of pyrocatechin-sulphonic acid and intermediate products. June 23.

15,953. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Production of new dyes relating to the oxyphenanthronaphthazine series, and of new intermediate products and materials therefor. June 23.

17,382. C. Uffelmann. Manufacture of a solid paste or soap compound for dyeing all kinds of textile materials. June 23.

18,020. W. H. Claus, A. Rée, and L. Marchlewski. Production of new azo-dyes. June 30.

18,221. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. Improvements in the manufacture and production of phthalic and sulpho-phthalic acids. June 23.

19,831. S. Pitt.—From L. Cassella and Co. Production of black colouring matter. July 14.

20,676. O. N. Witt. Manufacture of derivatives of para-amido-alpha-naphthol and of colouring matter therefrom. July 21.

21,437. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation. Manufacture of a new black mordant-dyeing colouring matter. July 14.

1897.

12,180. H. Imray.—From The Chemical Works Bindschedler. Obtaining new dyestuffs of the phthaleine series. June 23.

12,181. H. Imray.—From The Basle Chemical Works Bindschedler. Process for obtaining new dyestuffs of the phthaleine series. June 30.

12,961. L. Lederer. Process for the extraction of the hydroxylated ethers of phenol from mixtures. July 21.

14,375. H. Imray.—From F. Hoffmann and La Roche and Co. Manufacture of phenetidine-sulphonic acid and its salts, as also of acetyl derivatives of the latter. July 21.

V.—TEXTILES, COTTON, WOOL, SILK, Etc.

Applications.

14,642. E. Knecht. Improvements in lace manufacture. June 17.

14,739. J. W. Knights. The prevention of nuisance in the retting of flax, hemp, or other fibrous plants. June 18.

14,868. J. M. MacDonald and H. H. Boyle. Improvements in the treatment of rhea grass and fibrous plants, and in apparatus therefor. June 19.

14,927. S. H. Sharp. Improved manufacture or treatment of cotton and other fabrics. June 19.

15,381. G. B. Garlach, of the firm of Kettling and Braun. Improvements in the process of and means for finishing or dressing textile fabrics and other goods. June 28.

15,642. C. W. Radeke. Improvements relating to the treatment of textile vegetable fibres. July 1.

16,518. F. W. Ashton and Co., Ltd., and E. C. Kayser. Improvements in the production of printed textile fabrics. July 13.

Complete Specifications Accepted.

1896.

15,893. J. Waugh.—From Amos and Co. Method of waterproofing fabrics made of fibre, paper-tissues, and the like. June 23.

16,840. E. Edwards.—From F. A. Bernhardt. Improvements in the process of what is known as "mercerising" fabrics composed of vegetable fibres. June 30.

17,841. W. Grimshaw. Improvements in and in means for ornamenting textile and other fabrics or materials. July 7.

18,360. F. N. Turney. Improvements in apparatus for degreasing wool, cotton waste, and other fibrous materials. June 23.

18,517. W. J. A. Donald. See Class XIII.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

Applications.

14,639. W. Woodcock, J. Longthorpe, and W. Dargue. Improvements in machinery for dyeing, sizing, and washing hanks of yarn and slubbing. June 17.

14,831. M. L. Kellogg. Improvements in dyeing hair or fur. Complete Specification. June 18.

15,198. J. H. Gartside.—From Romann, France. Improvements in bleaching textile fibres, yarns, and fabrics. June 25.

15,293. J. Nelson and A. Nelson. Improvements in the method of and apparatus for dyeing fabrics and otherwise exposing them to the action of liquids. June 26.

15,587. H. Koechlin. See Class IV.

15,913. J. T. Reid and H. Thorp. Improvements in dyeing textile fibres, yarns, and fabrics. July 5.

16,191. J. H. Gartside.—From J. J. Romann, France. Improvements in bleaching vegetable textile fibres, yarns, and fabrics. July 8.

Complete Specifications Accepted.

1896.

17,692. C. Dratz. Process and apparatus for printing multicoloured designs upon fabrics and warp chains. July 14.

22,522. A. M. Clark.—From C. H. Boehringer and Sohn. An improved mordanting agent and process for vegetable fibres. July 14.

VII.—ACIDS, ALKALIS, and SALTS.

Applications.

14,836. C. C. Moore. Improvements in the treatment of crude borates and in obtaining boracic acid. June 18.

15,976. E. M. Polet. Improved process relating to the treatment and production of phosphates. July 5.

Complete Specifications Accepted.

1896.

16,877. D. Bray. A still or tank for the distillation, boiling, and holding of acids, alkali, and other corrosive liquids. June 30.

17,127. J. C. Graham. Improved process for the preparation and purification of solutions of copper. July 14.

17,607. J. R. Wylde, J. W. Kynaston, and J. Brock. Improvements in the manufacture of hydrochloric acid free from arsenic. June 23.

17,839. H. Brewer. See Class X.

19,222. J. Y. Johnson.—From The Chemische Fabrik Electron Actien Gesellschaft. Improvements in the treatment of bleaching powder to preserve it from loss of active chlorine. July 21.

1897.

3795. G. G. M. Hardingham.—From T. Huntington and F. Heberlein. See Class X.

13,551. W. Majert. Process for the production of ammonium bichromate. July 21.

VIII.—GLASS, POTTERY, and ENAMELS.

Applications.

14,381. J. Gullich. Apparatus for the covering of earthenware, porcelain, or metallic objects with glaze or enamel. June 14.

14,481. C. E. Blue, jun. Improvements in machines for the manufacture of glassware. Complete Specification. June 15.

15,217. E. Gobbe. Improved continuous kiln for baking pottery or burning cements. June 25.

15,618. T. Küpper. Improved apparatus for glass-blowing. Complete Specification. June 30.

16,062. E. F. W. Hirsch. Improvements in glass-melting plant. July 6.

16,435. F. Ohnemüller. Method for printing upon baked-hard porcelain or other earthenware before glazing. July 12.

16,528. W. Cliff. Improvement in or connected with the enamelling of fire-clay baths and other articles. July 13.

16,873. R. Good and R. Good, jun. Improvements in machinery for manufacturing glass vessels. Complete Specification. July 16.

16,883. D. Laffao. Improvements in apparatus for forming, necking, and annealing glass bottles or other hollow glassware. July 16.

16,947. J. T. Szék. Improvements in the manufacture of floor tiles, and the like. July 17.

16,964. W. Thomson. An improved glass tile. July 17.

Complete Specifications Accepted.

1896.

14,927. T. Hausermann. *See* Class X.

17,599. H. Sturm. Improvements in continuous ceramic furnaces with heated movable soles. July 7.

19,746. F. Hancock and C. B. Winzer. Apparatus for pottery purposes. July 14.

1897.

8484. L. Mondron. Improvements in the manufacture of plates of glass design for covering walls, ceilings, and other surfaces. July 7.

10,691. P. Delorme Fils. Manufacture of hollow castings of glass and the like, and apparatus therefor. June 23.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, and CEMENTS.

Applications.

14,970. J. M. Danielli. Improvements in the imitation of coloured marbles. June 21.

15,050. P. Rückert. Improvements in and connected with artificial stones. Complete Specification. June 23.

15,083. F. D. T. Lehmann and P. N. Kohlsaat. Improvements in fireproof buildings and structures. Complete Specification. June 23.

15,632. E. Marshall Fox. Improvements relating to uninflammable wood. June 30.

16,260. E. P. A. Hugues. Improvements in cement building materials. July 9.

16,664. H. H. Lake.—From J. Seidler and A. J. C. Graf, Germany. Improvements in and relating to the manufacture of artificial blocks or plates for building and other purposes. July 13.

Complete Specifications Accepted.

1896.

16,892. T. R. Jones. An improved process for hardening stone. July 7.

18,295. J. F. Cleine. Improvements in fireproof ceilings and floors. July 7.

19,419. A. McLean. Improvements in artificial stone. July 21.

1897.

8261. J. Howden. Improvements in fireproof floors. July 7.

13,026. W. Chrometzka. Fireproof ceilings. July 7.

14,068. J. A. Fisher. Fireproof ceilings and partitions. July 14.

X.—METALLURGY, MINING, Etc.

Applications.

14,392. C. Wetherwax. Improved method of and means for the preliminary treatment of gold and silver ores. June 14.

14,438. W. G. M. Foote. Improved method of and apparatus for extracting metals from their ores or other compound bodies containing them. Complete Specification. June 15.

14,567. O. Herrmann. An improved alloy. June 16.

14,662. A. S. Bower. Improvements in the treatment of spent oxide of iron for obtaining products of commercial value. June 17.

14,693. T. Levoz. Improvements in casting ladles for steel and iron. Complete Specification. June 17.

14,833. J. F. Fairley. Improvements in the process of annealing metals. June 18.

15,115. S. Alley.—From J. Campbell and J. W. H. James, United States. Improvements in furnaces for treating ores containing precious metals and other ores. June 24.

15,203. S. Alley. Improvements in and connected with furnaces for treating ores containing precious metals and other ores. June 25.

15,455. A. E. Thomas. To refine or purify Cleveland or other impure iron (to make it suitable for use in the manufacture of cast-iron work, wrought iron, and wrought and cast steel). June 29.

15,569. T. G. Bowick. Improvements in and relating to the recovery of gold and other precious metals. Complete Specification. June 30.

15,631. T. A. Middleton and H. R. Gregory. Improvements in and connected with the extraction of gold from solutions containing the same. June 30.

15,674. C. Wetherwax. An improved process of treating gold ores. July 1.

15,700. W. Muir. Improvements in the manufacture of potassium and sodium. July 1.

15,742. R. McAlley. New process for the production of cobalt and its oxides from its ores, and of by-products therefrom. July 2.

15,791. N. W. Edwards. A new or improved method of and process for treating antimonial and antimonial gold ores. July 2.

15,999. J. Parry and G. H. Llewellyn. Improvements in the manufacture of iron and steel. July 6.

16,127. S. Curteis and J. E. Chaster. Improvements in the extraction of gold from ores or other substances containing same. July 7.

16,808. F. Ellershausen. Improvements in the treatment of complex sulphide ores and apparatus therefor. Complete Specification. July 15.

16,922. H. J. Kloos. An improved powder for use in welding metals, and process of manufacturing same. July 17.

Complete Specifications Accepted.

1896.

13,565. J. W. Spencer. Face-hardening metals. June 30.

13,641. C. J. Claus, jun. Improvements in or relating to the treatment of tin or antimony ores, and the separation of the gold and silver contained therein. June 23.

14,493. J. Garnier. Processes and apparatus for classifying, amalgamating, and cyanising gold and silver ores. Filed June 30. Date applied for May 7, 1896, being date of application in France. July 7.

14,927. T. Hausermann. Process for dyeing or ornamenting objects of metal, porcelain, or the like. July 7.

16,032. W. H. Hyatt. Improvements in apparatu for separating gold from other materials. July 21.

16,831. W. T. Sugg. Improved metallic alloy. July 7.

17,299. E. F. Turner. An improved process for the treatment of zinciferous ores. June 30.

17,839. H. Brewer. Improvements in and relating to the treatment of zinc ores or ore residues in the production of zinc, chlorine, sodium sulphate, and other products. June 23.

18,616. J. Legge. Improvements in puddling and other like furnaces. June 30.

19,178. J. Shanks. Improvements in moulds for casting. July 7.

19,140. J. Lones and E. Holden. An improvement or improvements in the manufacture of buffer-iron or steel for buffer-barrels or cylinders of railway carriages, trucks, and waggons. June 30.

19,640. W. van Wert, F. W. Poop, and J. J. Bradley. Improvements for producing certain kinds of metal, especially adapted for use in the manufacture of the tubes and other of the component parts of cycles of all kinds, autocars, and other wheeled vehicles, which improvements are also applicable for other industrial purposes. July 14.

19,718. G. Bennett. Improvements in ingot moulds for the casting of ingots of iron, steel, or other metals, such as iron or steel alloys. July 7.

20,131. W. Roberts. Improvements in the machinery or appliances for the manufacture of plates and sheets of copper, brass, and German silver. July 14.

24,657. R. F. Strong. Improvements in the treatment of ores for the production of iron, steel, and other metals. June 30.

1897.

3795. S. G. M. Hardington.—From T. Huntington and F. Herberlein. Improvements in the treatment of sulphide ores of lead preparatory to smelting the same, and incidentally in obtaining sulphurous acid. July 7.

8376. G. Kentler and F. Steinert. Process and apparatus for recovering iron for slag-heaps. June 23.

8873. W. Noad and W. Agate. Improvement in the obtainment of copper from ores and other copper-bearing substances. May 19.

14,693. T. Levoz. Casting ladles for steel and iron. July 21.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

Applications.

14,508. I. Kitsée. Improvements in primary batteries or cells. Complete Specification. June 15.

14,708. G. H. Nash. Improvements in apparatus for coating metal articles by electro-deposition. June 17.

14,709. G. H. Nash. Improvements in apparatus for coating metal articles by electro-deposition. June 17.

15,013. S. Pitt.—From L. Bomel and Société Bisson, Bergès, et Cie., France. Negative electrode for zinc accumulators. Complete Specification. June 21.

15,047. A. Heinemann and W. Schäfer. Improvements in and relating to electric accumulators or storage batteries. June 23.

15,080. A. J. Boult.—From J. Trillet, France. Improvements in or relating to electric batteries. June 23.

15,090. H. C. Carter. Improvements in electric accumulators. June 23.

15,222. L. Fiedler. Improvements in and relating to electric accumulators. June 25.

15,323. A. Heil. Improvements in electrical accumulators. June 26.

15,510. J. Y. Johnston.—From J. P. Clare, G. E. Hatch, J. H. Taylor, United States. Improvements in secondary or storage batteries or accumulators and in the manufacture of the same. Complete Specification. June 29.

15,513. C. A. Jensen.—From P. Jebsen, Norway. Process for producing peat charcoal by means of an electrical current. Complete Specification. June 29.

15,850. J. B Brooks and J. Holt. Improvements in certain apparatus to be employed in connection with the electro-deposition of metals. July 3.

15,879. W. J. S. Barber-Starkey. Improvements in and relating to plates for secondary batteries. July 3.

15,903. A. D. Seton and J. L. Dobell. Improvements in or connected with electric batteries. July 3.

15,939. E. A. Jahncke. Improvements in the manufacture of protective coverings or envelopes for zinc plates of electric batteries and for other purposes. July 5.

15,941. E. A. Jahncke. Improvements in or connected with electric batteries. July 5.

16,069. L. W. Pullen. Improvements in electric batteries. Complete Specification. July 6.

16,361. E. W. Jungner. Improvements in or relating to accumulator plates for secondary batteries. Complete Specification. July 10.

16,362. E. W. Jungner. Improvements in or relating to the electrodes of electric accumulators. Complete Specification. July 10.

16,665. C. Therye. Improvements in electric batteries. July 13.

16,797. G. J. Erlacher and M. A. Besso. Improved controlling apparatus for accumulator batteries. July 15.

16,816. H. Leitner. Improvements in the construction of secondary batteries. July 15.

16,942. C. Bertolus. An electric furnace for the application of polyphase currents in processes of fusion or decomposition by means of the voltaic arc. Complete Specification. July 17.

Complete Specifications Accepted.

1896.

7222. A. S. Elmore.—From J. O. S. Elmore. Apparatus for manufacturing tubes by electrolytic deposit. July 14.

13,688. A. E. Bonna, A. Le Roger, and P. van Berchem. Apparatus for the treatment of air, gases, and vapours. June 23.

16,057. C. Kellner. Improvements in electrolytic apparatus. July 7.

17,608. The Cowper-Coles Galvanising Syndicate, Ltd., and S. O. Cowper-Coles. Improvements in means or apparatus for effecting the electro-deposition of zinc or other metals upon tubes or other circular or analogous objects. July 14.

17,671. L. Hoepfner. An improved method for obtaining porous metals by electrolysis. June 23.

19,308. W. Walker, jun., F. R. Wilkins, J. Lones, and J. Lones. Improvements in utilising the residual products obtained in the working of certain kinds of voltaic batteries. July 14.

25,365. H. Woodward. An improved electrode for secondary batteries. July 7.

XII.—FATS, OILS, AND SOAP.

Applications.

15,156. A. Jolles. A new manufacture of soaps and process therefor. Complete Specification. June 24.

15,927. W. Smith and J. E. T. Woods. Improvements in the purification and conversion of certain residual hydrocarbon oils produced in the making of camphor substitutes into oils suitable as disinfectants, and for other useful purposes. July 5.

15,972. C. Romen. Process for producing a disinfecting, deodorising, and bleaching soap powder. July 5.

16,382. F. Becker and O. Wolff. Improvements in the manufacture of soaps or cleansing compositions. July 10.

Complete Specifications Accepted.

1896.

14,308. A. Schuchardt and T. Otto. New washing substance and process for making it. July 7.

15,336. T. Coad. Improved treatment of fat or fatty waste. July 21.

1897.

13,807. I. Kohn and M. Kohn. Transparent soap blocks or tablets containing inscriptions. July 14.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

14,439. H. Edmunds. Improvements in the manufacture or production of an insulating material, and the application thereof to the insulation of electrical conductors. June 15.

14,911. P. Reidel and E. Kuhn. Improvements in the manufacture of a compound or material applicable for use as a substitute for leather and india-rubber, and for floor coverings and the like, and in apparatus therefor. June 19.

15,180. P. S. Watkins and P. J. Nunn. Improvements in the manufacture of sealing wax. Complete Specification. June 24.

15,269. F. Fenton. Further improvements in the production of artificial india-rubber and gutta-percha-like substances. June 26.

15,330. B. J. B. Mills.—From La Granitine Société Anonyme, Belgium. Process of manufacture of agglutinant enamels. Complete Specification. June 26.

15,678. R. Matthews. An improvement in the manufacture of white lead. July 1.

16,061. M. Greenberg. A liquid tarnish protector for unlacquered metals. July 6.

16,147. S. Rosenblum and S. Rideal. Improvements in the production of chemical compounds from a certain oil or oils, or the fatty acids of same, and the employment of such compounds in the manufacture of drying oils, varnishes, enamels, lacquers, anti-fouling compositions, and the like. July 7.

16,490. J. Stocker and H. Zander. An improved weather acid and fire-resistant material. Complete Specification. July 12.

Complete Specifications Accepted.

1896.

14,643. C. Day. Improved liquid varnish gum. June 23.

16,276. A. Gutensohn. Improvements in the manufacture of oxide of lead and means used therefor. July 7.

17,078. H. W. Aird and C. E. Felch. Improvements in paint and in method of producing and applying same. June 23.

18,485. W. Mouson. Improvements in acid-proof electrical non-conducting articles and composition therefor. July 21.

18,517. W. J. A. Donald. Improvements in the impregnating of materials with india-rubber, gutta-percha, and the like. July 7.

18,864. D. Forbes and J. S. Weymouth. A gelatinous resilient composition applicable for wheel tyres and other purposes. June 30.

19,046. Siemens Bros. and Co., Ltd., and E. F. A. Oballa. Improved process and apparatus for the extracting of gutta-percha from the leaves and twigs of the Isonandra Gutta and kindred plants. July 14.

1897.

5121. J. G. Ingram. Improvements in the manufacture of parti-coloured india-rubber. June 23.

11,500. T. Grantham and J. J. Grantham. Improved boot varnish. July 21.

14,105. F. Boyling, C. L. Garland, and A. Ogden. Enamel paint, and method of applying the same. July 14

XIV.—TANNING, LEATHER, GLUE, and SIZE.

Applications.

15,352. A. Mario. Improvements in or connected with tanning. June 26.

15,404. A. J. Boult.—From G. Brigalant, France. Improved manufacture of artificial leather or leather substitute. Complete Specification. June 28.

16,477. J. L. Garcin. Improvements in or relating to the manufacture of washable leather or skins. Complete Specification. July 12.

16,804. C. Michel. Improved process and apparatus for the rapid tanning of skins and hides. July 15.

16,927. J. T. McQuinn. Improvements in quick tanning process. July 17.

Complete Specifications Accepted.

1896.

21,210. W. Clark.—From C. H. Boehringer and Sohn. Improvements in tanning hides and skins. July 7.

21,774. H. Gardner.—From C. W. Suther. Improved manufacture of waterproof glue. June 23.

1897.

13,778. W. Sagar, sen., A. P. Sager, and W. Sager, jun. Apparatus for removing grease or fatty matter from sheepskins and other leather and the like. July 14.

XV.—AGRICULTURE and MANURES, Etc.

Application.

14,695. E. Edwards.—From C. J. R. Le Mesurcer, Ceylon. An improved manure. June 17.

Complete Specifications Accepted.

1896.

17,151. C. Fell. The natural fertiliser. July 21.

1897.

13,811. I. Kovacs. Process for making a consistent friable artificial manure. July 14.

XVI.—SUGARS, STARCHES, GUMS, Etc.

Applications.

14,647. H. W. Aitken. Improvements in sugar-cane mills. June 17.

15,144. J. G. Boers. Improvements in means and apparatus for depositing or crystallising sugar or other substances upon confections or other articles. June 24.

15,295. L. P. Bauer. Improvements in the manufacture of glucose or sugar. Complete Specification. June 26.

Complete Specifications Accepted.

1896.

20,434. L. F. Haubtman. Improvements in apparatus for clarifying saccharine solutions. June 30.

1897.

11,842. Z. Lubinski and R. Krajewski. Centrifugal machines for use in the manufacture of sugar. July 7.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

14,454. B. J. Atterbury. Improvements in the manufacture of alcohol and in apparatus therefor. June 15.

15,200. A. Fraser and Maclay and Co., Ltd. Improvements in or relating to the manufacture of pale ale. June 25.

15,825. G. Pütz. Improvements in or relating to the clarifying or fining of worts and apparatus therefor. Complete Specification. July 2.

16,232. H. Hempel. A process for producing a carbonaceous agent for enriching spirit. Complete Specification. July 8.

16,454. R. D. Bailey and L. P. Ford. An improved false bottom for the mash tuns, conversion vessels, hop backs, and malt and hop kiln floors found in breweries, distilleries, malt houses, hop factories, glucose manufactories, and vinegar works. July 12.

16,503. A. J. Boult.—From P. E. Paulet, France. Improved process for the rectification of wines, spirits, and other alcoholic liquors. July 12.

16,609. W. B. Turner. Improvements in the treatment or preparation of malt, corn, or grain for brewing or other purposes. July 13.

16,836. J. Johnson. *See* Class I.

Complete Specifications Accepted.

1896.

19,245. A. C. Field. Improvements relating to malting. July 7.

19,616. K. Pattermann. Improvements in or connected with germination of barley, and apparatus therefor. July 7.

27,477. J. Grossé. An improved evaporating or vacuum apparatus for the granulation of crystallisable liquids. July 14.

1897.

4926. C. Ameye. A machine for aërating wine, beer, and other fermented and unfermented liquids. June 30.

6463. F. W. H. Hempel. Process for producing luminous spirit. June 23.

14,106. N. Bendixen. Propagating apparatus for developing pure culture of yeast and bacteria. July 21.

XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

Applications.

A.—Foods.

14,522. J. E. Lyndall. A new food or food ingredient. June 15.

14,621. R. Haddan.—From A. Mamy, France. Improvements in the process of preserving food. June 16.

15,145. T. Hill-Jones. The manufacture of a new alimentary extract. Complete Specification. June 24.

15,884. L. Michon and M. F. Gouliet. An improved process or method of preserving alimentary products. Complete Specification. July 3.

16,088. W. Horlick. Improvements in edible and soluble tablets, and method of producing the same. Complete Specification. July 6.

16,154. J. A. Parker. An improved method of preserving fruit, eggs, and other perishable articles. July 7.

16,338. E. Wadham. A new or improved medicinal and nutritive compound or preparation. July 10.

16,586. J. Hogarth. Improvements in and relating to the treatment of alimentary substances and beverages. July 13.

16,930. F. A. Furlonge. An improved process for preserving fresh meat or produce for transport. July 17.

B.—Sanitation.

14,895. J. O. Braithwaite. Improvements relating to the purification of water. June 19.

16,103. E. W. Ives. An improved method of extracting the precipitated sludge from the bottom of flat-bottomed tanks used in the purification of sewage and manufacturers' waste waters. July 7.

16,925. C. G. T. Dixon, J. Phythian, and F. F. Bennett. Improvements in the treating of town refuse in destructors, and manufacturing certain by-products therefrom. July 17.

C.—Disinfectants.

Applications.

15,927. W. Smith and J. E. T. Woods. *See* Class XII.

15,972. C. Romen. Process for producing a disinfecting, deodorising, and bleaching soap powder. July 5.

Complete Specifications Accepted.

A.—Foods.

1896.

9908. M. P. Hatschek. Production of compressed baker's yeast. July 14.

16,016. A. Backhaus. An improved treatment of the milk of cows or other domesticated animals to render it suitable as food for infants and children. June 23.

19,675. J. E. Duke. Improvements in the manufacture of liquid extracts of coffee or other substances and in apparatus for the purpose. July 17.

1897.

6915. K. I. O'Doherty. Improved method of preventing the decomposition of animal, vegetable, or other alimentary substances or products. Filed March 16. Date applied for Aug. 27, 1896, being date of application in Queensland.

8586. T. Hyatt. Improvements in the treatment and utilisation of chicory. June 23.

10,873. W. T. Murray. Process for preserving milk. July 21.

B.—Sanitation.

1896.

16,078. J. Royston. An improved mode of treating night-soil. June 23.

1897.

3830. B. Iribarnegaray. An improved process for use in preserving butter and other foodstuffs. July 7.

XIX.—PAPER, PASTEBOARD, Etc.

Applications.

15,601. A. Von Feszty. Improvements relating to the production of paper pulp from peat. June 30.

15,797. K. A. Zschörner. Manufacture of a fibrous material suitable for making paper from peat. July 2.

16,315. H. Schulte. An improved process of and apparatus for incorporating dissolved and suspended substances into paper, cardboard, or pasteboard in long sieve or cylinder machines. Complete Specification. July 9.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

Applications.

14,610. B. Willcox.—From C. F. Boehringer and Soehne, Germany. Improvements in the manufacture or preparation of theobromine. Complete Specification. June 16.

14,611. B. Willcox.—From C. F. Boehringer and Soehne, Germany. Improvements in the manufacture or preparation of iso-eugenol and derivatives thereof. Complete Specification. June 16.

14,612. B. Willcox.—From C. F. Boehringer and Soehne, Germany. A process for the production of trichloropurine. Complete Specification. June 16.

14,613. A. Verley. Improvements in the manufacture and production of perfumes. June 16.

15,093. A. M. Clark.—From V. Kaufmann, Germany. Process for the manufacture of soluble compounds of vegetable globulins. June 23.

Complete Specifications Accepted.

1896.

14,954. C. Goldschmidt. The preparation of a condensation product from vanilline and para-phenetidine. June 23.

18,035. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Improved manufacture of oxycamphor. June 23.

1897.

9701. E. Dolle. Manufacture of improved preparations of iron for medicinal purposes.

12,399. S. Smithson. Machinery or apparatus for treating myrobolams for obtaining extracts therefrom June 23.

XXI.—PHOTOGRAPHY.

Complete Specifications Accepted.

1896.

21,495. W. S. Wain. Improvements in dishes or baths employed in developing and like photographic and chemical operations. July 21.

1897.

12,922. T. C. Porter. Sensitive plate or film, and method of producing, developing, fixing, and toning the same. July 21.

XXII.—EXPLOSIVES, MATCHES, Etc

Applications.

14,690. A. Liedbeck. Improvements in the manufacture of nitrocellulose. Complete Specification. June 17.

14,989. J. T. Maguire —From The Diamond Match Company, United States. Improvements in matches. Complete Specification. June 21.

15,483. B. Baron. Improvements in matches, candles, and the like, and in the method of manufacturing same. June 29.

15,499. H. Maxim. Improvements in the manufacture of explosive compounds. Complete Specification. June 29.

15,693. F. G. du Pont. Improvements in and relating to process and apparatus for treating guncotton and other purposes. Complete Specification. July 1.

16,206. G. de Wolf.—From B. C. Pettingell, Canada. A new explosive. July 8.

16,777. J. Macnab. Improvements in blasting and blasting cartridges. July 15.

Complete Specifications Accepted.

1896.

16,858. H. Maxim. Improvements in the manufacture of pyroxylin smokeless explosives and in apparatus therefor. July 7.

1897.

4672. L. Roux. Improvements in the preparation of explosives. July 7.

10,362. O. F. Carlson. Explosives. July 7.

13,522 W. Majert. Explosive compound. July 7.

14,690. A. Liedbeck. Manufacture of nitrocellulose. July 21.

PATENT UNCLASSIFIABLE.

Application.

15,170. W. P. Thompson.—From M. Rosenbaum, Germany. Improvements in or relating to the production of porous substances. June 24.

Printed and Published by Eyre and Spottiswoode, East Harding Street, London, E.C., for the Society of Chemical Industry.

THE JOURNAL

OF THE

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No. 8.—Vol. XVI.] AUGUST 31, 1897. [Non-Members 30/- per annum; Members 21/- per Set of extra or back numbers; Single Copies (Members only) 2/6.

NOTICES.

Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, part of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows:—

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Meeting held on Friday, April 23rd, 1897.

PROF. C. F. CHANDLER IN THE CHAIR.

A NEW PETROLEUM WELSBACH LAMP.

BY C. K. MUNNS.

THE Washington light, although a new thing in America, is in extensive use in Europe. The two most prominent places illuminated by it are the Boulevard Haussmann in Paris and the Grand Place in Brussels. It is also in use in Spain, Italy, Russia, and Portugal.

The plant consists of a tank or reservoir for holding oil, a copper tube for conducting the oil to the lamp, and the lamp itself. The tank has three openings, one for filling, one for pumping air in upon the oil for the purpose of forcing the oil out of the tank, and a third through which the oil leaves the tank. If desired, a pressure gauge may also be added. The tube, through which the oil is forced, is enlarged inside the tank and contains a filter, consisting simply of waste sufficient only to retard any solid matter in the oil. However, the ordinary kerosene used is comparatively pure, and this filter plays but a small part.

The pressure necessary to force the oil to the lamp at the consumption rate is 40 lb. per square inch, but, by raising it to 55 lb., it in no way affects the burning of the lamp and allows 15 lb. through which the pressure may drop. A few strokes of the pump once a day with this tank is sufficient. The question of constructing tanks, to deliver any quantity of oil for only one pumping, is a question of but little thought. Utilising this drop of pressure, a tank can be constructed which, when once the pressure has been raised to 55 lb., it would not be necessary to again touch until 5 galls. of oil had been consumed. In this lamp this would be after burning 100 hours. For inside lighting, this would mean about 20 days, and for outside lighting about 10 days.

The tube which conducts the oil to the lamp is seamless drawn copper tubing, that used in this case having an outside diameter of $\frac{3}{32}$ in. and an inside diameter of $\frac{1}{32}$ in. This tubing is made in four sizes and can be furnished in lengths of from 50 ft. to 100 ft., annealed so that it may be bent around a small diameter without cracking or breaking. The outside diameter of one size is the same as the inside diameter of the next size smaller. This enables us to make joints by simply introducing one size into the next larger size, about one-half an inch, and soldering the joint; or, for joining two pieces of the same size, by using a short piece of the next size larger, and slipping it over both ends and soldering. Tees and crosses are made in

the same way. In the six months the lamp has been burning in our offices in Wall Street I have not experienced the slightest trouble from clogging of the tube, ordinary kerosene being always used. In piping for a number of lamps to be supplied from one tank, it is only necessary to run one large feeder pipe of a larger size and take branches from it to each lamp.

The lamp consists of only three principal parts, viz., the vaporising tube, the mantle supports, and the body of the lamp. The vaporising tube is the tube which passes up in the centre of the lamp, and is so called because the oil is vaporised in it by the heat of the mantles on either side. The small copper tubing leading from the tank enters the vaporising tube near its lower end at the bottom of the lamp. Referring to the drawings, Figs. 1 and 2 show

Fig. 1.

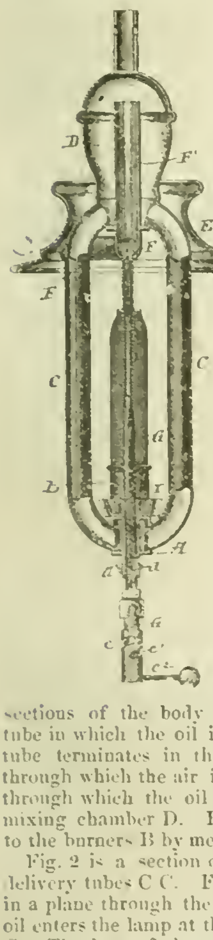
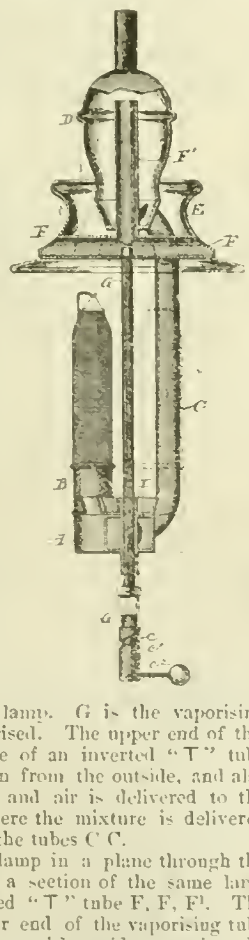


Fig. 2.



sections of the body of the lamp. G is the vaporising tube in which the oil is vaporised. The upper end of this tube terminates in the centre of an inverted "T" tube through which the air is drawn from the outside, and also through which the oil vapour and air is delivered to the mixing chamber D. From there the mixture is delivered to the burners B by means of the tubes C C.

Fig. 2 is a section of the lamp in a plane through the delivery tubes C C. Fig. 3 is a section of the same lamp in a plane through the inverted "T" tube F, F, F¹. The oil enters the lamp at the lower end of the vaporising tube G. The heat of the mantles on either side vaporises the oil in it. The vapour rushes out through a minute hole in the upper end of the tube up into the tube F¹. In doing so it draws air in through the tubes F F, and the air and vapour passing up through F¹ strike against the top of the mixing chamber, and are thoroughly mixed. Thence it is forced from the mixing chamber by more vapour coming in, and is delivered to the burners, where it burns with a blue flame. This flame heats the mantles to incandescence and produces a light of great intensity, yet mellow and pleasing to the eyes. Lamps can be made having one, two, three, or any number of burners. The consumption of oil is very small.

To test consumption of oil a two-burner lamp was cleaned thoroughly, new mantles were put on, and it was lighted. The light was burned 162 hours, and the consumption was 8 galls. of oil, *i.e.*, at the rate of 10·1 hours for ½ gall. of oil. During the entire test the lamp was not touched in any way except to light it and turn it out.

The safety of the lamp is one of its principal features. The body of oil is in the tank, and is thus so far from the lamp, and conducted to it in such small quantities, that it makes explosion impossible. This feature, combined with the portability, the cheapness and beauty of the light, makes it highly commendable for country houses. For outside lighting, the tank may be put in the bottom of the lamp-post, making each one an isolated plant.

In one of my tests, the lamp was placed in such a strong draught that one side of the mantle nearest the source of the draught was cooled and consequently not incandescent, while the rest of that mantle and the entire other mantle were as bright as usual. Upon the draught being stopped, the whole mantle again became bright. This is simply to show what little effect a draught has upon the burning of the lamp.

The mantle supports differ from ones similar to those used on the Welsbach gas lamp. In this lamp, the Washington mantle has at its top, a bail so to speak, and the wire support for it has at its upper end, a hook, upon which the mantle is hung. This makes the support really a point, enabling the mantle to swing in any direction, while in mantles having suspensions similar to the Welsbach, they crack off near the top because they are suspended upon an axis, and can only swing two ways, backward and forward.

Another important point in favour of the hook supports is that the supports, never having to be removed, to change mantles, the new mantles are the same height above the burner, on both burners, and consequently both burn with equal brilliancy. This height being once fixed, for maximum brilliancy, is never changed when necessary to replace old mantles. The Washington mantles are, and of course, have to be, much stronger than mantles for gas burners. The intense heat, baking them so hard that they become like china, in so doing prolongs their life, making them last about 1,000 hours.

In the Washington lamps, it will be noticed that there are no shadows from the side tube, the reason being that the sources of light are so large that the rays overlap, and consequently can produce no shadows. For this reason, together with the steadiness of the light, it is admirably adapted to stereoptican use.

Discussion.

Mr. Munns, in reply to a question from the chair, said that the lamp was stated to be 1,000 candle power. He had compared it with the standard arc lamp, to which it proved equal. He could turn the lamp down until the mantle burned red, but, as the vaporisation depended on the heat from the mantle, he would not advise turning it lower than about half candle-power. It was somewhat affected by cold and outside disturbances, but when burning full a cold draught did not affect the vaporising tube and it burned steadily.

Mr. Lewis T. Wright said that anyone who had had occasion to review fuel gas must have remarked the number of recent lamps to burn alcohol or light petroleum spirit. Every month he had noticed numbers of inventions on the Continent of this form of lamp—a form which, no doubt, would be very useful in places unsupplied with gas or electric light; but he thought that the author's estimate of 1,000 candles somewhat high, and that a strict determination would show that these mantles were giving about the same amount of light as a Welsbach mantle with a gas supply, *viz.*, about 60 to 80 candles per mantle. The author had not informed them as to what class of oil was used, but he presumed that it was gasoline of very light gravity.

Mr. Munns said it was ordinary kerosene of about 150 flash test, such as was in general use.

Mr. Wright, continuing, said that assuming 80 candles to be the light given by one of these mantles and the consumption for 10 hours to be half a gallon, that would give the same duty as oil burning in an ordinary petroleum lamp, which would appear to be rather low, so it was perhaps possible the amount of light emitted was in reality rather more than from the ordinary Welsbach gas burner.

Mr. Munns, in reply, said that a Welsbach lamp burning by the side of this lamp appeared as if about half-turned down, but on turning out the Washington lamp it proved to be giving a full bright light. As regarded the amount of heat generated he had made no tests, but he could say that in the centre of the flame, with the mantles removed, platinum melted, indicating a temperature of about 3,600 degrees.

Journal and Patent* Literature.

I.—PLANT, APPARATUS, AND MACHINERY.

Steam Plant, Leakages in. Engineer, **84**, July 23, 1897, 84.

The importance of leaks in a steam plant is brought out with startling distinctness by the following calculation:—A square inch of area will discharge $\frac{1}{70}$ lb. of steam per second for each pound absolute of pressure. Suppose a station to have 125 lb. gauge, 140 lb. absolute pressure, then a square inch of area would discharge 2 lb. of steam per second, or 7,200 lb. an hour, which, at the standard rate of 30 lb. per hour per horse-power, is 240 horse-power. The inquiry is made as to how many leaks it takes to aggregate a square inch. In a small station, it is thus suggested, the unsuspected leaks may assume a very large proportion of the steam produced.

Formation of a Dangerous Boiler Incrustation by Lime washed out of Concrete. Dingler's Polyt. Jour. **305**, [4], 96.

A bad case of boiler incrustation, due to lime dissolved out of the cement used in a newly constructed water-channel, is reported from the laboratory of Drs. Hundeshagen and Philip (Stuttgart), in a communication to *Giessler's Baumaterialienkunde.* The feed-water, which was drawn from a pure, soft river supply, which had never before occasioned any trouble, was found, after passing through the conduit, to contain 0·06 grm. of calcium hydrate per litre; in consequence, a very hard incrustation formed in the boiler, causing the overheating and burning of the boiler-plates. On analysis, the incrustation was found to have the following composition:—Moisture, 0·30; combined water and trace of organic matter, 21·87; calcium oxide, 67·20 (as hydrate, but partially combined with silica); magnesium oxide, 0·78 (as hydrate, but partially combined with silica); calcium carbonate, 7·50; calcium sulphate, 0·28; alumina, 0·40; ferric oxide, 0·16; soluble silica, 1·40 (combined with calcium and magnesium); and clay, with trace of sand, 0·11 per cent.

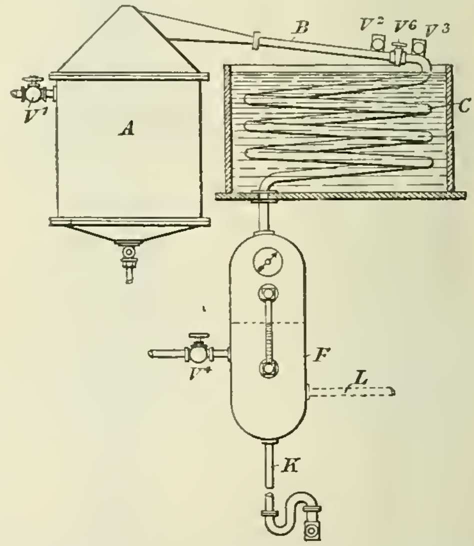
As lime decreases in solubility in proportion as the temperature of the water rises, such an incrustation is deposited most readily on the hottest parts of the boiler plates.—H. H. B. S.

* Any of these specifications may be obtained by post by remitting 8*d*.—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

PATENTS.

Drying, Distilling, Concentrating, and other like Purposes, Impts. in Vacuum Apparatus for, and in the Method of and Means for Obtaining and Maintaining Vacuum therein. J. Longshaw, Burlington Street, Liverpool. Eng. Pat. 9084, April 30, 1896.

The vacuum pan or evaporator A is connected through the pipe B and condenser C with the air-tight receiver F, from which the water can be drawn off through the long dependent pipe K, so as to always maintain a partial vacuum in the evaporator. Instead of, or in conjunction with, the pipe

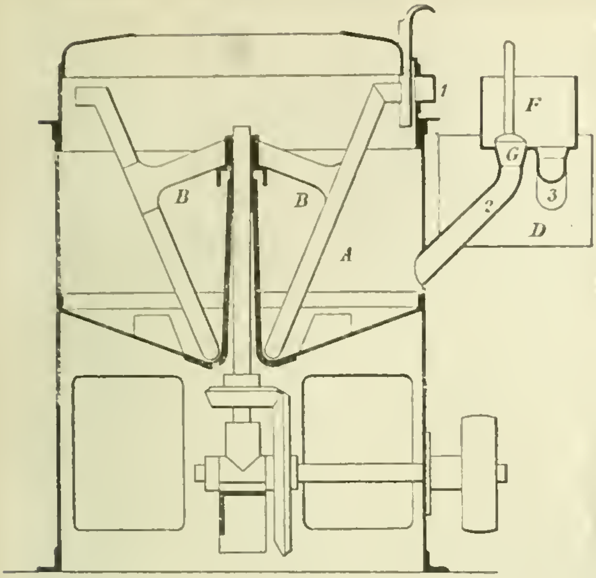


K, a pump, &c. connected to the pipe L may be employed to draw off the water. In starting the apparatus, the main valve V^6 is closed, and the branch valve V^2 opened, steam being admitted through the valve V^1 and blown off through the valve V^2 until all the air is expelled from the evaporator. Next, the valves V^1, V^2 are closed, and the water-supply valve V^4 and branch valve V^3 opened until the receiver F and coil C are filled with water. Finally, the valves V^3, V^4 are closed, and the valve V^6 and the discharge valve of the pipe K or L opened.

The claims, given briefly, are:—(1.) For the apparatus as a whole, and as described and figured. (2.) For dependent pipes, such as K, for the purpose given. (3.) The method of producing a vacuum in vacuum apparatus, by the aid of a water elevator or of a dependent pipe, K, or both.—R. A.

Washing or Leaching Granular Substances and Separating the same from the Liquids used, Impts. in Apparatus for. R. Moodie, Alkham Road, Stoke Newington, London, N. Eng. Pat. 10,784, May 19, 1896.

The liquid and granular materials are placed in proper proportions in the tank A, in which they are mixed by the revolving stirrer B. The arms of this stirrer consist of upwardly and outwardly directed tubes or open channels, through which the mixed materials are lifted and discharged through the passage 1 into a settling tank. From this tank the liquid flows through the receiver F into the tank D. By removing the plug G from the pipe 2 to the pipe 3, the liquid can be passed back through the tank A, to wash out any solid matter remaining therein. An adjustable elbow



pipe is provided to drain the remaining liquid from the settling tank into the receiver F at the finish of the operation. —R. A.

Filtering Apparatus, Impts. in. J. A. Orme, Egremont, County of Chester. Eng. Pat. 11,080, May 21, 1896.

The filtering medium is placed between perforated plates in a vertical cylinder. The feed-water enters at the bottom of the cylinder and, flowing upwards through the filtering media, escapes through a pipe attached to the cylinder cover. Soluble chemicals may also be added to the material to purify the water in transit through the apparatus. —O. H.

Evaporating Apparatus, Improved. R. H. F. Finlay, Belfast. Eng. Pat. 16,552, July 27, 1896.

A chamber is attached to the bottom of the vacuum-pan, so that, during evaporation, the solids deposited may be from time to time removed without stopping the process. The solid deposits are caught on a perforated plate in the chamber, while the liquor passing through may be aspirated again into the vacuum-pan. Suitable valves are fitted to enable these operations to be carried on, while a cleaning door is fitted in the chamber above the perforated plate for removal of the sediment.—O. H.

A Still or Tank for the Distillation, Boiling, and Holding of Acids, Alkali, and other Corrosive Liquids. D. Bray, Cinderford, Gloucester. Eng. Pat. 16,877, July 30, 1896.

The base, sides, and cover of a still or cistern are formed of single blocks of a selected stone, such as the grey "Forest of Dean" or "Pennant" stone, squared and dressed, and having grooves made in their touching surfaces to receive pieces of solid rubber as packing, forming tight joints. The bottom and sides are clamped together, the iron bands being tightened by bolts and nuts or the like.—E. S.

Decanting Apparatus, Impts. in. W. L. Wise, London. From Messrs. Solvay and Co., Brussels, Belgium. Eng. Pat. 17,980, Aug. 13, 1896.

The apparatus consists of a box or casing, funnel-shaped at the bottom, with a valved aperture, and an overflow at the top. The turbid liquid flows into a compartment formed on one side by a solid plate reaching nearly to the bottom, so that it may not overflow before being cleared, by passage upwards through a series of inclined parallel plates arranged to divide the interior of the box into many equally spaced and inclined compartments. Each of these plates is provided on its under side with a series of half partitions or vanes, approximately at right angles to the plate, forming inverted angular troughs, which impede the upward flow of the liquid, and give time for deposition to take place on the inclined ledges which these vanes constitute. Thus, the deposit, as it forms, settles continually to the bottom, whilst the liquid, cleared as it rises, flows off at the top. It is stated that "the arrangement is such that the decanting apparatus comprises a very large number of small decanting chambers or passages, each of which acts as an approximately horizontal or slightly inclined tube of rectangular section." The apparatus is shown in two cross sections taken at right angles to one another, one of the sections having an upright, and the other an inclined form. —E. S.

Condenser for Steam-heated and Similar Pans or Vessels, chiefly applicable to those used in treating Aromatic Substances; A New or Improved. J. F. Henderson, Manchester. Eng. Pat. 7413, March 23, 1897.

The patentee claims the combination of a hollow dome provided with a cover, in and through which a current of cold water circulates, in pans or vessels for extracting the aromatic principles of hops, &c. The whole arrangement forms a condenser, and it is fitted with an inlet pipe near its lowest side and circumference, with the delivery end tangentially disposed to the circumference. It is also fitted with an outlet or discharge pipe in its uppermost and central part.—O. H.

II.—FUEL, GAS, AND LIGHT.

Illuminating Gas: Papers read at the Annual Congress of the Société Technique du Gaz en France. J. Gas Lighting, 1897, **70**, 109—110.

The grate invented by J. Kudliez, of Prague, for the combustion of breeze, coal dust, &c., was described by M. Jouanne. It is constructed of cast-iron plates, and has conical perforations, 0·15 in. wide at top and 0·7 in. at bottom, numbering 128 per square foot. Below the plates is an underblast chamber into which a steam injector forces air at a pressure of 1 inch of water, and this blast of air and steam fans the fuel on the plates into vigorous combustion. With this grate, 1 lb. of breeze has been found to evaporate 7 to 7·4 lb. of water; whilst in another experiment 70 per cent. of the available calorific power of the fuel was utilised. The application of the grate has hitherto been chiefly for boiler furnaces, but it is adapted for any high-temperature furnace, and is to be tried shortly for heating a setting of gas retorts.

A hinged scoop for charging gas retorts was described by M. Lautain. It is 16 ins. wide and 6 ins. deep and contains the whole charge. It is built up of two quadrants of sheet steel, which are drawn apart by means of lever handles when the scoop is in the retort. The coal then drops through the longitudinal opening in the scoop on to the bottom of the retort. The scoop is used in conjunction with a stirrup support, which travels on an overhead rail in front of the bench of retorts. Three men are necessary for its manipulation, and 30 to 40 seconds are occupied in charging each retort.

A scheme for converting transverse to longitudinal benches of retorts for the better accommodation of stoking machinery in the retort house was given by M. Leclaire. A feature of the scheme is the erection of settings of 12 retorts. Six of the retorts are connected in pairs, with one ascension pipe per pair. Each setting is 12 ft. 4 ins. wide, and is capable of carbonising 11·8 tons of coal per 24 hours.—J. A. B.

Gas Industry, Notes on the By-Products of the. H. Bunte. J. für Gasbeleucht. **40**, 1897, 405—407.

The relation subsisting between the staple product and the by products of the gas industry has altered greatly in recent years. A glance at the present and probable future position of the by-products—coke, tar, ammoniacal liquor, and cyanogen—is not inopportune.

The importance of coke as a household fuel is gaining recognition, but, except in gas- and waterworks, coke and breeze are very seldom used as boiler fuels. Yet

their use in large towns would afford a simple solution of the problem of smoke-prevention. A grate constructed to burn coal requires considerable alteration before it becomes suitable for burning coke. The heat of combustion of coke is the most important factor in computing its value as a fuel; but very scanty information exists on the composition and calorific value of coke. The results given in the annexed table refer to average samples of coke from the gasworks at Berlin, Breslau, Munich, and Hamburg. The air-dried coke has an average value of 6,959 calories per kilo., with a maximum deviation of about ± 8·5 per cent. Though this calorific value is less than that of good coal, yet uniform and smokeless combustion of coke may be attained in suitable furnaces, and a higher duty than from other fuels secured.

Chemical Composition and Calorific Value of some Gas Cokes.

Source of the Coals from which the Coke was made.		Composition of the Air-dried Coke.						Combustible Substance.	Composition of the Substance freed from Water and Ash.			
District.	Names of Pits.	Carbon.	Hydrogen.	Oxygen and Nitrogen.	Sulphur.	Water.	Ash.		Carbon.	Hydrogen.	Oxygen and Nitrogen.	Sulphur.
The Ruhr	Consolidation	85·18	0·70	4·04	0·87	1·79	7·42	90·79	93·82	0·77	4·45	0·96
"	Rhine-Elbe and Alma	85·30	0·81	4·80	0·88	1·71	6·50	91·79	92·93	0·88	5·23	0·96
"	Ewald	80·68	0·90	3·74	1·17	2·33	11·18	86·49	93·28	1·04	4·32	1·36
"	Bonifacius	82·03	1·07	3·61	1·02	1·53	10·74	87·73	93·50	1·22	4·12	1·16
The Saar	Camphausen	82·91	1·00	2·60	1·43	1·79	10·27	87·94	94·28	1·14	2·96	1·62
"	Heinitz	88·08	0·78	2·85	0·81	0·96	6·52	92·52	95·20	0·84	3·08	0·88
Upper Silesia	Deutschland	88·24	0·75	3·13	0·96	3·20	3·72	93·08	94·80	0·81	3·36	1·03
" "	Konigin Louise	86·35	0·54	2·01	0·96	3·73	6·41	89·86	96·09	0·60	2·23	1·08
Lower Silesia	United Glückhilf	82·38	0·92	2·34	1·24	1·52	11·60	86·88	94·82	1·06	2·69	1·43
Bohemia	Sulkov	84·60	0·53	2·96	0·90	1·39	9·62	88·99	95 07	0·60	3·33	1·00
"	Pankraz	80·73	0·83	3·84	0·81	2·31	11·48	86·21	93·64	0·96	4·46	0·94
England	(Unknown)	80·71	0·76	4·41	1·10	1·69	11·33	86·98	92·79	0·87	5·07	1 27

Source of the Coals from which the Coke was made.		Carbonisation.			Calorific Value.		Calorific Value of the Combustible Substance.	
District.	Names of Pits.	Yield of Coke.	Fixed Carbon.	Volatile Matter.	By Dulong's Method.	By the Calorimeter.	By Dulong's Method.	By the Calorimeter.
The Ruhr	Consolidation	98·00	90·58	0·21	6,967	7,057	7,686	7,785
"	Rhine-Elbe and Alma	96·25	89·75	2·04	6,982	7,071	7,617	7,716
"	Ewald	95·16	83·98	2·51	6,675	6,716	7,734	7,781
"	Bonifacius	95·30	84·56	3·17	6,841	6,851	7,808	7,819
The Saar	Camphausen	95·41	85·14	2·80	6,935	6,936	7,899	7,900
"	Heinitz	98·30	91·78	0·74	7,271	7,268	7,865	7,862
Upper Silesia	Deutschland	94·34	90·62	2·46	7,257	7,265	7,817	7,826
" "	Konigin Louise	94·34	87·93	1·93	7,080	7,111	7,903	7,938
Lower Silesia	United Glückhilf	97·14	85·54	1·34	6,877	6,960	7,926	8,022
Bohemia	Sulkov	96·85	87·23	1·76	6,913	6,991	7,778	7,865
"	Pankraz	95·22	83·74	2·47	6,647	6,702	7,726	7,790
England	(Unknown)	95·40	84·07	2·91	6,616	6,695	7,618	7,708

Tar and ammonia have suffered depression from the output of coke-ovens. Ammonium sulphate has fallen in value continuously in recent years, owing to over-production at gasworks, coke-ovens, and the Scotch shale distilleries, and it can in future only yield a very moderate return. The chief use of ammonia (about 90 per cent.) is as sulphate, for manurial purposes, and this use is now threatened by the appearance of another competitor in "nitragin," a preparation for inoculating soil, so as to aid the assimilation of atmospheric nitrogen by plants. Experiments with nitragin have not, so far, been very successful, and its competition has not yet been keenly felt (see this Journal, 1896, 767). Ammonia in other forms than the sulphate yields better returns, but the production of other ammonium salts is too specialised an industry for general adoption in gasworks. In some medium-sized works, a concentrated gas-liquor containing about 15 per cent. of ammonia, is now made in place of ammonium sulphate (which contains 25 per cent. of ammonia). Gas-liquor is now largely used for the production of liquefied pure ammonia for refrigerating purposes.

Cyanogen has recently been the most valuable constituent of the spent oxide from the purifiers, but the production of cyanide has now greatly increased, and equals, if it does not exceed the demand. Hence the value of spent oxide rich in cyanogen has dropped.—J. A. B.

Naphthalene in Modern Gas Manufacture, and the Carburetting of Illuminating Gas. W. Young and T. Glover. J. Gas Lighting, 1897, **70**, 16—19.

Modern gasworks practice aims at securing the highest illuminating value, expressed in sperm candles, from the coal upon the smallest area. Carbonisation at high heats, and condensation wherein dense liquid hydrocarbons are removed before they take into solution gaseous hydrocarbons, have been found to achieve that aim, but at the sacrifice of high illuminating power. Moreover, this system of carbonising and condensing favours the production of naphthalene and its deposition as a solid in the gas mains. The gas so produced is free from hydrocarbons of similar vapour tension and boiling point to naphthalene, though they occur in gas made at lower temperatures and in cannel gas. When they occur they are deposited as liquids whenever naphthalene is deposited, and take it into solution. But it is unscientific and wasteful to allow the products from common coal and cannel to commingle in the hydraulic main, as the carburetting products of the cannel are thus largely spent in absorbing the naphthalene from the common coal. The liquid condensation products of coal-gas absorb its naphthalene on prolonged contact, but at the same time absorb other illuminants and impoverish the gas. The ordinary enriching

agents have higher vapour tensions than naphthalene, and do not hinder its deposition as a solid.

Deposition of naphthalene might be avoided in three ways, *viz.*: (1.) By carbonising only a portion of the coal as at present, and another portion or some cannel, at lower heats, and mingling the gases, after condensation apart. The low-temperature products would then absorb the naphthalene left in the gas made at high temperatures. Or carbonisation might be pursued at low heats only, and the rich gas produced used to enrich carburetted water-gas made from the coke and liquid products of the carbonising process. (2.) By scrubbing gas after condensation, with a hydrocarbon liquid which absorbs naphthalene, and preferably also by vaporisation, which enriches the gas. The naphthalene would be subsequently removed from the absorbent by cooling and pressing or by distillation. (3.) By carburetting gas with hydrocarbons having the same vapour tension as naphthalene and depositing along with it as liquids which dissolve it. The volume of such hydrocarbons must, however, be restricted, as they carry down also other carburetting vapours and thus impoverish the gas.

—J. A. B.

Carburine [Petroleum Spirit] as an Enricher of Coal-Gas; On Three Years' Use of. J. Davis. J. Gas Lighting, 1897, **70**, 19—23.

Carburine, sp. gr. 0·680 [or petroleum spirit], has since the autumn of 1893 been used by means of the Maxim-Clark carburetter for raising the illuminating power of coal-gas at the Gravesend works to 16 candles. Prior to that time "North Wales Curley" cannel, costing 19*s*. 4*d*. per ton delivered, had been used, and an enrichment of 1½ candles then cost 2·24*d*. per 1,000 cb. ft. of gas. Carburine cost 8½*d*. per gallon delivered; and the same degree of enrichment cost 2·07*d*. during a year in which carburine alone was used as the enricher, for nine months. One ton of the above cannel appeared to be equivalent to 63 gals. of carburine, whereas Field takes one ton of "Lesmahagow" cannel as equivalent to 50 gals. of carburine. Coal-gas tested at the outlet of the exhauster was found to range from 12 to 17 candle-power within one hour. Carburine enrichment was most economically carried out when the gas in one holder was raised from 20 to 22 candle-power, and the requisite quantity of this rich gas was worked out with the unenriched gas from other holders as required. Sugg's "Illuminating Power Meter" indicated little or no loss in illuminating power of the carburine-enriched gas at points a mile distant from the works. Carburine enrichment was found to hinder deposition of naphthalene.

Experiments in which one fluid ounce of carburine was taken up by 10 cb. ft. of gas showed that the volume of gas was raised to 10·15 cb. ft., and its candle power from 14·88 to 19·86. One gallon of the carburine thus represents 24 cb. ft. of 352-candle gas.—J. A. B.

Calcium Carbide Industry: Its Economic Importance to Germany. A. Frank. Zeits. f. Elektrochem. 1897, **3**, 427.

In spite of the recent "acetylene panic," the calcium carbide industry merits careful attention, especially in countries which are not able to supply their own sources of light. Germany produces about 750,000,000 cb. m. of coal-gas, mostly from her own coal. In addition to this she consumes over 8,000,000 hectolitres of petroleum, for which she is dependent upon foreign countries. Not only is Germany unable to control the price of this, but within a few years she may have to submit to a sudden and enormous increase in the price if the Russian and American petroleum-producers succeed in creating the universal monopoly for which they are striving. Six litres of petroleum give about the same amount of light as 15 cb. m. of coal-gas in ordinary burners; so that an import of 8,000,000 hectolitres of petroleum corresponds in illuminating value to 2,000,000,000 cb. m. of coal-gas. This indicates a large dependence upon foreign countries, to which must be added imports of other lamp-oils, tallow, &c. Altogether it is computed that Germany pays 100,000,000 mks. per annum to foreign countries for sources of light.

In countries which possess large sources of water-supply, such as Switzerland and Norway, large factories for the production of calcium carbide are already being erected. Germany is not so well supplied with water-power; but in her peat-moors she has an almost inexhaustible source of power. The author draws special attention to the great peat-moor in East Friesland, of which scarcely any use has been made, although the Prussian Government has provided facilities for opening it up by constructing a canal. Attempts to export the turf have not proved remunerative; for, on account of its relatively low heating power, it cannot compete with the neighbouring Westphalian coal. But it offers excellent conditions for the fabrication of carbide on the spot, especially as the other materials required can be obtained within a reasonable distance. 30 tons of peat will supply the electrical energy required for the production of 1 ton of calcium carbide.—D. E. J.

Cerium, The Purification of. Wyrouboff and Verneuil. Comptes Rend. **124**, 1230.

See under XX., page 696.

Cerium Oxide, Preparation of. H. Moissan. Comptes Rend. **124**, 1233.

See under XX., page 696.

Barium Sulphocyanide from Spent [Gasworks] Oxide, Manufacture of. V. Holbling. Zeits. angew. Chem. 1897, [10], 297.

See under VII., page 680.

PATENTS.

Coking Ovens, Impts. in. B. Grau, Kratzwick. b. Stettin, Germany. Eng. Pat. 17,198, Aug. 4, 1896.

To enable the linings or inner walls of the heating flues to be made of less thickness than usual and thereby obtain a more intense heating effect, they are formed of bricks of ⊏- or ∟-section.—R. S.

Coke from Anthracite and Similar Coal, An Improved Process for Making. L. J. Davies, Cardiff. Eng. Pat. 12,096, May 15, 1897.

Anthracite or similar coal is powdered, then mixed with either about 20 per cent. of pitch, or 25 per cent. of tar, or with a mixture of the two. The whole is then coked as usual.—R. S.

Siemens' Regenerative Furnaces, Impts. in and connected with. F. Svoboda, Altsohl, Hungary. Eng. Pat. 10,763, May 19, 1896.

The encircling or outer plates of the furnace are made sloping instead of vertical, to effect economy in the material used in their construction, to assist in cooling, and for greater durability. The air- and gas-canals or pipe conduits are divided into two or more compartments between the reversing device and the heat accumulators, and the rate of inflow or outflow in these compartments is regulated by means of dampers, which are operated automatically by the reversing apparatus.—R. S.

Gas Generators, Impts. in and relating to. T. Hennessy, Kansas City, U.S.A. Eng. Pat. 28,718, Dec. 15, 1896.

The plant consists of a superheater and two generators working alternately or concurrently. The fuel is fed to the latter through hoppers situated on one side above the generators, and air is blown in to drive the products of combustion into the superheater, where they ascend through the system of checker bricks, with which the latter is filled, and are ignited at the top for the purpose of heating a secondary current of air passing between the walls of a superimposed hood, and thence, *viâ* a second annular heating chamber at the top of the superheater, into a pipe debouching into the last-named at a lower level, and there igniting the carbon monoxide from the generators. In this way the temperature of the superheater is raised to about 2,190° F., whereupon the mouth of the superheater is

closed, and steam is passed through the fuel in the generator, the water-gas thereby produced being delivered to the upper part of the superheater. Here it is carburetted by means of gas-naphtha or other suitable oil vaporised by passing through a heating coil in the head of the superheater, the operation being facilitated by diverting a jet of the water-gas into the oil pipe, the high temperature of the gas (about 2,000° F.) rendering it a more efficient agent for this purpose than the steam usually employed. The carburetted gas is thoroughly fixed during its passage downwards through the superheater, and is conducted through a water-seal and discharge pipe at the bottom of the apparatus, to the condenser.

The apparatus may be worked either with an upward or downward draught through the generators, and can be modified for working with either an air blast or an exhauster.

The generator grates consist of piping, through which is passed a current of water, which may then be used for heating adjoining compartments.—C. S.

Water-Gas, Improved Method and Apparatus for Removing Gaseous Iron Compounds from. H. Strache, Vienna. Eng. Pat. 16,984, July 31, 1896.

In this apparatus the gas, entering at the bottom, passes upwards through a lead-lined scrubber containing pumice moistened with concentrated sulphuric acid, which removes the objectionable iron compounds and dries the gas. The acid is sprayed by being dropped through a pin-valve on to a curved leaden disc situated above the pumice, and since its capacity for freeing the gas from iron is very great and it leaves the apparatus in a condition of 50 per cent. dilution, only 16 grms. are required per cubic metre of gas, or 0·5 grm. per second, when the apparatus is working at the rate of 100 cubic metres of gas per hour. —C. S.

Carbon Dioxide and Hydrogen, Improved Means for Producing. B. T. L. Thomson, London. Eng. Pat. 17,778, Aug. 11, 1896.

The inventor proposes to modify the treatment of these gases, generated in accordance with his patent (Eng. Pat. 6835 of 1896), in that they may be separated by dissolving the carbon dioxide in water under pressure, the residual hydrogen being freed from carbon monoxide by passing it through cuprous chloride, the dioxide being subsequently liberated by reducing the pressure. It is then stored for use. An alternative method is to absorb the carbon dioxide by means of an alkali carbonate in solution. An excessive current of steam in the carbon retorts is desirable, and the gases should be passed through another retort containing some inert material, such as firebrick, to facilitate the oxidation of the carbon monoxide.—C. S.

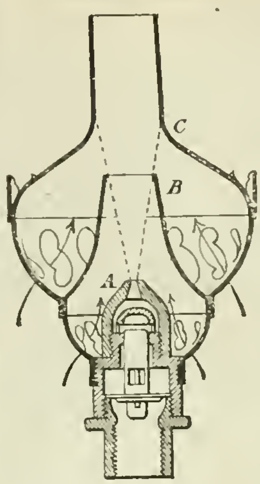
Luminous Flame, Process for making Spirit burn with a. F. W. H. Hempel, Berlin. Eng. Pat. 6463, March 11, 1897.

The spirit burning with a non-luminous flame is mixed with from one-sixth to one-half its weight of a volatile hydrocarbon; *e.g.*, turpentine oil previously enriched with carbon by rapid distillation along with naphthalene or other solid hydrocarbon. An addition of glycerin, in the proportion of 5 to 10 per cent. of the turpentine oil employed, will "soften" the liquid and enable the wick of the lamp to be lighted with ease, even after having been out of use for some time.

In distilling the turpentine oil, the naphthalene ($\frac{1}{3}$rd to $\frac{1}{6}$th part) is first dissolved by heat and the whole then heated rapidly (to ensure the naphthalene vapour being carried over with that of the turpentine) until merely a slight brown residue is left.—C. S.

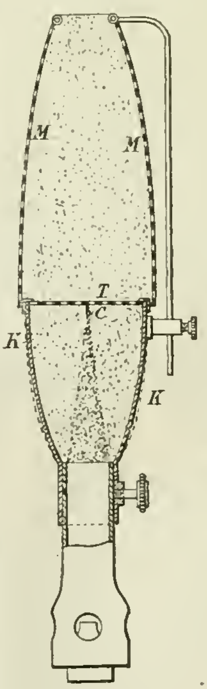
[*Incandescent Lighting.*] *Bunsen Burners, Impts. in.* A. Lecomte and L. Loeser, Paris. Eng. Pat. 8385, April 1, 1897.

The improvements relate to Bunsen burners working with gas at very low pressures (8 to 10 mm. of water), capable of being used under the ordinary incandescence burner galleries. The claim is for "the combination of an ejector, A, having a single central hole of conical form, forming an angle of about 30°, with two converging cones, B and C, arranged one above the other and having different angles, the lower cone, B, having an angle about equal to the angle at which the gas expands, and the upper cone, C, having an angle considerably greater—preferably about seven to nine times greater—than the angle of the lower cone." The burner shown in the illustration is slipped up into the burner-top in the usual way.—H. B.



Gas, Improved Method and Composition for making. A. A. Stephenson, Sydney, N.S.W. Eng. Pat. 10,371, April 26, 1897.

A composition is patented for producing gas, by passing air through it. It consists of camphorated chalk, resin, salt, and shale oil. This material is further treated with gasoline, ether, petroleum, and carbon bisulphide. The method is also patented.—C. S.



Incandescent Gas Lighting, An Impt. in Burners for. L. Denayrouze, Neuilly, France. Eng. Pat. 21,371, Sept. 26, 1896.

This invention can be understood from the illustration, which shows a mantle, M, suspended over the improved burner.

The claim is: "In combination with a Bunsen burner for incandescence, a fixed or adjustable shell, K, enclosing and approximately fitting the ovoid form assumed by the gas and air issuing from the burner tube, and terminating with a pervious diaphragm, T, on a level with what would be the apex, C, of the blue cone observed where there is a flame without an enclosing shell." —H. B.

Burning Liquid Fuel, Improved Method and Apparatus for. F. Altmann, Berlin, Germany. Eng. Pat. 29,258, Dec. 28, 1896.

This relates to the production from liquid fuel of a hot smokeless blue flame, suitable for incandescent lighting, cooking, heating, soldering, and other purposes, including the ignition of the combustible charges in gas and like engines. The process consists in simultaneously evaporating water and vaporising the fuel, and then mixing and burning the vapours produced. Two forms of lamp are illustrated, in each of which there are two distinct compartments, one for fuel, the other for water, and a small lamp for vaporising the fuel and water. The mixed vapours pass to an ordinary burner for ignition.—R. S.

Burner [Lamps], Improved Circular, for use in connection with Liquid Hydrocarbons having different Carbon Contents. F. W. H. Hempel, Berlin, Germany. Eng. Pat. 6462, March 11, 1897.

To admit of the regulation of the supply of air to the interior and exterior of circular burners of the kind indicated, according to the quality of liquid hydrocarbon used, the air is admitted to the slot leading to the interior of the burner through a perforated diaphragm, the space enclosed thereby being entirely or almost entirely cut off from any connection with the outside of the flame by a crown piece. Above the crown piece are two concentric perforated short cylinders, one fixed, the other rotatable, for admitting air to the outside of the flame, and the supply of air thereto is regulated by causing the holes in the two cylinders to coincide more or less.—R. S.

Liquid Fuel Burners, Impts. in. P. M. Justice, Chancery Lane, London. From The Société Anonyme du Générateur du Temple, Paris, France. Eng. Pat. 11,571, May 10, 1897.

Has reference more particularly to burners for firing steam boilers. A single burner may be used, or a number of them may be supported parallel to one another around a hollow cylindrical drum. Both arrangements are illustrated. Each burner is carried by two supports. Through the rear one is admitted the liquid fuel, which passes to the forward end through an adjustable hollow tube or needle; and through the forward one is admitted the compressed air or steam, which is delivered around the outlet of the liquid fuel, this annular outlet being made larger or smaller by means of the adjustable needle. By removing a plug in the rear, the needle may be easily removed for cleaning, &c. When several burners are used, the hollow supporting drum is provided with means for heating the liquid fuel before it enters the nozzles.—R. S.

Acetylene, Impts. in Apparatus for the Production of. G. F. Redfern, London. From "La Compagnie Continentale d'Eclairage par le Gaz Acetylène et ses Applications Industrielles," Paris. Eng. Pat. 15,061, July 7, 1896.

A generator containing several compartments for carbide is supplied with water from a tank above a bell gas-holder into which the gas generated passes. The rise and fall of the bell operates a valve on the water-supply pipe. The gas passing from the holder is purified by spongy material impregnated with suitable salts.—J. A. B.

Acetylene Gas, Impts. in and connected with Apparatus for Generating and Storing. L. T. F. Gibbon, South Kensington. Eng. Pat. 17,038, July 31, 1896.

The fall of a gas-holder bell opens a cock which allows a small vessel of water to be discharged into a carbide container. The rise of the gas-holder due to the gas thus generated allows the vessel to be refilled from an overhead water-tank. Or carbide agglomerated by means of paraffin, &c., may be discharged intermittently into water. Or, either the carbide or the reacting water may be mixed with suitable inert material to moderate the action.—J. A. B.

Acetylene, Impts. in Process and Apparatus for Generating and Liquefying. A. C. Fraser, New York. Eng. Pat. 17,678, Aug. 10, 1896. (Under Internat. Convention.)

Acetylene is generated in a close vessel of great strength, and cooled. It is then led to a cooler, terminating in a water separator; thence it passes to refrigerators and to a compressing pump, from which it goes through a cooling coil, and emerges in the liquid state. The gas is generated at a comparatively low pressure, and the final liquefaction is preferably effected at a very low temperature, and consequently low final pressure. Suitable apparatus for generating, cooling, refrigerating, and compressing the gas is described.—J. A. B.

Acetylene Gas, Impts. in Apparatus for Generating, Storing, and Delivering. E. Chesnay and L. Pillion, both of Dijon, France. Eng. Pat. 18,992, Aug. 27, 1896. (Under Internat. Convention.)

The counter-weight of a gas-holder bell, is a water reservoir communicating with the lower half of a generator, the upper part of which contains carbide on a suitable support.
—J. A. B.

Acetylene Gas, Impts. in or appertaining to the Production of, and Apparatus therefor. W. P. Thompson, Liverpool. From D. H. Chivert, of Neuilly-sur-Seine, France. Eng. Pat. 19,059, Aug. 28, 1896.

The bell of a gas-holder floats in an annular space surrounding but independent of a water reservoir. A hopper containing granulated carbide is placed within or above the bell, and the fall of the latter opens the feed-valve of the hopper and allows carbide to fall into the water in the reservoir, and generate acetylene.—J. A. B.

Acetylene Gas, Improved Apparatus for the Production and Storage of. E. A. Morton-Brown and F. Maundrell, both of Woodstock, Canada. Eng. Pat. 22,628, Oct. 12, 1896.

The carbide is contained in a perforated cylinder automatically revolved in the generator. The tank of the gas-holder is partially occupied by a condenser, through which the gas passes, for the abstraction of moisture. The rise and fall of the bell of the holder automatically control the admission of water to the generator. The gas-holder has a closed cover.—J. A. B.

Acetylene Gas, Impts. in Apparatus for Producing. G. Voigt, Berlin. Eng. Pat. 19,126, Aug. 29, 1896.

The rise and fall of the gasometer bell actuates, through suitable gearing, the supply of water from a reservoir to carbide receptacles. The water-feed pipes and gas-exit pipes are attached to a plate which forms the cover of the carbide receptacles. A worm in the reservoir is employed to cool the evolved gas.—J. A. B.

Acetylene Gas, Improved Apparatus for the Production and Storage of. J. G. Kerr and C. Fry, Niagara Falls, Canada. Eng. Pat. 8989, April 8, 1897.

The apparatus consists of a generating chamber containing two or more receptacles for carbide, to which a flow of water, regulated by a trap siphon which rises and falls with the gas-holder bell, has access. The receptacles are on a revolvable base-plate, so that they may be brought beneath the water supply and a recharging lock in turn. An electric alarm indicates exhaustion of the gas-holder.
—J. A. B.

Acetylene Gas, Impts. in or connected with Apparatus for the Production and Consumption of. W. P. Thompson, Liverpool. From "The Deutsche Acetylengas-Gesellschaft mit beschränkter Haftung," Berlin. Eng. Pat. 10,249, April 24, 1897.

The generator consists of a water-chamber above a carbide receptacle. Water passes to the carbide through a knee-pipe furnished with a diaphragm valve, which is closed automatically by the pressure of gas in the carbide receptacle. Condensation of moisture in the gas-exit pipe is prevented by carrying the latter entirely outside the water chamber. A safety device for removing the residual gas from lamps, &c., when they are extinguished, is provided.—J. A. B.

Acetylene Gas, A New or Improved Automatic Apparatus for the Production of. R. Quatannens-Moens, Dixmude, and E. Carreér-Dilger, Bruges, Belgium. Eng. Pat. 12,556, May 20, 1897.

This apparatus consists of a generator surrounded by a cooling reservoir. Water is supplied to the carbide in a suspended basket in the generator through a pipe entering the base of the latter from an elevated tank in which a fixed level is maintained. The gas passes from the generator, through a washer and purifier, to the burners, or to a reserve gas holder.—J. A. B.

Acetylene Gas Generators, Impts. in. F. A. Mitchell, Wilmington, Delaware, U.S.A. Eng. Pat. 13,496, June 1, 1897.

A GASOMETER moves in a water reservoir from which an adjacent generator is supplied. The water reservoir contains also a fixed overflow gas-holder, to which the stream of gas is diverted when the pressure in the gasometer becomes excessive. The supply of water to the generator is governed by a valve actuated by the gasometer. A pump for removing gas from the generator prior to recharging, is attached.—J. A. B.

Electric Incandescent Lamps, Impts. in Filaments [Borates] of. F. E. W. Bowen, Ealing. Eng. Pat. 16,435, July 24, 1896.

A FILAMENT is claimed, in the body of which is incorporated metallic borates of a refractory character. Cellulose, or cellulose filament, is soaked in solutions of the nitrate or other soluble salt of the metal and of boric acid, and caused to react to produce the borate (Eng. Pat. 3213 of 1896; referred to, this Journal, 1897, 149).—J. C. R.

Incandescence Vapour Lamps, Impts. in. P. D. de la Grée, Paris. Eng. Pat. 17,131, Aug. 1, 1896. (Under Internat. Convention.)

THE improved lamp comprises—(1) Means for giving pressure to the petroleum, either by injecting air into the reservoir or by means of a descending piston actuated by a spring within the reservoir; (2) an expansion device, on, say, the Carcel or Toecheboeuf system for regulating the pressure of the petroleum which arrives at the burner; (3) the burner proper; and (4) the lighting parts, consisting of a small flange or sleeve in which alcohol can be employed for the preliminary heating, the alcohol being contained in a reservoir located in the lamp body, whence it is raised by a small pump. The burner consists of a bent tube rising upwards from the expansion device and provided internally with a metal spiral, where the petroleum is vaporised; a "butterfly sieve" to filter the petroleum vapours; a casing surrounding the vaporiser and sieve, and terminated at its upper end by the usual gauze, where the vapours burn; and a heat-conducting rod, placed in the flame and supplying the heat to the vaporiser. The various combinations are claimed.—H. B.

Incandescence Burners for Vapour Lamps, Impts. in. P. D. de la Grée, Paris. Eng. Pat. 18,391, Aug. 19, 1896. (Under Internat. Convention.)

IN the improved burner, the socket from which the usual mantle support rises, consists of a metal rod, which at its lower extremity is in contact with the wick. The claim is for "an improved incandescent burner for ordinary petroleum lamps, in which burner the heat disengaged beneath the mantle is conveyed to the upper extremity of the wick of the lamp by the conductive action of a metal rod of appropriate shape placed in the flame; while the air required for producing a carburetted gas mixture arrives at the sides of the burner and is mixed with the vapours produced at the extremity of the wick, so that this mixture may then be ignited above a grate terminating the burner." —H. B.

Incandescent Mantles or Caps for Gas and other Burners, A New or Improved Method of Manufacturing. R. Moll and A. Palmer, Germany. Eng. Pat. 26,784, Nov. 25, 1896.

IT is proposed to manufacture mantles by impregnation in the usual way, as suggested in the following claim:—"A new or improved method of manufacturing incandescent mantles, caps, or like appliances, marked by the use of salts having sodium for their base and silicates or borates, and also of a smaller quantity than hitherto of salts based on thorium, cerium being entirely or partly dispensed with, substantially as set forth."—H. B.

[Incandescence Petroleum Lamp.] Vapour Burners for Heating Purposes and for the Production of Light with a Mantle, and Means of Lighting them; Impts. in and connected with. J. T. Armstrong, London. Eng. Pat. 11,433, May 7, 1897.

AN electrical current passed through a resistance suitably arranged round the gasifying portion of the petroleum supply-pipe, generates heat sufficient for the initial supply of vapour. An electrical switch, actuated by a rotatable part of the lamp casing, sends a current through other [platinum] resistances which ignite the vapour when required. The vapour mixes with air, drawn through inlet holes, and the mixture divides into two streams, the one passing to the Bunsen burner and mantle of the usual types at the upper extremity of the lamp, while the other burns in a hollow neck surrounding the first air-mixing chamber, supplying to the vaporiser sufficient heat by conduction, to maintain the supply of petroleum vapour. The claims are for the burner, for the means of electrically generating the initial charge of vapour and igniting the same, and for the various combinations of parts. The fourth claim is for the gasifier enclosed by the conducting part "so that the efficiency of such conductor will be decreased as it gets hotter, owing to its expansion, whereby the air space between it and the retort (gasifier) will be increased, and conversely."—H. B.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Petroleum Tanks, Safety of. Engineer, **84**, July 23, 1897, 84.

THE importation of petroleum into Trieste in tank steamers has given rise to an Austrian governmental inquiry as to the vapour of petroleum hanging about the tanks, and the dangers arising from the explosive mixtures to which it gives rise. The result is that a method has been adopted by which the air from various parts of the tank is extracted and subjected to a three-fold test with a eudiometer to see whether there is any diminution of volume on explosion, with a Davy lamp to see whether the flame is altered by the suspected air, and with an open flame to see if it is inflammable, and if so, what kind of flame is produced; and it is only when these three tests are satisfactory that a light can be allowed near the tank. Otherwise, ventilation must be rigorously carried out.

[Tar Still Explosion.] Boiler Explosions Act, Official Inquiries under the. Board of Trade J., Aug. 1897, 132.

A FORMAL investigation was held at Manchester on the 27th instant into the circumstances attending the explosion of a boiler, or still, at some chemical works at Bradford, Manchester, on the 19th June last.

On the 14th June, anthracene oil was run into the still, and after light oil and creosote had been distilled therefrom, the result of the distillation changed to anthracene on the night of the 15th June. The distillation of this product ceased at 3 a.m. on the 16th June.

Anthracene solidifies at a temperature of 70° F., and on the early morning of the 16th June the weather was unusually cold. On the morning of the 17th June some tar was run into the still, and the fire was lit about 8 a.m. At 10.30 a.m. there was a terrific explosion; the upper part of the still was blown over a wall, the released tar was set on fire, the stillman and boiler attendant being killed.

The Court found that the still was properly constructed for the purpose for which it was intended, but that it should have been fitted with an efficient safety-valve placed on the swan neck.

There was no evidence to show what means were taken by the stillman to ascertain whether the pipes were clear before lighting the fire on the morning of the 17th June.

The Court were of opinion that the explosion was due to the pipe leading from the still to the worm becoming sealed up from the solidification of anthracene.

It was proved at the inquiry that stills of this description have been in work for at least 35 years, and that it has not been the practice to fit them with efficient safety-valves, as in their ordinary working conditions they are not liable to steam pressure.

In these circumstances the Court exonerated the owner and his manager from blame, and they made no order as to costs.

Wood Spirit, Presence of Ethylic Alcohol in Raw. G. Arachequesne. Bull. Assoc. Chim. **14**, [11], 1108—1111.

The presence of traces of alcohol having been demonstrated on several occasions in wood spirit by the official method of Riche and Bardy, both in that from distilling wood and from the dry distillation of vinasses, it has been suggested that its presence might be due to the use of carboys which had previously contained ethylic alcohol and had not been rinsed. But as sealed samples taken from the vat before filling into carboys, also showed coloration indicating traces of ethylic alcohol, this explanation had to be abandoned. Various samples of wood spirit were tested without result until, after distilling the base of a pile of wood, very damp and attacked by fungus, traces of alcohol were again found. The same result followed on two other occasions when brushwood was used with some of the leaves still attached, and again from vinasses distilled by the Vincent process. In the latter case the explanation is easy, although no fermentation alcohol could have been left in the vinasses, as they are not introduced into the retorts for dry distillation until they have been brought to a pasty condition by evaporation on the hearth of a furnace similar to the Porion furnace; but the molasses, and consequently the vinasses derived from it, must have contained lactate of lime from the fermentation in the char filters universally employed in sugar works. From this to the synthesis of alcohol by the dry distillation of calcium lactate is but one step :—$C_3H_6O_3 + CaO = CaCO_3 + C_2H_6O$.

This explanation could scarcely apply in the case of the distillation of wood, even supposing the fungus had formed lactic acid at the expense of the cellulose, since the reaction requires an excess of lime.

In the production of acetone by distilling crude grey pyrolignite of lime, which contains calcium formate and other salts of fatty acids, aldehyde is formed, from which alcohol may be produced by hydrogenation. No trace of the reaction is found on distilling pure calcium acetate in the laboratory.

The proportion of ethylic alcohol, when it exists, is so small as to offer little interest of itself industrially, except as regards suspicion of fraudulent evasion of the excise regulations.—L. J. de W.

* *Wood, Modern Distillation of, for the Production of Acetic Acid, Wood Spirit, and Acetone in a Pure Form.* M. Klar. Chem. Ind. 1897, **20**, [7], 152—158, and [8], 176—180.

Although the primitive method of making charcoal by charring wood in heaps (Meiler) is no longer carried on in Germany—the carbonisation being now almost exclusively conducted in retorts for the purpose of obtaining at the same time acetic acid, wood spirit, and acetone—the old process is still very generally worked in other countries where forest lands abound. Recently, however, efforts have been made everywhere to turn forest wealth to better account, and in this respect, America has led the way by starting a large number of works for the carbonisation of wood with special reference to the production of acetate of lime and wood spirit. Russia is also making rapid progress in this direction. According to private information communicated to the author by Titschenko, the carbonisation of wood is carried on almost exclusively, and in a most primitive manner, by Russian peasants in the governments of Welsk, Vologda, and Archangel, in the crown forest and the forests of the apanage resorts. Latterly, however, great changes have taken place, partly owing to the rapid development of the Russian textile industry, but mainly on account of the increase in the import duty on acetate of lime and acetic acid in 1891.

The conditions which influence the yield of acetic acid in distilling wood in retorts, are the species and the nature (moisture) of the wood, the size of the charge, the mode of firing, the temperature and duration of the carbonisation, the pressure in the retort, and the construction of the latter. Since woods with foliage, grown on a dry soil and felled in mid-winter give the best yield, well-seasoned timber is mostly employed, such as oak, barked birch, and especially red beech. (Air-dried wood contains about 20 per cent. of water.) The wood of coniferous trees is less adapted for the production of acetic acid; however, inasmuch as it yields oil of turpentine, resin, and tar in large quantities, special appliances for their rapid removal from the retorts and their subsequent condensation have been devised. Experience has proved that the best yield of acetic acid is obtained by heating the wood gradually to a maximum temperature of 350°. It is further essential to withdraw the acid vapours, as they are liberated, rapidly from the retort, for which purpose exhausters are used. By allowing the gas to remain in contact with the heated iron walls of the retort and failing to adopt measures for preventing the formation of pressure therein, decomposition takes place and loss ensues.

The apparatus most generally employed may be divided into three groups: (1.) Carbonising apparatus for leaved woods, (*a*) with horizontal retorts, (*b*) with vertical retorts. (2.) Carbonising apparatus for woods of coniferous trees. (3.) Carbonising apparatus for wood waste, sawdust, and the like. Almost all the drawings accompanying this paper are reproductions of typical forms of apparatus constructed by the firm of F. Leinhaas, of Freiberg, Saxony. The diagrams exhibited in Figs. 1 and 2 represent the side and

Fig. 1.

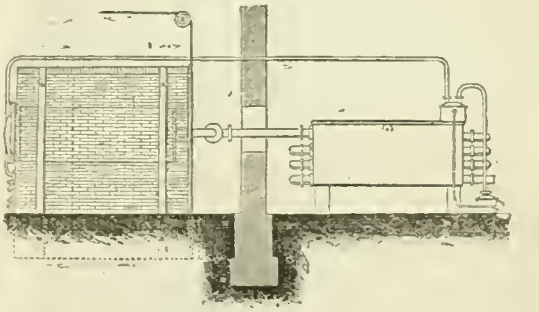
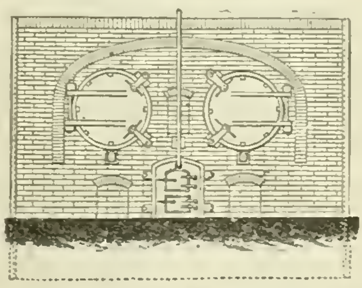


Fig. 2.



front elevations respectively of a carbonising plant worked with horizontal retorts, this form being most generally adopted in Germany. The retort consists of a wrought-iron cylindrical vessel, to the front end of which an iron charging door, working on hinges, is attached, the volatile products being carried off by a pipe at the back. The size most usually chosen renders it possible to work off two charges in 24 hours, the retort being capable of taking two billets lengthways. The charging is effected by manual labour—mechanical contrivances, as used in gas-making, not having as yet been applied. Each furnace contains two retorts, the flues and arches being arranged so that the flame does not play directly on to the retort. The retorts are heated by the gas liberated from the wood, which is aided by a coal fire. Wood tar is used when its market price is lower than its fuel value. Other

* For earlier literature of Wood Distillation, see Muspratt's Dict. 1860, Art. "Acetic Acid"; Spon's Dict., Art. "Acetic Acid"; E. T. Chapman, Chem. News, Feb. 24, 1871, 91—93, also 105; Watson Smith, J. Chem. Soc. 1871, 1104.

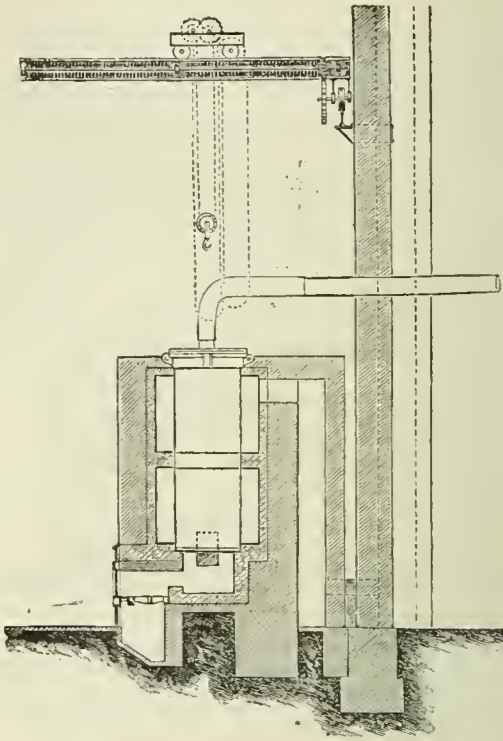
sources of heat are superheated steam and hot gases, such as carbonic oxide, carbonic anhydride, nitrogen, generator- and water-gas. Although when using superheated steam it is possible to control the temperature more accurately and attain greater uniformity, its adoption in practice has not been attended with profitable results, owing to the fact that it enhances the cost of the entire apparatus, as the vessels must be strengthened in every part and the system of condensation augmented. The dilution of the products is another objection. Zwillinger (this Journal, 1891, 129) proposes to effect the carbonisation by forcing air charged with steam and heated to 320° to 400 into the retort, having previously freed the air as much as possible from oxygen by washing it at 90°. Leinhaas has patented a charring oven, which is heated by gas, the waste heat, instead of passing away to the chimney stack, being introduced into the actual coking apartment, wherein a temperature of 200° to 260° is maintained by the admission of an easily regulated supply of air. The gaseous products are exhausted by means of high-pressure exhausters, the liquid products being condensed, whilst the gases, after passing through a washer, escape to the oven, where they are burnt. The advantages are: (1.) The oven being constructed entirely of brickwork, the repairs are small. (2.) The production of solid, hard charcoal in large pieces. (3.) The formation of acetic acid containing from 10 to 12 per cent. of pure acid. (4.) Saving of a chimney stack and of fuel. (5.) Saving in labour, it being possible to carbonise logs 1½ metres in length and 300 mm. in thickness.

To economise heat, the ovens are usually arranged in groups and joined together, the furnace gases being utilised, before they escape to the chimney, for heating evaporating pans, drying chambers, and the like. By working the retorts in batteries and introducing the charges at different stages, so that the distillation is completed, say, in the first retort as the last is receiving its load, the process is facilitated and the labour utilised more judiciously. The charcoal is transferred by various mechanical contrivances to wrought-iron coolers, advantage being frequently taken of an iron disc to which a chain is attached. This is drawn to the front after the carbonisation is completed, the charcoal falling into the subjacent cooler. The pipe destined for carrying off the gaseous products is led to the condenser, each containing two coils for cooling the gases from two retorts. The worm consists of copper pipes, which are placed at a certain inclination. The elbow pieces connecting the pipes are on the outside of the condensing tank, and, being readily disconnected, the pipes can be easily and efficiently cleaned. On distilling woods yielding much tar, a tar separator is inserted between the retort and the condenser. To prevent any return of the gas towards the retorts a water seal is employed. The crude acetic acid is collected in wooden vats or underground tanks lined with planks, in which it is allowed to settle as long as possible to separate all tarry matter—an acid which has been well settled, offering numerous advantages in its subsequent treatment.

For the carbonisation of wood in vertical retorts the condensing and separating plant is similar to that used in the case of horizontal retorts. The retorts are wrought-iron cylinders set in pairs in brickwork, as illustrated in Fig. 3, special provision being made to prevent injury to the retort by direct contact with the flame. A crane travelling on rails above the brickwork lifts the retort out of its bed, after the charge has been worked off, and conveys it to the charcoal store; whilst another retort, previously charged, is immediately lowered into the vacant space with a second crane. The heat radiating from the brickwork is thus economised and the necessity for providing charcoal coolers obviated. The product is said to be greater and of superior quality. Such an installation is, however, very costly, as twice the number of retorts is required, and the frequent removal of the retorts gives rise to greater wear and tear.

For distilling the wood of coniferous trees, an arrangement for separating the oil of turpentine and resin is required in addition to the usual condensing apparatus. For this purpose Hessel's thermo boilers are used; they are vertical retorts set in brickwork, the flues being arranged so that the shell of the retort only is exposed to the fire, the bottom being protected. The retorts are stationary, and carry the necessary mountings for removing the gaseous

Fig. 3.



products and introducing steam, superheated or otherwise. Immediately above the bottom of the retort, provision is made for discharging the charcoal after the carbonisation is completed. The more volatile products, before entering the condenser, are passed through a separator or a series of separators, in which the tarry constituents are collected, whilst the fused resin and the heavier products are carried away through a pipe near the bottom of the retort. The pipe which conducts the gaseous products to the condenser is connected with two coolers, one of which condenses the oil of turpentine and the other the ordinary products, such as acetic acid, light tars, &c.

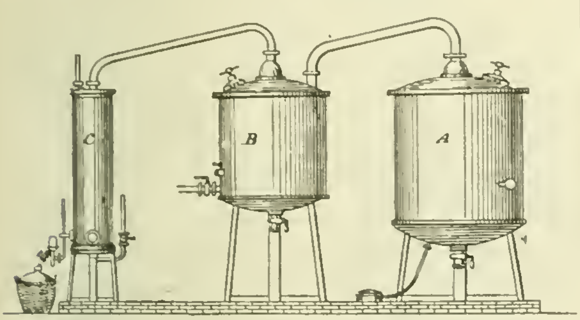
For the production of oil of turpentine, the retort is heated at the commencement of the carbonisation by a gentle fire, the operation being aided by the simultaneous introduction of steam, which is eventually superheated. Before condensing the gaseous products they are passed through milk of lime. When the distillate ceases to show particles of oil, the steam is shut off and the carbonisation completed by external firing. The oil of turpentine is in the first place redistilled with ordinary or superheated steam; it is then rectified by distilling it under a partial vacuum, to free it from creosote, and finally fractionated in column stills. By subjecting the resinous constituents to fractional distillation, a further quantity of oil of turpentine is obtained. The lighter portions are successively washed in emulsion apparatus with soda, water, acid; again with water, and finally redistilled with steam.

A number of patents have been secured by Bergmann for the carbonisation of wood waste, such as sawdust, spent dye wood, and tanner's bark (this Journal, 1895, 857). It is proposed to mould the materials into perforated briquettes, by subjecting them to great pressure, after which they are heated in horizontal retorts. The charcoal is said to be sufficiently solid to be transported without falling to pieces.

To draw the products of distillation out of the retort immediately they are formed, and to carry them at once to the condenser, Schmidt has patented a method, which consists in blowing through an injector, heated air at a suitable point into the discharge pipe of the distilling retort.

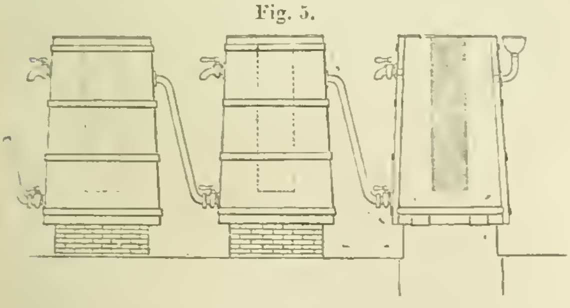
Crude acetic acid obtained from wood, the carbonisation of which has been properly conducted, contains, besides tarry substances and the homologues of acetic acid, about 10 per cent. of acetic acid, 1 per cent. of methyl alcohol, and 0·1 per cent. of acetone. On subjecting it to distillation, the tarry constituents are in a great part removed and wood spirit distils over. The separation of the latter is effected either by a simple process of distillation or by first combining the acid with a base and expelling the spirit therefrom by the aid of heat. For the distillation the apparatus shown in Fig. 4 is mostly employed. It is capable of

Fig. 4.



yielding from 3,500 to 7,000 litres of product in 24 hours. The crude acid is pumped into the copper still A and heated with steam. The volatile products are carried into the receiver B, containing slaked lime, wherein the acetic acid is absorbed, whilst the wood spirit passes through the condenser C, which is provided with a spirit seal, and is collected in a suitable vessel. Owing to the accumulation of tarry matter in A, which necessitates frequent cleaning, attempts have been made to remove the greater part before introducing the acid into the still. For this purpose an apparatus has been patented by the firm E. Leinhaas, of Freiberg, which is illustrated in the accompanying diagram (Fig. 5). It consists of three vats fitted with copper and

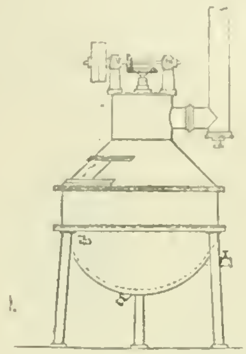
Fig. 5.



phosphor-bronze mountings. The first contains a funnel pipe, through which a suitable clarifying substance is introduced. Having charged the vat with crude acid, a gentle heat is applied by means of a steam coil, when the clarifying agent, as it dissolves, causes the tarry matter to separate out and rise to the surface of the acid. The filtration is effected in the second vat and the cooling in the third, from which the greater part of clarifying agent is recovered, the balance remaining behind in the first vat.

When the slaked lime in B (Fig. 4) has been sufficiently saturated with acetic acid, the acetate liquor is passed through a filter press to separate excess of lime and tarry impurities. According to Black, the lime sludge remaining in the press is mixed with sawdust, tanner's bark, or similar waste material, moulded into briquettes, and subjected to destructive distillation, the volatile products, such as acetic acid, acetone, and tar, being condensed, whilst the gas and charcoal are utilised as fuel. The acetate of lime liquor is evaporated in copper pans, the usual construction being that shown in Fig. 6. The pan is heated by a steam jacket.

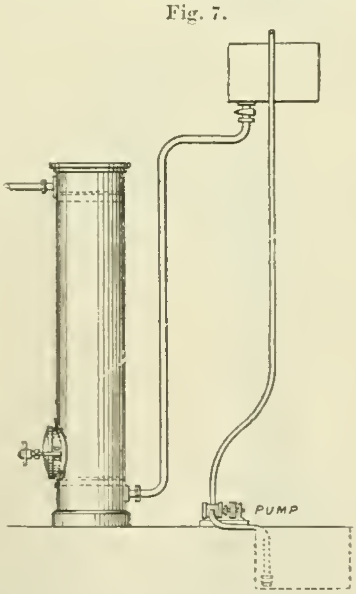
Fig. 6.



and contains a set of stirrers to prevent the acetate from burning to the bottom. The tarry matter separated during this operation and after acidification with hydrochloric acid, is removed through a sliding door, through which the progress of the operation is simultaneously watched. The acetate is then dried in suitable rooms heated with waste furnace or retort gases. During this operation the last traces of tarry substances are decomposed, a grey product remaining, which contains from 80 to 82 per cent. of calcium acetate.

The purification of crude spirit is effected by subjecting it to filtration through wood charcoal in a tower, the construction of which is shown in Fig. 7. The object is to remove the greater part of the ketones, tarry bodies, and

Fig. 7.



hydrocarbons, after which the rectification is proceeded with. This is performed in column stills in the presence of lime. Figs. 8, 9, and 10 represent the forms most generally adopted. The stills have a capacity ranging between 1,000 and 3,000 litres. The apparatus consists of a wrought-iron or copper still, a copper condenser contained in a wrought-iron water tank, a copper separator fitted with return pipe, a refrigerator provided with seal and the necessary steam and water fittings. To maintain a uniform pressure in the column, a manometer is used, as shown in Figs. 8 and 10, in addition to which a thermometer is frequently employed to control the flow of spirit. The water used for cooling is introduced into the refrigerator at the bottom,

and having reached the requisite temperature, passes into the water tank containing the condenser, whence it either runs away or is pumped to the upper part of the column. The temperature of the water as it leaves the apparatus should be ascertained from time to time, to determine whether it is that which experience has shown to give the best result. The first distillate, known as first runnings, is always more or less coloured. The temperature is then carefully noted, together with the density of the distillate, and the receiver is changed as soon as the middle fraction, constituting

Fig. 8.

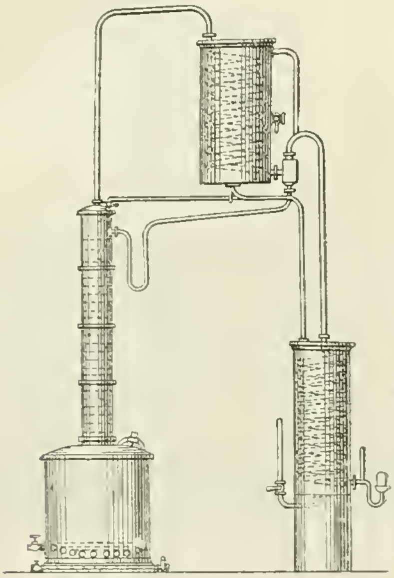
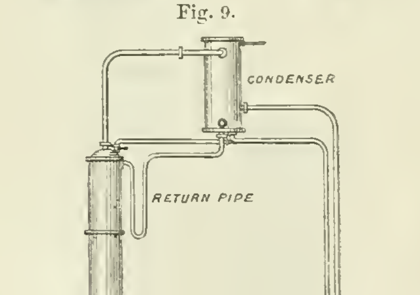
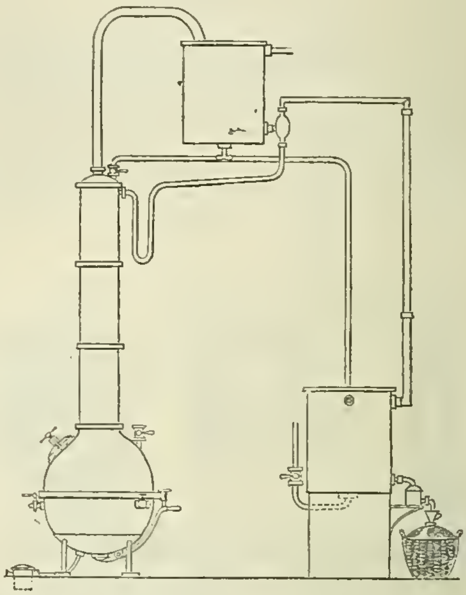


Fig. 9.



spirit of the highest concentration, begins to distil over. After this, higher boiling products come over, their presence being first noticeable by the turbidity of the distillate produced when water is added to it. Subsequently the distillate itself is rendered turbid, and eventually it comes over in two layers—oil and water, the quantity of the latter increasing until, at the end of the distillation, water only distils over, impregnated with empyreumatic substances. The fraction from which water separates oily products, is washed with water, and the washings are added to the turbid distillate. The mixture is then redistilled in the next still with a fresh charge of crude spirit. The oily fractions are mixed together and redistilled separately, when a further quantity of middle fraction is obtained.

Fig. 10.



The treatment of wood tar for the production of creosote and guaiacol is not practised very largely by wood-coking establishments, most works being contented with its utilisation as waggon grease, or for the preservation of timber, for which purpose it is dehydrated. For the recovery of its products, it is treated with lime and subsequently dehydrated in stills having a steam jacket at the bottom. The fractionation is effected in cast-iron tar stills of 1,000 litres capacity, heated by a direct fire. The first product is wood spirit; then acetic acid comes over. The distillate up to 150°—known as light oils—has a specific gravity of about 0·980, whilst the heavy oils distilling up to 250° have a specific gravity of about 1·01. Above this temperature the quantity of paraffin and gaseous products increases, and towards the end, anthracene comes over. The residue in the still is wood-tar pitch, and forms a considerable proportion of the original material. For the production of creosote, the light and heavy oils are mixed together and neutralised exactly with soda to remove fatty acids. They are then fractionated, the portion distilling between 150° and 250° being collected separately. This is treated with a concentrated solution of potash or soda, allowed to settle, run off, boiled to expel hydrocarbons, and acidified with sulphuric acid. This operation is repeated until the product is completely soluble in alkali. The final traces of phenol are removed by washing the oil with weak soda or ammonia. It is then redistilled, oxidised with chromic acid mixture, and fractionated in glass, the portion boiling between 205° and 220° being collected separately. This has a specific gravity of not less than 1·07, and represents the creosote of the German Pharmacopœia.

Guaiacol, which is the active principle of wood creosote from beech tar, of which the latter contains about 60 per cent., is now prepared in a pure form by subjecting wood creosote to distillation and treating the fraction boiling between 200° and 205° repeatedly with moderately strong ammonia to separate acids and phenols. The product is then redistilled. The low-boiling portions are dissolved in ether and treated with a strong alcoholic solution of potash. The potassium salt is washed with ether, recrystallised from alcohol, decomposed with dilute sulphuric acid, and fractionated, the distillate boiling at 200°—202° forming pure guaiacol. On cooling the latter by a freezing

mixture to $-10°$, chemically pure guaiacol separates out. It boils at 205° and has a specific gravity of 1·113. According to von Heyden, Nachfolger, the separation of guaiacol and creosol from phenols is effected by preparing the corresponding barium salts and separating the more readily soluble phenol salt by fractional crystallisation. Rumpf for the same purpose recommends their conversion into magnesium compounds, the phenol salt being readily soluble.—D. B.

Paraffin in Brown Coal-Tar Products, Estimation of. H. Eisenlohr. Zeits. angew. Chem. 1897, [10] and [11], 300 and 333.

See under XXIII., *page* 701.

IV.—COLOURING MATTERS AND DYES.

1.1'-Dihydroxynaphthalene Sulphonic Acid. L. Paul. Zeits. f. angew. Chem. 1896, 619—620.

By fusing naphthol disulphonic acid S [α-naphthol-β-disulphonic acid (Schöllkopf)] with caustic soda, it is converted into 1.1'.4-dihydroxynaphthalene sulphonic acid, which gives with diazo and tetrazo compounds, dyestuffs of very bright blue shades, capable of forming chrome lakes. Thus the dyestuffs from diazotised sulphanilic or anisidine sulphonic acids yield dyestuffs similar in shade to magenta, but possessing much greater fastness to milling and light than the sulphonic acids of phenylrosinduline, known as azocarmine. For the preparation of the 1.1'.4-dihydroxynaphthalene sulphonic acid, 6 kilos. of caustic soda and 2 litres of water are heated to 120° C. until dissolved, in a cast-iron vessel provided with an agitator. 4 kilos. of caustic soda in small pieces are then added, and the whole raised to 180° C. in about $3\frac{1}{2}$ hours, until completely dissolved. In the course of $2\frac{1}{4}$ hours, 4 kilos. of sodium α-naphthol-δ-disulphonate (92 per cent.) are then added, the temperature rising to 210°—215° C., which is maintained for about $2\frac{1}{2}$ hours. A sample of the melt is then dissolved in water, filtered, acidified with acetic acid, and treated with diazobenzene solution until all the dihydroxy acid is precipitated as dyestuff. When the filtrate, on the addition of sodium acetate and diazobenzene, gives no further precipitate of dyestuff and does not on the addition of ammonia yield an orange colour, due to the presence of undecomposed disulphonic acid, the reaction is complete, and the melt is run out. After cooling, it is dissolved in 31 kilos. of hydrochloric acid (20° B.), boiled up with wet steam, and filtered from the precipitated hydroxy acid. The press cakes, weighing 7 kilos., are then dissolved in water, and, after titration with diazobenzene, the solution is used directly for the preparation of dyestuffs. From 3·68 kilos. of 100 per cent. α-naphthol-δ-disulphonic acid, 2·96 kilos. of 1.1'.4-dihydroxynaphthalene sulphonic acid were obtained, corresponding to 72·7 per cent. of the theoretical yield. —T. A. L.

α-Amido-β-Naphthol Ethers. L. Paul. Zeits. f. angew. Chem. 1896, 620—622.

By replacing α-naphthylamine by the ethers of its β-hydroxy derivative in the manufacture of dyestuffs, much brighter and bluer shades are produced. The following details are given with regard to the manufacture of these raw materials:—In a cast-iron vessel provided with an agitator, 4 kilos. of alcohol (95° Tr.) are quickly mixed with an equal weight of sulphuric acid (66° B.); then 4 kilos. of β-naphthol are added, and the whole is heated for 6—8 hours to 95° C. The melt is then transferred to another vessel, allowed to cool, and the upper layer of solid β-naphthol ethyl ether melted with 25 litres of water and 1·5 kilos. of soda lye (40° B.). After cooling, the ether is washed with 25 litres of hot water, fused, and dried, the yield being 4—4·2 kilos. In order to nitrate it, 3 kilos. of the ether are mixed with the same weight of water and heated in a brine bath to 90° C.; then nitric acid (40° B.) is run in until a reaction commences, which requires about 1·5 kilos. The temperature is maintained at about 95° C. by running in about 2 kilos. more of nitric acid during 5—6 hours. The crude nitro-product, after washing with hot water, weighs $3\frac{1}{2}$ kilos., and when crystallised from 15 kilos. of alcohol, weighs 2—3 kilos., and solidifies at 94° C. The recrystallised product is then dissolved in 8 kilos. of alcohol and rapidly stirred whilst cooling, in order to obtain it in a fine state of division, and after adding 6 kilos. of hydrochloric acid (20° B.), zinc dust (4·2—4·5 kilos.) is slowly added, the temperature being maintained at 80°—90° C. 7 kilos. of hydrochloric acid are then added, and later another quantity of 6 kilos., in order to precipitate the α-amido-β-naphthol ethyl ether hydrochloride. For purification, 4·5 kilos. of the crude hydrochloride are dissolved in 45 litres of water, slightly acidified, filtered, and precipitated with 20 kilos. of hydrochloric acid (20° B.). The hydrochloride is then stirred to a paste with water, and gives 8·1 kilos. of a 24·3 per cent. paste = 1·97 kilos. of 100 per cent. salt., or 45 per cent. of the theoretical yield calculated on the naphthol. The base is obtained by neutralisation with 1·25 litres of soda-lye (40° B.) and extraction with benzene, when it forms a slightly dark, crystalline mass. It can be acetylated by dissolving 280 grms. in 2·5 times its weight of benzene and adding 170 grms. of acetic anhydride. After crystallising from water, the substance forms thin colourless needles, melting at 114° C. The yield is 110 grms. The preparation of α-amido-β-naphthol methyl ether is carried out in the same way, 5 kilos. of nitric acid being required, and 3 kilos. of crude nitro product being formed, melting at 112°·5 C. On recrystallisation, this gave 1·8 kilo., melting at 125°·25 C. It is, however, doubtful whether the amidonaphthol ethers will have a general technical employment, owing to their cost.—T. A. L.

Nitrobenzene, The Fusion Point, Boiling Point, and Specific Gravity of. R. J. Friswell. Proc. Chem. Soc. 1897, [182], 147—148.

Great discrepancies are found in the books as to the above constants.

Schultz gives in his first edition, 1882, 1·2002 at 0° and 1·1866 at 14·4°; in his second edition, 1·208 at 15°. Fusion point + 3°.

Beilstein gives the same, and quotes Mitscherlich for the fusion point and Kopp for the specific gravity.

Gmelin gives 1·209 and + 3°, and quotes Mitscherlich. As to boiling points, Gmelin, quoting Mitscherlich, gives 213°; Schultz, 1882, gives 210°; in 1886, 206°—207°; Beilstein, quoting Städeler, 205° at 730 mm.

It would thus appear that several of these numbers have been quoted unverified for over 60 years.

No statement of specific gravity of solid nitrobenzene has been published, excepting that in *Watts' Dictionary*, given to A. G. Green in a private communication by R. J. Friswell. Determinations of the various points have been made. Calculated for comparison with water at 4° the sp. grs. are:—

——	*t*	*d*
Solid	1·5	1·3440
Liquid	3·8	1·2220
"	13·0	1·2160
"	28·0	1·1931

Boiling point corrected 209°·0. Melting and solidifying point + 5°.

Nitrobenzene is remarkable as having a distinctly coloured vapour very closely resembling that of chlorine. The colour is easily visible in a thickness of about 2 ins., and is strongly marked when 6—8 ins. are examined. The author is not aware of any other organic vapour of so simple a constitution which is visibly coloured.

No bands of absorption are shown in the visible spectrum when light is transmitted through this vapour. The violet and blue are absorbed as with the fluid, but less strongly.

Nitrobenzene, The Action of Light on a Solution of, in Concentrated Sulphuric Acid. R. J. Friswell. Proc. Chem. Soc. 1897, [182], 148—149.

Nitrobenzene is, as has long been known, readily soluble in concentrated sulphuric acid of 1·84 sp. gr. and upwards, but a comparatively small amount of dilution precipitates it, and at about 1·7°, the solubility is very slight.

When a solution in pure concentrated acid is exposed to light it slowly darkens. When exposed to direct sunlight, the darkening goes on with great rapidity, and in a few minutes the solution becomes quite black and opaque; then the action ceases. The light from burning magnesium produces the same effect.

The solution has been kept unchanged in the dark for upwards of four years. If the exposure to light takes place in a stoppered bottle, a slight odour of sulphurous acid is perceptible after some time in the air above the solution.

If the nitrobenzene thus used be recovered and redistilled, and then redissolved in sulphuric acid, it behaves in exactly the same way.

Attempts were made to increase the change by spreading the solution on glass beads and between sheets of glass, but the depth of colour of the product soon brought all power of perceiving further change to an end.

Several hundred grammes of the black solution were prepared and attempts made to isolate the products of the change, but though a brownish calcium salt was obtained and an ammonium salt in solution, the latter decomposed on evaporation with a caramel-like odour; what was left was treated with phosphoric chloride, but no satisfactorily pure product could be obtained. The matter needs further investigation.

The rate of blackening of the solution is undoubtedly a measure of the actinic power of the light.

The author thought it quite possible that the change in the solution of nitrobenzene in sulphuric acid when exposed to light was of the same kind as that induced by electrolysis. He had never observed any coloration on sulphonating nitrobenzene when the materials were pure and light was excluded.

Chloronaphthalenedisulphonic Acids into Dichloronaphthalenesulphonic Acids, Conversion of. H. E. Armstrong and W. P. Wynne. Proc. Chem. Soc. 1897 [182], 152—154.

In the course of their studies of naphthalene derivatives, the authors have had occasion to make great use of phosphorus pentachloride as an agent for displacing the SO_3H radicle by chlorine. It was therefore necessary to establish in every possible way the validity of this method of determining constitution in the naphthalene series, as it is obvious that the occurrence of isomeric change in any one case would materially weaken the force of all arguments based on its application.

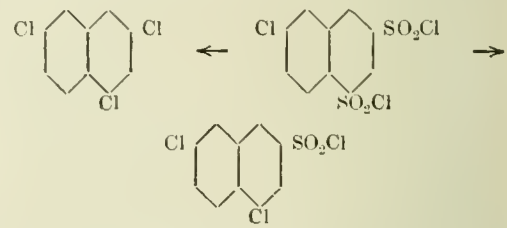
With regard to the nature of the interaction, it is to be noted that, in a previous communication (Proc. Chem. Soc. 1895, **11**, 83), it has been shown that the main function of the pentachloride is to promote the elimination of SO_2 from the SO_2Cl radicle.

In the case of the chloronaphthalenesulphonic chlorides, the amount of dichloronaphthalene obtained by means of phosphorus pentachloride is relatively considerable, and the residue left after its removal from the crude product by distillation with steam yields nothing but the original chloronaphthalenesulphonic acid on hydrolysis.

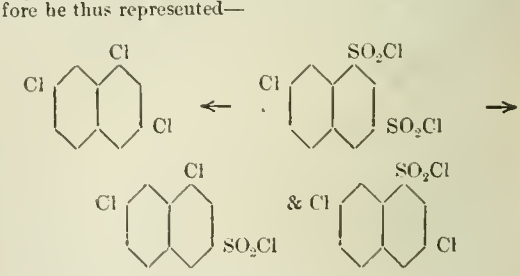
The chloronaphthalenedisulphonic chorides, however, behave somewhat differently, affording but a comparatively poor yield—rarely exceeding 30 per cent. of the theoretical amount—of trichloronaphthalene.

The authors have felt it to be incumbent on them to thoroughly examine the residues left after separating the trichloronaphthalenes, which they have had occasion to produce on a large scale (Proc. Chem. Soc. 1895, **11**, 86). Although, in view of the uniformity of the end-products, it was improbable that any change in orientation had taken place at the somewhat high temperatures at which the interactions were effected, it was obviously important to ascertain in every possible way whether such was the fact. The results to be recorded are of interest, as they serve in every case to justify the conclusion previously arrived at, that the treatment of sulphonic chlorides with phosphorus pentachloride may be thoroughly trusted as a means of determining constitution in the naphthalene series. Two cases may be quoted as typical of the behaviour of chloronaphthalenedisulphonic chlorides in general.

When 2-chloronaphthalene - 4′ : 2′ - disulphonic chloride (this Journal, 1890, 800—801) is heated with the theoretical quantity of phosphorus pentachloride at 175° during two hours, it yields both 2 : 4′ : 2′-trichloronaphthalene and 2 : 4′-dichloronaphthalene-2′-sulphonic chloride in about equal proportions, about 50 per cent. of the material remaining unchanged. *2 : 4′-Dichloronaphthalene-2′-sulphonic acid* affords a sparingly soluble *barium* salt, crystallising with 3½ molecular proportions of water in microscopic needles; a sparingly soluble *potassium* salt, containing 1½ molecular proportions, in thin scales; a *chloride* crystallising from benzene in small prisms melting at 156°; an *amide* crystallising from dilute alcohol in slender needles melting at 196°; and when the chloride is heated at 180°—185°, with phosphorus pentachloride it is converted into 2 : 4′ : 2′-trichloronaphthalene. On hydrolysing the chloride in sealed tubes with concentrated hydrochloric acid at 290°, or the potassium salt with a mixture of sulphuric and phosphoric acids and superheated steam, 2 : 4′-dichloronaphthalene is obtained. The course of change may therefore be thus represented:—



When 2 - chloronaphthalene - 1′ : 3′ - disulphonic chloride (this Journal, 1890, 283) is similarly heated with phosphorus pentachloride, it yields 2 : 3′ : 1′-trichloronaphthalene-3′-sulphonic and 2 : 3′-dichloronaphthalene-1′-sulphonic chlorides (Proc. Chem. Soc. 1890, 84), about 50 per cent. of the material remaining unchanged. *2 : 1′-Dichloronaphthalene-3′-sulphonic acid*, the isomeride present in the larger proportion, yields an anhydrous *potassium* salt, a *chloride* crystallising from benzene and light petroleum, and an *amide* crystallising from dilute alcohol. When the chloride is heated at 180°—185° with phosphorus pentachloride, 2 : 1′ : 3′-trichloronaphthalene is formed. On hydrolysing the chloride in sealed tubes with concentrated hydrochloric acid at 290°, or the potassium salt mixed with sulphuric and phosphoric acids, in superheated steam, 2 : 1′-dichloronaphthalene is obtained. The course of change may therefore be thus represented—



The other α-β-disulphonic chlorides behave similarly, the tendency being, however, as in the first of the above instances, to form only one of the two possible isomeric dichloronaphthalenesulphonic chlorides, no doubt because the SO_2 of the SO_2Cl radicle, like the SO_3H radicle, is more easily displaced from α- than from β-positions. It is not certain that these products are intermediate in the strict sense of the term, as the effect of prolonging the heating with phosphorus pentachloride at the minimum temperature at which the reaction takes place serves only to increase the yield both of the dichloro- and trichloro-derivatives. As the dichloronaphthalenesulphonic chlorides produced in these interactions decompose at temperatures a few degrees higher—10° to 15° in most cases—than those at which the

corresponding chloronaphthalenedisulphonic chlorides from which they are obtained undergo change, it is not difficult to understand why they escape attack by phosphorus pentachloride under the conditions observed.

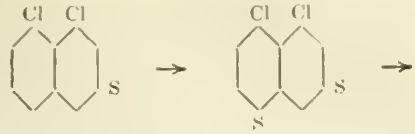
1 : 1′- *into* 1 : 4′-*Dichloronaphthalene by Hydrogen Chloride, Conversion of. The Products of Hydrolysis of* 1 : 1′-*Dichloronaphthalene* - 3 - *Sulphonic Acid.* H. E. Armstrong and W. P. Wynne. Proc. Chem. Soc. 1897, [182], 151—186.

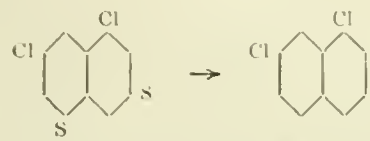
When 1 : 1′-dichloronaphthalene is heated with concentrated hydrochloric acid at 290°, it is wholly converted, save for a trace of carbonisation, into the isomeric 1 : 4′-dichloronaphthalene. This remarkable isomeric change does not seem to occur at temperatures below 200°, but is noticeable at 250°, and complete at 290°; it does not occur when 1 : 1′-dichloronaphthalene is heated either alone or with water, or with concentrated phosphoric acid at 300°, but does happen when it is heated with sulphuric acid of a strength to cause considerable carbonisation. None of the isomeric dichloronaphthalenes show any tendency to change under these conditions.

The experiments which led to these results were made in consequence of the perplexing behaviour of 1 : 1′-dichloronaphthalene-3-sulphonic acid on hydrolysis. The isomeric α-sulphonic acid (Proc. Chem. Soc. 1890, **6**, 81), requires only a temperature of 230° to effect its hydrolysis, and gives only 1 : 1′-dichloronaphthalene, whatever be the hydrolytic agent used; the β-sulphonic acid, on the contrary, is not hydrolysed below 285°, and, according to the agent used, gives one or other of no less than three dichloronaphthalenes.

1 : 1′-*Dichloronaphthalene* 3-*sulphonic acid* is obtained in addition to about an equal proportion of 1 : 1′ : 3-trichloronaphthalene, when 1 - chloronaphthalene - 1′ : 3-disulphonic chloride (this Journal, 1890, 284) is heated with phosphorus pentachloride at 160° (compare preceding abstract). It forms a sparingly soluble, anhydrous *potassium* salt, a *chloride* crystallising from benzene, an *amide* crystallising from dilute alcohol, and 1 : 1′ : 3-trichloronaphthalene when its chloride is heated either with phosphorus pentachloride at 170°, or alone at 200°—230°. On hydrolysing the potassium salt with dilute acids, such as 1 per cent. sulphuric acid or 50 per cent. phosphoric acid, at 290°, about 5—10 per cent. of the theoretical quantity of 1 : 1′-dichloronaphthalene is obtained, the residue being unchanged salt—a result by which the constitution of the acid is determined beyond doubt. When heated with 5 per cent. sulphuric acid or 60 per cent. phosphoric acid, carbonisation largely occurs, and with these and stronger acids a small amount of 1 : 4′-dichloronaphthalene is the only substance obtained, a better yield—some 20 per cent. of the theoretical—being got when the chloride is heated with concentrated hydrochloric acid at 290°. The production of 1 : 4′- instead of the expected 1 : 1′-dichloronaphthalene under these conditions is to be referred to the action o hydrogen chloride, either present or formed during the carbonisation of the salt.

On effecting hydrolysis by heating the potassium salt, mixed with sulphuric and phosphoric acids, in superheated steam instead of in sealed tubes, an unexpected result was obtained, pure 1 : 2′-dichloronaphthalene, to the extent of 40 per cent. of the theoretical amount, being the product, the remainder of the salt being carbonised. The explanation of this change has yet to be given. It is certain that the 1 : 2′-compound is not an intermediate step in the conversion of 1 : 1′- into 1 : 4′-dichloronaphthalene during hydrolysis in sealed tubes, both because it is unaffected by prolonged heating with concentrated hydrochloric acid, and because 1 : 2′-dichloronaphthalene-3-sulphonic acid cannot be detected in the material which has escaped hydrolysis, and, moreover, behaves normally on hydrolysis (compare preceding abstract). It is possible that, under the conditions specified, further sulphonation may precede hydrolysis, and that in consequence of the transference of chlorine to the para-position being thereby prevented, 1 : 2′-dichloronaphthalene is formed, thus:—





Further experiments are being made to test this view.

Of the trichloronaphthalenes, the 1 : 2 : 8-modification is the only one which undergoes change when heated with concentrated hydrochloric acid. Its sulphonic and disulphonic acids behave similarly, but the course of the action has not yet been worked out, owing to want of material.

Diphenylene Disulphide, An Isomer of. P. Genvresse. Bull. Soc. Chim. 1897, **17**, 599—609.

On heating benzene with sulphur in the presence of aluminium chloride, a mixture of diphenylene disulphide (Bull. Soc. Chim. **15**, 1035) and diphenylene isodisulphide is produced; the former is removed by extraction with glacial acetic acid, excess of sulphur by carbon bisulphide, and the diphenylene isodisulphide then recrystallised from boiling benzene, in which it is slightly soluble. Diphenylene isodisulphide, $(C_6H_4S)_2$, melting at 295°, is obtained in the form of fine needles by sublimation; it is slightly soluble in benzene and chloroform, insoluble in glacial acetic acid, carbon bisulphide, &c. Sulphuric acid gives with the isodisulphide an emerald green coloration, whilst the disulphide produces a violet.

Isodisulphide of Trioxyphenylene, $C_6S_2H_4O_3$.—This colouring matter is obtained by the action of 10 parts of fuming sulphuric acid on diphenylene isodisulphide at 120°—125° in a sealed tube. The green contents of the tube dissolve in water to a reddish solution. After nearly neutralising with barium carbonate and evaporating on the water-bath, the substance is deposited; it is then dissolved in alcohol, the solution evaporated, and these processes repeated to free it from sulphuric acid. The compound is hygroscopic, very soluble in water and alcohol, the solutions being red; in the solid state it is brown; sulphuric acid colours it green. It has not been obtained crystalline, and is insoluble in benzene, chloroform, petroleum ether, &c.

It dyes unmordanted silk a greyish copper colour and unmordanted wool a wine red; the shades are fast to soap and light, strong in an acid bath, but feeble in an alkaline bath. It does not dye unmordanted cotton. The action on successive patterns shows that the substance is not a mixture of colouring matters. The addition of a solution of common salt to the aqueous solution precipitates a compound with the colouring matter. The addition of caustic soda (1 mol.) turns the solution black; it is now alkaline, and slowly deposits a precipitate. Carbonates give no effervescence. By the action of excess of baryta the compound $C_6S_2H_2O_3Ba$ is obtained; this is a white substance, very slightly soluble in water. The addition of baryta until the aqueous solution changes colour, produces the crystalline substance $(C_6S_2H_3O_3)_2Ba$. By the action of acetic anhydride the compound $C_6S_2O_3H(CO.CH_3)_3$ was formed; this and other reactions point to the substance being a trihydric phenol. The colouring matter gives diazo colours with amines. The reaction by which diphenylene isodisulphide is produced appears to be deep-seated. The molecular weight of the compound was found by Raoult's method to be 181, agreeing with the formulæ given.—A. C. W.

Dyestuffs [Congos], and Raw Materials therefor; Manufacture of. L. Paul. Zeits. f. angew. Chem. 1896, 556—564.

The substantive cotton dyestuffs Congo Fast Blue R and and B, introduced by the Berlin Actiengesellschaft, possess valuable properties as regards fastness to light and milling,

although they do not give such bright shades as Benzazurin and Congo Blue 2 B. They are obtained by combining equimolecular proportions of tetrazoditolyl or dianisidine and α-naphthylamine, rediazotising, and combining with two molecular proportions of α-naphthol-δ disulphonic acid (Schöllkopf). It appears to be necessary, however, to employ an excess of α-naphthylamine (up to 25 per cent.), and in consequence, after the second diazotisation, in addition to the tetrazo intermediate product, there are also present unaltered tetrazoditolyl and α-diazonaphthalene, which do not yield such valuable dyestuffs. With regard to the manufacture of Congo Fast Blue R, 33 kilos. of tolidine are dissolved in 50 kilos. of hydrochloric acid (20° B.) and so much water that the solution measures about 585 litres. After determining and adding the amount of hydrochloric acid necessary for the diazotisation, to the cold decanted tolidine solution, and diluting with ice and water to about 450 litres, the tolidine hydrochloride is diazotised with 28 kilos. of a 25 per cent. nitrite solution, diluted with an equal volume of water. On the previous day, 7·5—8 kilos. of α-naphthylamine are dissolved in 400 litres of water and 11·7 kilos. of hydrochloric acid (12° B.), and made up to 800 litres. The tetrazo solution is run into this and allowed to stand two hours, when ice, 6 kilos. of hydrochloric acid, and 14 kilos. of a 25 per cent. nitrite solution are added. The diazotisation is complete when a test, after adding potassium nitrite solution, shows a slight excess of nitrite in the filtrate. The whole is then combined with 25 kilos. of 100 per cent. α-naphthol-δ-disulphonic acid, as a 2·5—3·5 per cent. solution, together with 30 kilos. of soda lye (40° B.). After heating to 30°—40° C. the dyestuff is precipitated with 100—120 kilos. of salt, filtered under a low pressure, and dried, giving, after dilution, 95—100 kilos. of dyestuff, containing 66 per cent. of pure colour. A product having similar properties is obtained by substituting 1.3.1′ naphthol disulphonic acid (ε- or Andresen's acid) for the 1.4.1′-acid.

Congo Fast Blue B is obtained by dissolving 248 kilos. of dianisidine sulphate paste, corresponding to 49 kilos. of base to a 3·3 per cent. solution, allowing it to settle, and taking such a proportion of it that the solution contains 12·3 kilos. of base, adding 40 kilos. of hydrochloric acid (12° B.) and ice, so that the total volume is 1,180 litres. This is then diazotised with 28 kilos. of a 25 per cent. nitrite solution diluted with an equal volume of water. The combination with α-naphthylamine, rediazotisation, and combination with 1.4.1′- or 1.3.1′-naphthol disulphonic acid is then effected as in the first example. The yield is about 100—110 kilos., and gives much greener shades than Congo Fast Blue R.

Congo Blue 2 B is the product obtained by combining tetrazo-anisol with R salt and 1.4-naphthol sulphonic acid. One-third of the above 3·3 per cent. dianisidine sulphate is mixed with 34·5 kilos. of hydrochloric acid (20° B.), water, and ice, so that the total volume amounts to 1,400 litres. The diazotisation with 10·5 kilos. of a 25 per cent. nitrite solution, diluted with an equal volume of water, is carried out at 5°—10° C., the whole then amounting to 1,740 litres. This is then allowed to run into 263 litres of a 10 per cent. R salt solution, after which 38 kilos. of sodium nitrite, as a 10 per cent. solution is added, the whole stirred for one hour, and then mixed with a solution containing 18·5 kilos. of 1.4-naphthol sulphonic acid obtained by decomposing diazotised naphthionic acid by boiling with water. The mixture, after agitating for 12 hours, is boiled and the dyestuff precipitated with 50 kilos. of salt. The yield is 90 kilos., which is diluted with 15 per cent. of sodium sulphate.

The dyestuff components, the δ- and ε-naphthol disulphonic acids, are derivatives of 4.1′- and 3.1′-naphthalene disulphonic acid, and are obtained by sulphonating naphthalene, nitrating, reducing, and separating the isomeric naphthylamine disulphonic acids by crystallisation. On boiling the diazo compounds of these with water, they yield the corresponding naphthol disulphonic acids. The sulphonation of the naphthalene is carried out by melting 100 kilos. of it in a cast-iron pot provided with an agitator and running in 280 kilos. of fuming sulphuric acid during 4—5 hours. After cooling to 30° C., which takes 10—12 hours, 100 kilos. of nitric acid (40° B.) are added in the course of 18 hours. The melt is then run into milk of lime and the calcium salt of the nitro disulphonic acid converted into the sodium salt. The reduction is carried out by means of iron and sulphuric acid, and after evaporation of the sodium salt to 28° B. the solution is allowed to stand for 8 days, when the sodium salt of α-naphthylamine-δ-disulphonic acid crystallises out. The filtrate treated with salt and hydrochloric acid gives crude α-naphthylamine-ε-disulphonic acid. The yield of each acid is about 20 per cent. The mother-liquor contains a β-naphthylamine disulphonic acid which shows a blue fluorescence not possessed by the other acids. On diazotisation, and boiling with dilute sulphuric acid, the δ- and ε-acids are converted into the corresponding naphthol acids, the 2—3 per cent. solutions of the sodium salts obtained, being employed directly for the preparation of dyestuffs. A better yield of the ε-acid is obtained by starting from β-naphthalene sulphonic acid. 200 kilos. of this melt, corresponding to 100 kilos. of naphthalene, are heated with 400 kilos. of sulphuric acid (66° B.) and worked up in the same way as above. The yield of α naphthylamine-ε-disulphonic acid is 27—30 per cent. of the theoretical. By the use of fuming sulphuric acid (23 per cent. SO_3) and separation of the α-amidonaphthalene-ε-disulphonic acid as acid zinc salt, a yield of about 22 per cent. of α-naphthol-ε-disulphonic acid was obtained. Both the δ- and ε-naphthylamine sulphonic acids when fused with caustic soda yield amidonaphthol sulphonic acids, the sulphonic acid in the 1′ position being replaced by hydroxyl. Chicago blue is obtained by combining tetrazo anisol with 1.1′.4-amidonaphthol sulphonic acid (δ-amidonaphthol sulphonic acid), which is formed from α-amidonaphthalene-δ-disulphonic acid by heating 4 kilos. of the recrystallised sodium salt with 2 litres of water and 12 kilos. of caustic soda to 190° C. in an oil-bath for 12 hours. One litre of water is then added, the melt is heated for a further six hours, run out, dissolved in a mixture of hydrochloric acid and 30 litres of water, but left slightly alkaline; it is then filtered, and afterwards precipitated with 15 kilos. of hydrochloric acid (20° B.). With regard to the formation of ε-amidonaphthol sulphonic acid, the best proportions appear to be the following:—450 grms. of sodium α-amidonaphthalene-ε-disulphonate, 450 grms. of caustic soda, and 450 c.c. of water were heated for 7—8 hours in an autoclave at a temperature of 180°—190° C. and a pressure of 12 atmospheres, when 150 grms. of ε-amidonaphthol sulphonic acid were obtained. Meldola's base diazotised and combined with this acid, does not give a black of technical importance.

—T. A. L.

Indigo Testing. J. Grossmann. J. Soc. Dyers and Colourists, 1897, 124.

See under XXIII., *page* 701.

α- and β-Naphthol, A Reaction to distinguish between. E. Léger. Bull. Soc. Chim. 17, [11], 546.

See under XXIII., *page* 700.

Fusions, Distillations, and Sublimations, Apparatus for. L. Paul. Zeits. angew. Chem. 1896, [20], 619.

See under XXIII., *page* 699.

PATENTS.

Pyrocatechin-sulphonic Acid and Intermediate Products, Impts. in the Manufacture of. G. B. Ellis, London. From "La Société Chimique des Usines du Rhone anciennement G. P. Monnet et Cartier," Lyons, France. Eng. Pat. 14,931, July 6, 1896.

Starting with the parasulphonic acid of phenol, the patentee converts this into a chloro or bromo derivative, which, on fusing with potash, yields the sulphonic acid of pyrocatechol. When this latter product is heated with dilute sulphuric acid in an autoclave it yields pure pyrocatechol. Details of the invention are as follows:—Phenol-*p*-sulphonic acid is obtained by heating together on the water-bath for several days, equimolecular proportions of phenol and sulphuric acid (66° B.). The melt, after cooling, is dissolved in one-third of its weight of water,

5—10 per cent. of sulphuric acid is then added, and a current of chlorine or bromine is passed into the solution in the proportion of two atoms of chlorine or bromine for each molecule of phenol sulphonic acid; for instance, 94 kilos. of phenol require 71 kilos. of chlorine or 160 kilos. of bromine. During the operation, the temperature rises from 20° to 60° C. The melt, after pouring into water, is limed and converted into the sodium salt, which is then mixed with half its weight or an equal weight of caustic soda in an aqueous solution, and heated either in an open vessel or under pressure for 8—10 hours to 250° C. or above. The product is dissolved in water, neutralised with 30 per cent. sulphuric acid in slight excess, the solution evaporated to a syrupy consistency, filtered from the sodium sulphate which crystallises out, and the concentrated aqueous solution of the sodium salt of pyrocatechol sulphonic acid obtained is finally heated in an autoclave with dilute sulphuric acid, yielding pure pyrocatechol.—T. A. L.

New Dyes [Yellows] relating to the Oxy-phenanthro-naphthazine Series and of New Intermediate Products, The Manufacture and Production of. J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 15,953, July 18, 1896.

An extension of Eng. Pat. 18,374 of 1893 (this Journal, 1894, 942), which described the preparation of new basic dyes from phenanthrophenazine. The present specification relates to the production of similar dyestuffs derived from phenanthronaphthazine, which contain a hydroxyl group and give very fast yellow shades on wool and silk. These products are obtained by condensing phenanthraquinone (*a*) with peri-hydroxydiamidonaphthalenes and subsequent sulphonation; (*b*) with peri-hydroxydiamidonaphthalene sulphonic acids; and (c) with certain 1.2-diamidonaphthalene sulphonic acids (the 1.2.4- and 1.2.4′-diamidonaphthalene sulphonic acids are not suitable). In this last-mentioned condensation, products are obtained in which, by subsequent fusing with alkalis, a hydroxyl is substituted for a sulphonic acid group. The final products, if insoluble, may be sulphonated. The following is one of the examples given:—The azo dyestuff, prepared in an acid solution from diazosulphanilic acid and 2.1′.3′-amidonaphthol sulphonic acid, is dissolved in about 20 times its weight of hot water and decolorised by adding hydrochloric acid and stannous chloride. On cooling, 1.2.1′.3′-diamidonaphthol sulphonic acid crystallises out, and is washed with dilute hydrochloric acid. To effect the condensation, 402 kilos. of a 5 per cent. paste of phenanthraquinone are mixed with 260 kilos. of a 10 per cent. paste of 1.2.1′.3′-diamidonaphthol sulphonic acid. About 350 litres of water are added, and the mixture is heated to 90°—95° C. until the phenanthraquinone has disappeared and a sample dissolves with a yellow colour in water. The condensation product is then salted out and filter-pressed. Like the sodium salt, it is very sparingly soluble in water, and dissolves in concentrated sulphuric acid with a greenish-blue colour. By employing in place of the above acid an equivalent quantity of 1.2.1′.3.3′- or of 1.2.1′.4′.2′-diamidonaphthol disulphonic acid, more soluble dyestuffs are obtained, which also give purer yellow shades. The sulphonic acids are obtained as above by reduction of their azo compounds with stannous chloride and hydrochloric acid. All these dyestuffs are characterised by their fastness to alkalis.—T. A. L.

Azo Colours [Cotton Blues and Blacks], Impts. in the Production of. T. R. Shillito, London. From J. R. Geigy and Co., Basle, Switzerland. Eng. Pat. 16,582, July 27, 1896.

The new bases employed in the production of the azo dyestuffs referred to are *p*-diamidoditolylamine and *p*-diamidophenyltolylamine. They are obtained by the reduction of the corresponding indamines. Thus the former is obtained by reducing with hydrochloric acid and zinc dust that indamine which is produced by oxidising *p*-tolylene diamine with *o*-toluidine, whilst the latter is formed from that indamine which is the product of the oxidation of *p*-phenylene diamine with *o*-toluidine or of *p*-tolylene diamine with aniline. The diamines may be employed in the usual manner for the production of symmetrical or unsymmetrical disazo or trisazo dyestuffs, an example of which follows:—30 kilos. of *p*-diamido ditolylamine hydrochloride in 500 litres of water are diazotised at 0° C. with 40 kilos. of hydrochloric acid (21° B.) and 14 kilos. of sodium nitrite, and mixed with a solution of 40 kilos. of sodium carbonate, to which a solution of 26 kilos. of sodium 2.1′.3′-amidonaphthol sulphonate is then added. The intermediate compound forms a dark brown precipitate, and is mixed with a solution of 12 kilos. of *m*-tolylene diamine. After standing some hours, the dyestuff is filtered off and dried. It dyes unmordanted cotton black shades. By the use of components which, after combination, are capable of further diazotisation, trisazo dyestuffs may be obtained, which also yield dark shades.

—T. A. L.

New Colouring Matters [Cotton Blacks], Manufacture or Production of. I. Levinstein and Levinstein, Ltd., Manchester. Eng. Pat. 17,065, Aug. 1, 1896.

This is an extension of Eng. Pat. 2946 of 1896 (this Journal, 1897, 136). By diazotising or tetrazotising the amido-azo dyestuffs obtained from diazotised 1.4.2-naphthylenediamine sulphonic acid and suitable amines or amidonaphthols, &c., and combining these products with phenols, amines, diamines, and the like, valuable black dyestuffs are obtained which are applicable to cotton as well as to wool. Thus 25 kilos. of amidodiazonaphthalene sulphonic acid obtained from 23·8 kilos. of 1.4.2-naphthylenediamine sulphonic acid are stirred into an alkaline solution of γ-amidonaphthol sulphonic acid at a temperature of about 20°—25° C. The dyestuff produced, is salted out and filter-pressed, and gives deep violet-black shades on wool. 53·2 kilos. of the product so obtained are dissolved in 600 litres of water, and the solution is diazotised in presence of ice with 70 kilos. of hydrochloric acid (18° B.) and 7·2 kilos. of sodium nitrite at a temperature of 8°—10° C. In a few hours the original dyestuff is converted into a dark-coloured diazo compound, which is run into an aqueous solution of 12·5 kilos. of *m*-tolylene diamine. The free acid is neutralised with sodium acetate, and the combination is complete after a short time. The whole is then made alkaline, and the new dyestuff is salted out, filter-pressed, and dried. It gives deep black shades on animal and vegetable fibres.—T. A. L.

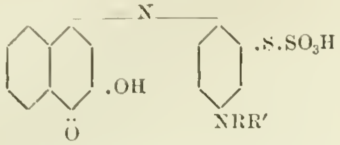
Disazo [Brown] Dyestuffs, Impts. in the Manufacture of Primary. S. Pitt, Sutton. From L. Cassella and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 17,590, Aug. 8, 1896.

The patentees find that salicylic acid is capable of taking up two azo groups by combining derivatives like diazotised *p*-nitraniline with the easily soluble azo dyestuffs obtained from diazo sulphonic acids and salicylic acid. For example, 41·6 kilos. of the dyestuff from 2.2′-diazonaphthalene sulphonic acid and salicylic acid (which dyes wool yellow) are mixed in a dilute solution with the diazo compound from 13·8 kilos. of *p*-nitraniline, the whole being kept alkaline by means of sodium carbonate. Combination at once takes place, and the dyestuff is filtered off after a few hours. It dyes chromed wool brown. The dyestuffs obtained according to this specification are fast to light and dye much more level shades than the monazo derivatives. Moreover, since they contain an azo group ortho to the hydroxyl, they are not sensitive to alkalis.—T. A. L.

Dyestuffs [Blue] on Fibre, The Production of. H. E. Newton, London. From "The Farbenfabriken vorm. F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 17,591, Aug. 8, 1896.

According to Eng. Pats. 825, 4757, 6035, 6035a, and 8093 of 1893 (this Journal, 1894, 31, 245, 383, and 510), blue thiazine dyestuffs are obtained which are known as brilliant alizarin blues. In the present specification a process is described by means of which these dyestuffs are produced on the fibre by development during steaming (without

pressure) from intermediate compounds, which are oxindophenolthiosulphonic acids of the general formula—



For example, such an intermediate compound is formed by mixing in an aqueous solution, equimolecular proportions of sodium dimethyl-*p*-phenylenediamine thiosulphonate and 1.2.4-naphthoquinone potassium sulphonate (*vide* Eng. Pat. 4757 of 1893, *loc. cit.*). The product is then salted out, and, when boiled in an alkaline solution, it is converted into the corresponding brilliant alizarin blue. When printed on a fabric together with a chromium salt and steamed, the chrome lake of the colour is developed on the fibre. A solution of 3 kilos. of the above intermediate compound in 20 litres of water is stirred into 61 kilos. of a neutral starch tragacanth thickening and subsequently mixed with 6 kilos. of a chromium acetate solution (20° B.) and 10 kilos. of a 25 per cent. sodium thiosulphate solution. This paste is printed on the fabric, which is then steamed for an hour without pressure and subsequently chalked, malted, and soaped in the usual manner. The print shows a very level, bright indigo colour, fast to washing and even boiling sodium carbonate. By employing the intermediate compound from dimethyl-*p*-phenylenediamine sodium thiosulphonate and potassium 1.2.4.3′-naphthoquinone disulphonate, a print is obtained which is more greenish-blue than that above described.—T. A. L.

Azo Dyes [Blues], Production of New. W. H. Claus, A. Rée, and L. Marchlewski, Manchester. Eng. Pat. 18,020, Aug. 14, 1896.

The products described are derivatives of *p*-diamines and γ-amidonaphthol sulphonic acid, of which the following is a typical example. The tetrazo compound from 21·2 kilos. of tolidine is added to an alkaline solution of 22·7 kilos. of 1.4-naphthol sulphonic acid, and to the intermediate compound produced there is added an alkaline solution of 49 kilos. of the amido-azo compound obtained by combining diazotised γ-amidonaphthol sulphonic acid with the acid itself. After agitating some time, the reaction is completed by heating to 50° C. and finally to 80° C. The product gives reddish-blue shades on cotton. If the alkaline solution be acidulated, diazotised with 7 kilos. of sodium nitrite, and combined with 12·2 kilos. of *m*-tolylenediamine, it yields a dyestuff which gives blackish-blue shades. For the production of the amido-azo compound referred to above, 24 kilos. of γ-amidonaphthol sulphonic acid are dissolved in 700 litres of water and the necessary quantity of sodium carbonate. The solution is made acid with hydrochloric acid, cooled with ice, diazotised with 7·1 kilos. of sodium nitrite, and run into 24 kilos. of γ-amidonaphthol sulphonic acid and 36 kilos. of sodium carbonate in 700 litres of water (see also Eng. Pat. 19,330 of 1890, and 9529 of 1894; this Journal, 1891, 917, and 1895, 478).—T. A. L.

Phthalic and Sulpho-phthalic Acids, Impts. in the Manufacture and Production of. J. Y. Johnson, London. From "The Badische Anilin and Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 18,221, Aug. 17, 1896.

The process consists in the production of phthalic acid and sulphophthalic acids by the action of fuming sulphuric acid on naphthalene at about 300° C. in presence of mercury sulphate, the metal itself or its oxides. About 100 kilos. of naphthalene, 1,500 kilos. of sulphuric acid (100 per cent. H_2SO_4), and 50 kilos. of mercury sulphate are heated until the naphthalene is dissolved. The melt is then heated in a distilling vessel, and at about 200° C. oxidation commences, proceeding more rapidly at 250° C. The melt turns dark and evolves sulphurous and carbonic acids. The temperature is finally raised to 300° C. or above, until the mass becomes viscous. The distillate consists of dilute sulphuric acid, phthalic acid, partly as anhydride, and some sulphophthalic acid. The phthalic acid is filtered off, and the residue in the retort containing the mercury sulphate can be used for another operation. In place of naphthalene, β-naphthol, naphthionic acid, or phenanthrene may be used. For the preparation of sulphophthalic acid, 100 kilos. of naphthalene are dissolved in 300 kilos. of fuming sulphuric acid (20 per cent. SO_3) and mixed with 1,200 kilos. of concentrated sulphuric acid (95 per cent. H_2SO_4). The melt is heated for 10 hours to about 250° C. A certain amount of phthalic acid distils over, and the melt is neutralised with calcium or barium carbonate and worked up in the usual way. The barium salt of a new disulphophthalic acid may be separated from water in crystalline nodules. If the temperature be maintained at about 220° C., a mono-sulphophthalic acid results.—T. A. L.

Red Colouring Matters, Impts. in the Production of. T. R. Shillito, London. From J. R. Geigy, Basle, Switzerland. Eng. Pat. 18,720, Aug. 24, 1896.

The rhodamines, like the colours of the patent blue series, show a remarkable fastness to alkalis, and both contain a substituent—the one a carboxyl and the other a sulphonic acid group—ortho to the methane carbon. The present specification describes dyestuffs of the rhodamine series which contain a sulphonic acid group in place of the carboxyl and show a similar fastness to alkalis. Benzaldehyde-o-sulphonic acid is condensed in an alkaline aqueous solution with alkylated *m*-amidophenols. The dihydroxylated alkyldiamidotriphenylmethane sulphonic acids produced, are heated with dehydrating agents—for instance, concentrated sulphuric acid—and finally the triphenylmethane oxide derivatives thus formed, are oxidised to dyestuffs in an aqueous solution by means of potassium bichromate, lead peroxide, ferric chloride, or the like. The following are the quantities employed:—37 kilos. of a 10 per cent. benzaldehyde-*o*-sulphonic acid solution, 7 kilos. of diethyl-*m*-amidophenol, and 1 kilo. of sodium carbonate are boiled for 8 hours. The solution of the tetra-ethyldiamidodihydroxytriphenylmethanesulphonic acid is evaporated to dryness, ground, and heated with 40 kilos. of concentrated sulphuric acid for 3—5 hours at 135°—145° C., diluted with 400 litres of water, boiled, and filtered. It is then oxidised with 18 kilos. of a 33 per cent. ferric chloride solution and heated to 90°—100° C. for 4—5 hours. The dyestuff separates partly in crystals, the remainder being salted out. After washing with brine it is boiled up with aqueous ammonia, which removes products soluble in alkalis, and is finally washed with water, dried, and crystallised from alcohol. The solutions, especially the alcoholic one, show a bright yellowish-red fluorescence. Hydrochloric or dilute sulphuric acid give yellowish-red solutions which turn bluish-red on dilution. These solutions dye wool and silk pure bluish-red shades perfectly fast to alkalis.

—T. A. L.

Dyestuffs [Yellowish-Reds] of the Phthaleïne Series, Process for Obtaining. H. Imray, London. From "The Basle Chemical Works Bindschedler," Basle, Switzerland. Eng. Pat. 12,180, May 17, 1897.

By condensing diethylamido-oxybenzoyl benzoic acid with *p*-amido-o-cresol, valuable dyestuffs are obtained which can be alkylated, and then form salts soluble in water, dyeing tannined cotton yellowish-red shades. A mixture of 31 kilos. of diethylamido-oxybenzoyl benzoic acid, 15 kilos. of *p*-amido-*o*-cresol ($CH_3:OH:NH_2 = 1:2:4$), 180 kilos. of 100 per cent. sulphuric acid, and 40 litres of water, is heated at 140°—160° C. until a sample dissolves in warm water. The melt is then poured into 1,000 litres of water and heated for some time. After cooling, filtering, and washing with water, the residue is suspended in 100 litres of alcohol and the same volume of 10 per cent. ammonia, heated, filtered, and mixed with 100 litres of 30 per cent. hydrochloric acid. On cooling, green crystals separate out, and 30 kilos. are heated under a reflux condenser with 100 litres of alcohol and 45 kilos. of sulphuric acid monohydrate for about eight hours. The melt is then poured into 1,000 litres of water, and the dyestuff is salted out with potassium bisulphate. It is purified by repeating the treatment. The dyestuff forms a yellowish-red powder, which dissolves with the same colour in water or in alcohol. In strong

sulphuric acid it dissolves with a yellow colour, whilst the ammoniacal alcoholic solutions have a yellowish-green fluorescence.—T. A. L.

Dyestuffs of the Phthaleïne Series, Process for obtaining New. H. Imray, London. From "The Basle Chemical Works Bindschedler," Basle, Switzerland. Eng. Pat. 12,181, May 17, 1897.

When dialkylamido-oxybenzoyl benzoic acids are condensed with resorcinol, dyestuffs are obtained which, when converted into their ethers, give yellowish-red shades on wool, silk, and tannin-mordanted cotton. A mixture of 28 kilos. of dimethylamido-oxybenzoyl benzoic acid, 11 kilos. of resorcinol, 180 kilos. of sulphuric acid monohydrate, and 180 litres of water, is heated on the water-bath until the colour no longer increases in intensity. The melt is then poured into 1,000 litres of water, heated for some time, and allowed to crystallise. 30 kilos. of the dimethylrhodol, 80 kilos. of alcohol, and 75 kilos. of sulphuric acid monohydrate are heated for several hours until a complete solution is obtained. The melt is poured into water and the precipitated base is converted into the hydrochloride, which is purified by crystallisation from water containing hydrochloric acid.—T. A. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

PATENTS.

Fibre, Paper, Tissues, and the like; Impts. in the Method of Waterproofing Fabrics made of. J. Waugh, Bradford, Yorkshire. From Amos and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 15,893, July 18, 1896.

See under XIX., *page* 696.

"Mercerising" Fabrics composed of Vegetable Fibres, Impts. in the Process of what is known as. F. A. Bernhardt, Zittau, Germany. Eng. Pat. 16,840, July 29, 1896.

During the process of mercerising, the fabric is wrapped on a hollow perforated cylinder and subjected to the pressure of a solid roller placed above the cylinder. The mercerising solution is forced through the fabric from the interior of the cylinder. It is necessary to continue the rolling pressure for some time in order to counteract the tendency of the fabric to shrink during mercerisation.
—R. B. B.

Degreasing Wool, Cotton Waste, and other Fibrous Materials; Impts. in Apparatus for. F. N. Turney, Nottingham. Eng. Pat. 18,360, Aug. 19, 1896.

The patentee describes a method of feeding material to, and withdrawing it from the apparatus, without admission of air or loss of the vapour of the volatile solvent. The receiving and degreasing chambers are connected by a flat tube or channel closed towards the degreasing chamber by a valve. At its other end the tube is provided with a series of stuffing boxes, through which work plungers.

The material is fed into the tube and is compressed by means of the plungers, which force it forward into the degreasing chamber, the material thus forming a plug and preventing the escape of vapour. For cleaning out the tube, a rod and cross-bar are attached to the plungers.—R. B. B.

Rendering Substances [Wood, Woven Fabrics, &c.] Incombustible, Impts. in. A. Issel, Genoa, Italy. Eng. Pat. 11,368, May 7, 1897.

See under IX., *page* 681.

Loading or Sizing Silk, An Impt. in. L. Hwass, Crefeld, Germany. Eng. Pat. 13,425, May 31, 1897.

It is claimed that in the weighting of silk by means of stannic chloride, sodium phosphate, and sodium silicate, the tendering action of the weighting agents is greatly diminished by the introduction, between the baths of sodium phosphate and sodium silicate, of a bath containing a soluble copper salt for dark colours or a soluble zinc salt for pale shades.—R. B. B.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Indigo Vats, Improved. J. Grossmann. J. Soc. Dyers and Colourists, 1897, 130—131.

In indigo vats of any kind, portions of the indigo are constantly being regenerated, and thus precipitated, by the oxygen of the air carried through the liquid by the cloth, and thus require constantly fresh additions of the reducing agent. Owing to this circumstance, there is a large accumulation of sediment or of foreign matters in solution, according to the kind of vat used, which tends to weaken the vat. To obviate this defect, the author prepares his vats, in the first instance, with "indigo white," thus entirely dispensing with the reducing agent at the "setting" of the vat, and during working adds small quantities only of the latter, corresponding to the quantity of indigo that is regenerated, to keep the vat in condition. In this manner a vat can be worked with $\frac{1}{3}$th of the reducing agent that would otherwise be required, giving much better results, whilst greatly minimising the risk of loss of indigo by over-reduction.—I. S.

p-Nitraniline Red Discharge on Indigo. R. J. Flintoff. J. Soc. Dyers and Colourists, 1897, 132—134.

C. Kurz has proposed a process for a *p* nitraniline red discharge on indigo (J. Soc. Dyers and Colourists, 1897, 90), which consists in padding the indigo-dyed cotton with sodium β-naphtholate, to which some potassium chromate has been added, drying and printing on the thickened solution of the diazo compound *plus* lead acetate. Proceeding in this manner, the chromic acid required for the destruction of the indigo is retained on the cloth as lead chromate, whilst the remaining chrome salt is removed during the subsequent washing process.

The author now submits a cheaper and simpler process for the same purpose. The indigo-dyed cloth is prepared with an alkaline solution of β-naphthol in the usual manner, and to the diazo nitrobenzene acetate is added the requisite quantity of sodium bichromate, which has no action on the diazo compound. The best proportions are 1,800 grms. of sodium bichromate to 4·5 litres of printing colour. The goods are dried on the drying cylinders, passed through a bath containing sulphuric and oxalic acids, well washed in cold water, and soaped at 120° F. for 10 minutes; again well washed, and dried.—I. S.

Wool Mordants, Tungsten and Molybdenum as. E. Knecht. J. Soc. Dyers and Colourists, 1897, 135.

The author has tested the value of tungsten and molybdenum as mordants on wool. The wool was boiled for an hour with 3 per cent. of sodium tungstate alone, and also with 3 per cent. of tungstate and 1 per cent. of sulphuric acid, after which it was well washed. The wool, which thus acquired a yellowish tinge, was then dyed, under the usual conditions, with the following dyestuffs: logwood, fustic, coeruleïn, alizarin, and gallocyanine. Full colours were thus obtained, the shades of which lie between those obtained on stannous and on chrome mordants, better results being obtained with sulphuric acid than without. But their fastness to light and to soap were not satisfactory.

With molybdenum the same mode of procedure was employed as for tungsten, the tungstate being replaced by ammonium molybdate. With molybdate alone the colour of the wool was not materially changed, but with an addition of sulphuric acid, the wool was coloured dull blue. In dyeing with the same colours as used for tungsten, shades were obtained which closely resembled those obtained with potassium bichromate, their fastness to light and soap being satisfactory.—I. S.

PATENTS.

Solid Paste or Soap Compound for Dyeing all kinds of Textile Materials, Manufacture of a. C. Uffelmann, Kassel, Germany. Eng. Pat. 17,382, Aug. 6, 1896.

A suitable soap with an equal weight of the required aniline colour, is dissolved in an excess of alcohol, and

added to an aqueous solution of the mordant mixed with glue, gelatin, or other binding material. After the addition of glycerin to the amount of one-third of the weight of the soap, the mixed solutions are at first gently heated, and then the temperature raised gradually until the mass has become homogeneous, and about two-thirds of the alcohol have been distilled off. The mass is then poured out, and, when cold, is cut into cakes, which are soluble in hot water and at once ready for dyeing textile materials. Although containing as much as 50 per cent. of soap, the paste does not become decomposed, owing to the large amount of mordant present.—C. A. M.

Mordanting Wool and other Animal Fibre, Impts. in the Process of. A. M. Clark, London. From C. H. Boehringer Sohn, Niederingelheim-on-Rhine, Germany. Eng. Pat. 18,896, Aug. 26, 1896.

A mordant is described consisting, for 100 kilos. of wool, of 2·65 kilos. of lactic acid (50 per cent.), 1·35 kilos. of potassium bichromate, and 0·9 kilo. of sulphuric acid, in a bath of 2,000 litres of water. The wool is entered at 160° F. and worked for half an hour; the bath is slowly heated to the boil and maintained at boiling point for half an hour. It is claimed that a complete reduction of the bichromate takes place, that the bath is completely exhausted, and that the shades are purer and faster and the fibre less affected than when other mordants are employed.
—R. B. B.

Artificial and Natural Colouring Matters, Process of Preparing Solutions of, for Dyeing and Printing Purposes. W. Clark, London. From C. H. Boehringer Sohn, Niederingelheim-on-Rhine, Germany. Eng. Pat. 21,211, Sept. 24, 1896.

Many colouring matters—*e.g.*, induline and indigo—which are insoluble or only soluble with difficulty in ordinary solvents, are readily dissolved in lactic acid or its acetyl derivatives and esters. Such solutions, it is claimed, are useful for printing on cotton or woollen fabrics, for dyeing silk, and other purposes.—R. B. B.

VII.—ACIDS, ALKALIS, AND SALTS.

Thirty-third Annual Report on Alkali, &c. Works, by the Chief Inspector. May 7, 1897.

This, Mr. R. Forbes Carpenter's second Report, shows only slight variations in the number of works registered under the Act, and in the scheduled processes of manufacture. The most notable differences are: an increase of the salt processes of from 52 to 58 as compared with 1895; and in the fibre-separation processes an increase of from 38 to 44; while arsenic processes have diminished from 44 to 37. Alkali works have diminished from 101 in 1895 to 98 in 1896, but "other works" are increased from 964 to 976. The escape of acid gases, reckoned in grains per cubic foot of total gases, does not materially differ from that of 1894–95; but the hydrochloric acid escaping as compared with that produced, which in 1895 was 1·714 per cent., is for 1896 only 1·411 per cent. The "acidity of all chimneys" given as grains of SO_3 per cubic foot, was 0·709 in 1895 and 0·803 in 1896. The acidity of gases from manure works is slightly diminished as compared with the preceding year.

The following table is given of the—

Salt decomposed in the Leblanc and Ammonia-Soda Processes (including Scotland).

——	1896.	1895.	1894.
	Tons.	Tons.	Tons.
Leblanc process.......	360,929	408,173	434,298
Ammonia-soda process.	431,577	428,614	361,603
Total	792,506	836,787	795,901

From this it will be seen that the relative positions of the Leblanc and ammonia-soda processes for 1894 and 1896 are almost exactly reversed.

Over the period covered by the Report, the manufacture of caustic soda and chlorine by electrolysis has been chiefly experimental, but progress was made at the works of the Castner-Kellner Alkali Company for starting in the present year, and the Hargreaves-Bird electrolytic process is to be conducted at Chauny by the St. Gobain Company. In the manufacture of chlorate of soda, Mr. J. Hargreaves has applied on a large scale his process for chlorinating hydrated sodium carbonate directly, in an absorbing tower, in which lixiviation of the products to remove sodium chloride is also conducted, the intermediate production of calcium or magnesium chlorate being avoided. In France, Germany, Switzerland, and Sweden, it is stated that processes for the direct electrolytic formation of chlorates have received wide extension. Processes for obtaining chlorine simultaneously with zinc or copper by the electrolysis of zinc or copper chlorides do not appear as yet to have been tried on a large scale, although the inspector considers that "Dr. Hoepfner's processes, as regards the extraction of zinc, will be tried in the first instance by ammonia-soda manufacturers, to whom indeed they would strongly appeal, as offering a promising means of rendering available the chlorine of the calcium chloride solution now run to waste in such enormous quantities." A nitric acid chlorine process has been successfully carried out in Middlesbrough, bleaching powder of the highest strength having been produced; but there is a serious drawback in the cost of concentrating the large quantities of weak sulphuric acid resulting from the decomposing operations. It is suggested that the Kessler plant for rectifying the acid would prove serviceable.

The continuous processes adopted for rectifying sulphuric acid in glass, porcelain, or enamelled iron beakers, stepped in series, have not given entire satisfaction to the Inspector, owing to the liability to the escape of acid by breakages or corrosion; and not only is a grave warning given, but a suggestion is made that some amendment of the Act of 1881 may be needed, to reduce the "maximum acidity" permitted under section 8. The "Kessler" rectifying plant, however, is described as working well and with great regularity. An acid of about 97 per cent. monohydrate is produced, not of the best "colour and brightness," but well suited for application in nitrating and in some other operations. In Scotland, the production of sulphuric acid has been active, notwithstanding the restricted condition of alkali works, as is shown by an increase of the quantity of pyrites burned of 1,817 tons over the preceding year.

The loss of ammonia in the production of soda-ash (ammonia-soda process) has been reduced, but is still equal to 2·0 per cent. of sulphate per ton of 58 per cent. alkali, which is equivalent to the loss of 5,395 tons of sulphate of ammonia for 1896. An increasing amount of ammonia is used in the manufacture of cyanides. The following table is inclusive of ammonia, either lost, or applied in manufacture without going upon the market:—

Amount of Sulphate of Ammonia produced in the United Kingdom.

——	1896.	1895.	1894.
	Tons.	Tons.	Tons.
Gasworks	127,498	119,645	113,634
Ironworks	16,511	14,588	10,075
Shale-works	37,822	38,335	32,891
Producer-gas, coke, and carbonising works.	9,078	7,083	3,448
Total	190,909	179,651	160,048

It is observable that the ammonia from gasworks has not increased so much as might have been expected from the increased production of gas. This is attributed to the introduction in part of the gas derived from the interaction of incandescent coke and steam, from which no ammonia is obtained. In America this displacement of coal-gas has reached more than 70 per cent.; and it is anticipated that the displacement will go on here at an increasing rate, even if there be more danger of poisoning from carbon monoxide

with the water-gas than with the coal-gas. There is a prospect, however, that the supply of ammonia from other sources will be increased. In the large ironworks at Merthyr and Dowlais, in South Wales, where hitherto by-products have not been collected, changes in the machinery make it probable that in the future tar and ammonia will be recovered. The successful development, also, of Dr. Mond's process for recovery of ammonia from gaseous fuel used in melting steel is referred to. In shale distillation, the introduction of the "Pentland" retort of Messrs. Young and Beilby, in 1884, appears to have been the means of the salvation of the Scotch oil trade, which has for many years been subjected to "severe stress of competition" from the American and Russian supplies. An improved form of the Pentland retort has been introduced during the year, for which it is claimed that no additional fuel for distillation is required beyond the gases derived from the retort itself.

The experiments conducted at the works of the Luton Gas Company, in using oxide of iron purifiers, with concurrent revivification by regulated steam and air admission, have been continued during the year, and a table is given of the results of analyses of the samples taken. Artificial oxide gave a very prolonged run without fouling, but the working throughout shows loss of available sulphur. Mixed Flitwick and Irish bog ores gave better results than the Flitwick used singly. In another works, where the method of alternate downward revivification, without steam, is in use, with three hours' spells of work, a sample of oxide from the lower part of the purifier, worked for six months, gave 55·66 per cent. of total sulphur, of which 82·3 per cent. was free or available.

A new change valve has been introduced at the Richmond Gas Company's works, which is easy to lift and turn, and at no time is the foul gas main unsealed, so that no escape of hydrogen sulphide can take place during change. A drawing is given.

An elaborate investigation into the constitution of ammoniacal liquors has been conducted, the results of which are tabulated, and the methods of analysis used are described. High commendation is given to Mr. F. Lennard's continuous tar still, two units of plant of which deal daily with 16,000 and 24,000 galls. of tar respectively. The freedom from nuisance is said to be absolute, and "the pitch is delivered in a constant stream, which can be cooled to any desired degree before appearing in the open on the pitch bays." One plant is already at work on the Continent, and another is being erected in Scotland.

The Report consists of 118 pages, 11 of which are devoted to Scotland. No. 4 district has been divided into two portions: one, North and East Lancashire, under Mr. Porter; and the other (4A), East Midland, under Mr. Fletcher.
—E. S.

Percarbonate of Potassium: Its Preparation and Properties.
A. v. Hansen. Zeits. f. Elektrochem. 1897 3, 445—448.

The author (with E. J. Constam. This Journal, 1896, 815) has already given a description of the preparation of potassium percarbonate by electrolysing a concentrated solution of potassium carbonate at a low temperature. The clear solution in the neighbourhood of the anode becomes milky, owing to the separation of percarbonate, and by filtering off, a salt of sky-blue colour is obtained. This is potassium percarbonate, containing a few per cent. of potassium carbonate as an impurity. Further experiments have now been made to ascertain the best conditions for successful preparation. The electrolysis was carried out in a porous cell of 200 c.c. capacity, placed in a beaker of such size that the outer cell gave about 120 c.c. as anode space. The anode was a platinum spiral, the cathode of sheet nickel.

I. *Influence of Temperature.*—If the electrolysis is started at about $-15°$ C., small variations of temperature have but little effect on the yield, provided the anode liquid is kept concentrated. The temperature may even rise to 0° without much effect upon the reaction. But if the specific gravity of the potash solution fall below 1·52, the yield diminishes rapidly as the temperature rises. Thus the temperature should be kept low towards the end of the reaction, when the solution has become weaker owing to separation of percarbonate.

II. *Influence of Concentration.*—If the concentration of the anode liquid drop even slightly below the saturation point, little or no percarbonate is obtained, for the substance is extremely soluble in dilute solutions. Furthermore, in dilute solutions potassium bicarbonate is produced and forms the greater part of the crystalline precipitate obtained.

III. *Influence of Current Density.*—A high current-density is of the greatest importance, otherwise much bicarbonate is formed. Finally the author recommends that special care should be taken to ensure high current-density and concentration of the solution; the employment of temperatures as low as $-10°$ and below, is not necessary if the solution be always kept saturated. A saturated solution of pure potash at ordinary temperatures has a sp. gr. of 1·555 or 1·556, and it is a remarkable fact that such a solution exhibits no tendency to crystallise out when its temperature is lowered. On the other hand, a concentrated solution of commercial potash rapidly deposits crystals when it is cooled a few degrees below zero. In order to prevent the separation of crystals inside the porous cell, it is well to use as the cathode liquid a less concentrated solution. The method above described is not well adapted for preparing percarbonate in quantity: for this purpose the following modification is recommended.

The outer vessel is provided with an outflow tube at a level just below the rim of the porous cell. As the electrolysis proceeds, and in proportion to the amount of percarbonate formed, fresh solution is introduced into the lower part of the anode space by means of a funnel. The specific gravity of the electrolysed fluid is less than that of the fresh potash solution; hence the two do not mix. The solution containing the percarbonate rises and flows away through the outlet and is at once filtered. The yield per ampère-hour is from 2·2 to 2·4 grms. of solid salt containing 87 to 93 per cent. of percarbonate. After all adhering liquid is pumped away, the salt is spread on a porous plate and dried in a current of dry air. This works much more rapidly than drying in a desiccator. Towards the end of the operation the air can be heated to about 40°; above this, decomposition sets in. The salt thus prepared is an amorphous powder with a slightly blue colour. The pronounced blue colour of the moist salt disappears almost entirely on drying. It is easily decomposed, yielding up oxygen to oxidisable bodies. But it is not so unstable as was at first supposed. In order to decompose it in a short time by heating, a temperature of 200°—300° is requisite. On immersion in water it only slowly decomposes at ordinary temperatures. But at somewhat higher temperatures it rapidly decomposes with evolution of oxygen; in fact, it is a convenient laboratory source for preparing pure oxygen. The salt has only to be placed in water and warmed to about 45°; a very steady current of oxygen then comes off. The reaction goes on of itself without need of any further heating, and scarcely a trace of percarbonate is left. 100 grms. of the salt give about 5 litres of oxygen. In order to retain any carbon dioxide that may be developed, soda is added to the water.

Like the peroxides of sodium, barium, and hydrogen, it can be used as an oxidising agent. By adding it to diluted acids a more or less concentrated solution of hydrogen peroxide is obtained. If an excess of acid be present, the solution keeps well. The dry percarbonate keeps very well, and is a convenient source for rapidly preparing a solution of hydrogen peroxide. Potassium percarbonate cannot be crystallised from its aqueous solution. If water at 0° be saturated with it and then cooled to $-15°$, a rich crop of crystals separates out, but these consist mainly of ice. It is, however, only slightly soluble in alcohol; and by making use of this property it can be purified as follows. An excess of the salt is added to a strong solution of caustic alkali and is digested with it for some time at $-5°$ to $-10°$. The bicarbonate contained in the salt is decomposed by the alkali. The potash produced goes into solution, and, after filtering, we obtain percarbonate containing only a small quantity of caustic alkali as impurity. By treatment with absolute alcohol this is removed and a product containing 95 to 99 per cent. of percarbonate is obtained. The author

has not succeeded in preparing the sodium or ammonium salt of percarbonic acid.—D. E. J.

Barium Sulphocyanide from Spent [Gasworks] Oxide, Manufacture of. V. Hölbling. Zeits. angew. Chem. 1897, [10], 297.

If pure Prussian blue be boiled for a short time under a pressure of 2 or 3 atmospheres with a very large excess of barium sulphide, or with a small excess of barium sulphide and some free sulphur, it is converted into barium sulphocyanide according to the following reactions:—

$$Fe_4.3Fe_2(CN)_{12} + 12BaS = 6Ba_2Fe(CN)_6 + 8FeS + 4S.$$
$$2Ba_2Fe(CN)_6 + 2BaS + 12S = 6Ba(CNS)_2 + 2FeS.$$

As spent oxide naturally contains a large amount of sulphur, it is only necessary to add some 10 or 15 per cent. excess of barium sulphide and to boil it for half an hour at a pressure of 3 atmospheres in order to recover the whole of the blue as thiocyanate. The filtered liquid requires purification from barium sulphide; and this may be effected either by treatment with sulphurous or carbonic acid gas. In the former case the sulphide is chiefly converted into the insoluble thiosulphate of barium and free sulphur, a small quantity still remaining in solution as sulphite, which may be removed by decantation after the liquor has been evaporated till its specific gravity reaches 1·38. The barium thiosulphate is heated until it splits up into sulphide and sulphur, and it is then ready for use over again. If carbonic acid be employed to decompose the barium sulphide, the sulphuretted hydrogen is absorbed in the usual manner, the resulting carbonate converted into sulphate, and the latter ignited with coal to form fresh sulphide.

The purified sulphocyanide solution is evaporated to a density of 1·75 to 1·79 and allowed to crystallise, the yield being sufficiently pure for use by dyers without further treatment.—F. H. L.

Acetic Bacterium, A New. Zeidler. Zeits. für Spiritusind. 1897, 20, [20] and [21], 164 and 171—172.

See under XVII., *page* 692.

PATENTS.

A Still or Tank for the Distillation, Boiling, and Holding of Acids, Alkali, and other Corrosive Liquids. D. Bray, Cinderford, Gloucester. Eng. Pat. 16,877, July 30, 1896.

See under I., *page* 661.

Hydrochloric Acid, Impts. in the Manufacture of, Free from Arsenic. J. R. Wylde, J. W. Kynaston, and J. Brock, Liverpool. Eng. Pat. 17,607, Aug. 8, 1896.

Hydrochloric acid gas resulting from the treatment of sodium chloride with ordinary sulphuric acid containing arsenic, is taken through a length of pipes, and, with addition of a little chlorine, is passed through a "dry tower," charged with coke or the like. The temperature of the gases passing through the tower should not exceed 55° C., under which condition the small quantities of hydrochloric acid and steam which condense to liquid acid upon the coke, retain practically all the arsenic present, converted into the comparatively fixed condition of arsenic acid under the influence of the added chlorine. The arsenic-free gas issuing from the dry tower is passed through an ordinary condensing or "wet" tower, down which water is passed, to obtain the acid.

The gases issuing from the "decomposer" in Deacon's chlorine process, containing hydrochloric acid, chlorine, aqueous vapour and nitrogen, &c., are arsenical, and, after passage through cooling pipes, are taken successively through the dry and wet towers as described, to obtain a liquid acid free from arsenic. Any trace of chlorine remaining in the product, may be removed by passing air through the cold acid, or by addition of a deoxidising substance such as ferrous chloride.—E. S.

VIII.—GLASS, POTTERY, ENAMELS.

PATENTS.

Sulphate Glass, Manufacture of. K. W. R. Goerisch, Dresden, Germany. Eng. Pat. 14,901, July 6, 1896.

To the glass batch are added fats, oils, resins, ceresin, or paraffin. All these matters are said to contain in an eminent degree hydrogen, in a form capable of reducing the sulphate of the sodium sulphate in the glass batch, to sodium sulphite, which is then quickly decomposed by the silica.—E. A.

Enamelled Ceramics with a Slaggy Biscuit, covered over with Coloured Enamels, Manufacture of. H. De Wit, Emptinne, Belgium. Eng. Pat. 10,430, April 27, 1897.

Slag of any kind, whether of refining, moulding, or steel, will do. It is agglutinated by clay or plastic earth, so that the biscuit may contain 50 per cent. of silica, 25 per cent. of calcium carbonate, and 20 per cent. of alumina. The complete baking of the product may be accomplished in one or several operations.—E. A.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Blast-Furnace Slag that has been treated with Nitric Acid, as an Addition to Hydraulic Cement. A. D. Elbers. Eng. and Mining J. 1897, 63, 661.

A process has been patented (U.S. Pat. 579,820, March 30, 1897), with the object of removing the objectionable calcium sulphide from, and otherwise improving the blast-furnace slag to be used as a constituent of cement. The finely ground slag is moistened with a weak solution of nitric acid, more water added when the acid is neutralised, and the leached mass dried. The complete treatment requires about ½ lb. of 60 per cent. nitric acid and 30 lb. of water per 100 lb. of slag.

The author arrived at the following conclusions with regard to the efficiency of slag treated as above:—High-class Portland cements—those which have been ground from carefully prepared stock that has been burned very hard—can only be improved by the admixture of small quantities, say, not exceeding 10 per cent. on the average. Inferior Portland cements may be mixed with about their own weight of the above-mentioned slag, and still give as good results as the neat. Briquettes made up of cement, slag treated as described, and sand, give rather poor comparative results as regards their test for tensile strength, unless the sand is added after the cement and the slag has been thoroughly wet-mixed. It is suggested that a fairer test would be to slurry the mixtures, just as is done in working the cement for the "second set," and to fill the slurry into the moulds without heating it.

A briquette, made by casting the mixture into the mould, and composed of 90 per cent. of high-class Portland cement and 10 per cent. of the prepared slag, the water added being 30 per cent., broke when 17 days old at 350 lb. tensile strain per square inch.—A. S.

Building Materials, Testing. Mitt. k. t. Versuchsanst. zu Berlin, 15, [1], 80—85.

1. *Defective Flooring Tiles.*—The upper surface of the tiles consisted of chippings of white and yellow-brown marble, cemented together by a greyish-black cement, and the defect complained of was the appearance of a number of small bubble-like cavities in the mass, which, on examination, proved to be due to imperfections of manufacture, since they were not caused by the dislodgment of any of the particles of marble—as the fact of their underlying many of the latter demonstrated; nor were they the result of washing with acid, as was proved by the fact that both concentrated and dilute hydrochloric acid dissolved the marble much more rapidly than the cement.

2. *Badly Mixed Concrete.*—(*a*) A sample was submitted for examination on account of its having become

brittle and perished, although only 2—3 years old. The proportion of cement to sand and gravel was ascertained to be 1:10, and the composition of the former to be that of normal Portland cement. An examination of the ratio of sand to gravel, however, indicated that the latter was in such excess that the interstitial spaces were incompletely filled, and also that, assuming the deficiency to have been made good by the use of cement, the ratio of cement to sand and gravel should have been 1: 1·1 instead of 1:10, *i.e.*, that an increased quantity of sand ought to have been employed.

(*b.*) Defective concrete pipes formed the subject of another examination. The load required to break the pipes was found to be only 1,713 kilos., whereas other pipes of the same dimensions (105 cm. long, 50 cm. wide, and 6 cm. thick) required for that purpose, on the average, a load of 3,867 kilos. According to analysis, the ratio of cement to gravel was normal (1:3·45), but the latter contained carbonates, and when washed by sedimentation exhibited a large number of snail-shells, the water running away being very turbid and indicating impure materials. Other impurities present in large quantity were: moss, leaves, grass, shives, and more or less rotten wood, thus explaining the defective condition of the pipes.

3. *Unsuitability of Sugar-Works Sediment for Mortar Making.*—A specimen of waste lime from sugar works was found to be fully saturated with carbonic acid and organic acids, and although, when mixed with three parts by volume of building sand, the blocks of mortar exhibited a tensile strength of 1·64 kilos. per sq. cm., and a resistance to compression of 7·2 kilos. after 14 days—*i.e.*, about the same as normal mortar—the same tests applied the 24th day after moulding gave only 1·45 and 8·4 kilos. respectively, or a considerable inferiority; and, in fact, no better result than would ensue from the cohesion of earth or mould in drying.

—C. S.

Asphalt, Testing Artificial. M. Gary. Mitt. k. t. Versuchsanst. zu Berlin, **15**, [1], 74.

See under XXIII., *page* 701.

Boiler Incrustation by Lime washed out of Beton (Concrete), Formation of a Dangerous. Dingler's Polyt. J. **305**, [4], 96.

See under I., *page* 660.

PATENTS.

Streets, Roads, Pavements, and the like, and on the Surfaces of Wood and Metal; An Improved Composition for Use on. S. P. Davies, London. From G. Frederick, Sydney, N.S.W. Eng. Pat. 15,316, July 10, 1896.

Coal-tar or pitch is heated for 2—4 hours at 350° F., till the specific gravity is about 1·261, when an equal quantity of "retorted shale," or shale treated in a retort, or ash of shale or coal in fine powder, is added slowly. The mixture is applied to the surfaces to be covered in the usual way.

—O. H.

Hardening Stone, An Improved Process for. T. R. Jones, Kensington, London. Eng. Pat. 16,892, July 30, 1896.

The process consists in placing the stone in a vacuum, and thus exhausting the air contained therein, and, whilst in the vacuum, introducing a solution of zinc sulphate, which readily penetrates the stone. The sulphate of zinc is said to combine with any lime present, and to harden the stone to the extent of the penetration.—E. A.

Rendering Substances [Wood, Woven Fabrics, &c.] Incombustible, Impts. in. A. Issel, Genoa, Italy. Eng. Pat. 11,368, May 7, 1897.

The wood or other material to be rendered incombustible, is thoroughly dried, and then kept for many hours in a boiling solution of stated proportions of boric acid and potash alum; or in the case of resinous woods, of boric acid, oxalic acid, and potassium carbonate. The solution for pasteboard contains boric acid, potash alum, and glue. The treated material is well dried.—E. S.

X.—METALLURGY.

Structural Steel: Relation of Tensile Strength to Composition. Engineer, **84**, July 23, 1897, 81.

A simple and easily remembered rule for ascertaining the relation of tensile strength to the composition of structural steel was recently brought to the notice of the American Society of Civil Engineers by Mr. A. C. Cunningham. It is as follows:—To find the approximate tensile strength of structural steel, to a base of 40,000 lb., add 1,000 lb. for every 0·01 per cent. of carbon, and 1,000 lb. for every 0·01 per cent. of phosphorus, neglecting all other elements in normal steels. Radical variations between calculated and actual strength indicate mixed steels, segregation, incorrect analyses, or unusual treatment in manufacture.

Silver-Copper Alloys. F. Osmond. Comptes Rend. **124**, 1234—1237.

In a former note the author stated that the microscopic appearance of silver-copper alloys, save those containing a very minute proportion of one ingredient, suggested a mixture of two components, apparently the metals themselves. The fact that the physical properties of the alloys, however, are far from being the mean of those of the metals throws doubt on this; and microscopic examination of alloys of known composition, in which a camera lucida drawing was made of a section of each button after heating to an annealing temperature to tint the copper, and the relative amounts of the two visible constituents estimated by measuring their areas on the drawing, showed that the accessory metal (the alloys went up to 4 per cent. of either metal) was always visible in much less than its true proportion. Careful microscopic comparison of these alloys with the pure metals showed that only after about 1 per cent. of silver (or copper, as the case may be) is reached, does the metal actually begin to separate out on cooling; whence the author supposes silver and copper to be mutually soluble to a limited extent.

Polished plane surfaces of silver and copper brought into contact, and heated in hydrogen for an hour to 650°—675° C., adhered so strongly, that the fingers could not separate them, and the surfaces after separation showed to some extent the characteristics of the silver-copper and copper-silver alloys.—J. T. D.

Aluminium, The Smelting of. Chem. Zeit. **21**, [42], 415.

In aluminium castings, a distinction must be drawn between castings which are practically finished on leaving the mould and castings which are subsequently subjected to a forging or rolling process. The difficulty of producing sound aluminium castings, lies in the great contraction of the metal on cooling. This difficulty can be overcome by adding phosphorus to the molten metal, in the proportion of about 1 grm. to 1 kilo. Thoroughly sound castings are in this manner obtained, but they are rather brittle and therefore unfit for rolling purposes. Castings suitable for the last-named process are obtained by pouring some rape oil upon the surface of the metal, which is fused in iron crucibles. When all the oil is burnt off, the metal is run into the hot iron moulds, and as soon as the contraction begins, more metal is added. The ingots obtained in this manner are excellent for rolling, forging, or wire-drawing purposes. Aluminium fused in graphite crucibles always has a tendency to crack in the rolling or forging, the more so the oftener it has undergone the process of melting in this kind of crucible. This appears to be due to the fact that aluminium takes up silicon from the crucible.

—C. O. W.

Tin in Tin-Plate, Estimation of. H. Mastbaum. Zeits. angew. Chem. 1897, [11], 329.

See under XXIII., *page* 700.

Aluminium Utensils, Analysis of. M. Balland. Comptes Rend. **124**, 1313.

See under XXIII., *page* 699.

Arsenic in Copper, Notes on the Estimation of. G. L. Heath. Eng. and Mining J. 1897, 63, 663.

See under XXIII., *page* 699.

PATENTS.

Iron and other Metals, A New Process and Apparatus for the Direct Production of, from their Ores. A. Karyscheff and S. Demmenic, St. Petersburg. Eng. Pat. 6654, March 26, 1896.

The ores are brought into the condition of pyrochemical solutions, which are then subjected to the separate or combined action of heat, chemical reagents, or electricity. The method is applicable to all metals and alloys.

For the production of iron, the charge is so arranged as to produce easily fusible slags that will readily dissolve oxide of iron, and particularly pyroxenes such as hedenbergite, diopside, or augite. If the ore contain phosphorus, dolomite is added. When compounds resembling libenite and phosphorocalcite are formed with a proper preparation of the charge, it is easy to convert all deleterious admixtures into slags, and to obtain metal of excellent qualities.

In some cases the ore is dissolved in, and the metal separates out from, these fused slags by the agency of heat alone; in others, a metallic compound, rich in combined carbon, is added, and, in addition, electricity of high tension may be employed.

Any kind of furnace may be used, but preference is given to those constructed on the regenerative system and heated by producer-gas, water-gas, or acetylene; oxygen, or air rich in oxygen, is also employed in some cases to produce a higher temperature.—J. H. C.

Ores [Iron], Impts. in the Treatment of, for the Production of Iron, Steel, and other Metals. R. F. Strong, London. Eng. Pat. 24,657, Nov. 4, 1896.

In order to bind together friable ores and to enable them to be smelted more successfully, the crushed ore is mixed with about 5 per cent. of crushed lime, and 10 per cent. of tarry pyroligneous acid, pressed into blocks, and conveyed direct to the furnace. Carbonaceous material and fluxes may also be added. Or a larger quantity (20—30 per cent.) of pyroligneous acid may be used if it is intended to serve as a fuel.—W. G. M.

Iron, Apparatus for the Preliminary Fining of. R. M. Daelen, Düsseldorf, Germany. Eng. Pat. 10,989, May 3, 1897.

A trough-shaped side-blowing Bessemer converter is mounted on trunnions and supported on rollers carried by a truck-frame running on rails. The air is passed through one trunnion (connection being made by a trumpet-mouthed delivery pipe) to the blast box, on one side of the converter; two rows of tuyères are superposed, but only one row is used at a time, the others being plugged. This allows for a fairly constant depth of blast delivery, in spite of variations in the capacity of the converter owing to the wear of the lining. Above the blast box is a hopper with a simply regulated valve for the admission of iron ore to be injected by the blast. On the middle of the side of the converter, opposite the tuyères, is a spout, through which the melted iron is charged and discharged. The usual rotating mechanism is attached to the free trunnion. By adopting this form of converter, the use of an intermediary ladle between the fining furnace and the open hearth is dispensed with; the air may be introduced quite close beneath the surface of the bath, and the apparatus, with a maximum width of 1 to 1·5 metres, may contain from 10 to 20 tons of crude iron; the apparatus is in equilibrium as far as possible during rotation; the distance from the charging opening to the bottom point of the hearth is relatively very small, although the usual hood is provided, to prevent splashing during the blow; and the discharge spout may be lowered to within 800 mm. of the line of rails in front of the open-hearth furnace, so that the latter may be charged direct.—W. G. M.

Alloys, Impts. in the Manufacture of. A. G. Street, Paris. Eng. Pat. 12,467, June 6, 1896.

The object of this invention is to obtain alloys of chromium, molybdenum, tungsten, uranium, and other easily oxidisable metals having a high melting point, with other metals, such as aluminium, having comparatively low fusing points.

The "infusible" metal or alloy is mixed or covered with melted cryolite and borax or other suitable flux, and then dropped into the fused metal; or the fused metal is poured over the mixture of "infusible metal and flux."—J. H. C.

Alloy, New Metallic. [*Al, Sn, Zn.*] G. Duceau, Toulouse. Eng. Pat. 15,699, July 15, 1896.

For ordinary parts of machinery the alloy is composed of: aluminium, 30; tin, 20; and zinc, 50 parts.

For parts subject to friction: aluminium, 15; tin, 10; and zinc, 75 parts.

In some cases a little copper is added.—J. H. C.

Alloy, Improved Metallic. W. T. Sugg, Westminster. Eng. Pat. 16,831, July 29, 1896.

An alloy which will not be discoloured or otherwise deleteriously affected by the action of acids or other products resulting from cooking operations, may be made by fusing 10 parts of aluminium with 1 of silver, and adding the resulting alloy to 89 parts of molten copper. The alloy is hard, tough, and homogeneous, and may be rolled or hammered.—W. G. M.

Chromium and similar Metals, Impts. relating to the Fusing of, and to the Manufacture of Alloys of such Metals. E. A. G. Street, Paris. Eng. Pat. 12,396, June 8, 1896.

The crucibles are lined with a fused mass composed of borax, cryolite, chloride of sodium, or fluoride of calcium, or a mixture of these bodies, and the oxide of the corresponding metal.—J. H. C.

Ores, Gold and Silver; Processes and Apparatus for Classifying, Amalgamating, and Cyaniding. J. Garnier, Paris. Eng. Pat. 14,493, June 30, 1896. (Under Internat. Convention.)

The finely divided ore is placed in a large cylindrical iron tank provided with a perforated false bottom, which is arranged to act as a piston with the aid of a subsidiary piston and cylinder placed beneath the apparatus. The false bottom is covered with coarse, heavy particles of gold ore, or of iron or lead, to act as a filter; and the cylinder is loosely covered with a plate from which depend strips of amalgamated copper. Beneath the false bottom is a lateral connecting tube communicating with a second smaller cylinder, having a closely fitting cover carrying a rod with amalgamated copper plates attached, dipping almost to the bottom of the vessel. At the top are two side tubes with stop cocks, one above the other, the upper one communicating with the air, the lower with a steam generator. Water being introduced first into the two cylinders, the ore is introduced into that which has the false bottom, the air-escape cock in the other cylinder being left open. This is at length closed, and steam is admitted through the lower one, until the mass of ore and water in the first cylinder has been raised to some extent; the steam is then shut off, and the ore subsides, becoming partially classified in the process; the operation is then repeated several times. Finally the water, with slimes in suspension, is expelled into a second apparatus by means of the piston. A part of the gold is caught on the plates and a part is dissolved in the liquid, if cyanide solution be employed instead of water. The apparatus for treating the slimes is somewhat similar, and consists of two cylinders, connected by an inclined tube. In the first a solid piston is used, and this carries an upright with amalgamated plates attached; the second cylinder is like that employed for treating the original ore, and the process is similarly conducted. The gold is in part caught by the amalgamated plates, and in part is dissolved by the cyanide liquors.—W. G. M.

Annealing Metals, A Method or Process for. L. Doig, London. Eng. Pat. 15,041, July 7, 1896.

The metal is heated in a closed vessel, together with carbonaceous or other gas-producing material, such as oil or sawdust. The vessel is provided with a pipe for the escape or admission of gas, which can be opened or closed at pleasure.—J. H. C.

Lead, Oxide of; Impt. in the Manufacture of, and Means used therefor. A. Gutensohn, London. Eng. Pat. 16,275, July 22, 1896.

Three pots are placed in cascade form, the upper two are connected near the bottom by a sloping pipe provided with a stopcock, and both are set above grates. The lower of the two has a circumferential gutter-like trough with an overflow into the third pot. The lead is melted and brought to a red heat in the uppermost pot, and is run down as required into the middle crucible, where it is subjected to the action of oxygen, or of air enriched with oxygen, which is either injected or blown upon the surface. A hood connected with a condensing plant is arranged over this crucible for the recovery of fumes. The oxide of lead formed, overflows into the gutter, and thence into the third pot at once, before it has time to exert any serious influence upon the material of the crucible.—W. G. M.

Lead Sulphide, Ores of; Impts. in the Treatment of, preparatory to Smelting the same, and incidentally in obtaining Sulphurous Acid. G. G. M. Hardingham, London. From T. Huntington and F. Heberlein, both of Pertusola, Italy. Eng. Pat. 3795, Feb. 12, 1897.

The process is directed to the conversion of galena into lead oxide. The ore is mixed with sufficient lime or other alkaline earth to react with the sulphur present, and the mixture is heated to bright redness (about 700° C.) in a reverberatory or other furnace. It is then cooled to a dull red heat (about 500° C.) and transferred to a vessel in which it is exposed to a current of air, which may be preheated, and which is introduced by means of a blower. Under these conditions rapid oxidation takes place, sulphur dioxide is evolved (and may be utilised); much heat is generated, and the mixture fuses and gradually settles down to a mass of oxide of lead with the gangue stuff of the ore. The oxide of lead, containing calcium sulphate and gangue stuff, may then be smelted in a blast furnace. It is believed that the reactions taking place are as follows :—

(1.) At 700° C. .... $CaO + O = CaO_2$.
(2.) At 500° C. ...... $4CaO_2 + PbS = 4CaO + PbSO_4$.
(3.) At fusing point. $PbS + PbSO_4 + O_2 = 2PbO + 2SO_2$.

Ferrous or manganous oxide may be substituted for lime in the process.—W. G. M.

Ores containing Zinc, Impts. in the Treatment of Solutions of, for the Recovery of Zinc as Oxide. A. M. Clark, London. From E. A. Ashcroft, of Newcastle, New South Wales. Eng. Pat. 16,312, July 23, 1896.

Zinc sulphate is converted into a paste by stirring in zinc oxide, which is strongly heated so as to drive off sulphuric or sulphurous acid gases. In some cases the pasty mass is mixed with carbon before the heating.—J. H. C.

Separation and Collection of Solid Matters [Dust of Ores, &c.] held in Suspension in Aëriform Fluids; Improved means applicable for use in the. F. D. Cummer, Cleveland, U.S.A. Eng. Pat. 17,105, Aug. 1, 1896.

This invention relates to dust or settling chambers, and the process consists in blowing the air or gas through a pipe fitted with a deflecting screen into a brick chamber, where the greater part of the dust is said to be deposited, the lighter portions passing through openings into a superposed chamber provided with apparatus for spraying liquids or vapours, and with cooling appliances, after which the air or gas is allowed to escape.—O. H.

Ores, Zinciferous Sulphide; An Improved Process for the Treatment of. E. F. Turner, Adelaide, South Australia. Eng. Pat. 17,299, Aug. 5, 1896.

The crushed ore is roasted, either alone or with carbon, in the presence of a current of superheated steam. In this way the ore is oxidised and hydrogen sulphide is evolved when it is treated alone, or hydrogen sulphide and carbon monoxide are produced when carbon is added. The gas is collected in a gas-holder and used as a fuel, and the products of combustion are employed to treat the oxidised ore in towers, where the roasted ore, accompanied by water containing salt, is allowed to fall and so to meet an upward current of sulphur dioxide. In this way the zinc oxide is converted into sulphite, and may be afterwards leached out together with any copper that may be present. Lead oxide is partly converted into sulphite, and silver sulphite into chloride. The liquor from the zinc lixiviating vats is passed over copper to remove any silver, and then over zinc turnings to recover any copper and lead; it is then conveyed to towers, through which hydrogen sulphide (obtained as above described) is passed. Zinc sulphide and sulphur are thus precipitated. The gangue material in the vats is heated, whereby lead sulphite is converted into lead sulphate and sulphide, and these, being in contact with unchanged lead oxide, yield a soft lead. If the gangue material contain silver, it is mixed with zinc chloride, with or without metallic zinc, and heated to obtain the lead in the soft metallic state, whilst the chlorides of silver, gold, and other metals remain with the zinc chloride, and may be separated by lixiviation or otherwise.—W. G. M.

Deoxidising Furnaces [Deoxidising Granular Oxide of Iron, &c.], Impts. in. H. A. Jones, Brooklyn. Eng. Pat. 18,259, Aug. 18, 1896.

The furnaces are made up of earthenware blocks fitted together, and secured by means of projecting flanges, thus forming a series of retorts, which are built into brickwork flues, the whole forming a zig-zag arrangement, which is heated by means of Bunsen burners or by liquid fuel. The retorts are supplied with hoppers, mixers, slides, cocks, and air pipes as may be required.

The claim is for the combination of these various components in the manner specified, for use as deoxidising furnaces for deoxidising granular oxide of iron and converting it into cast iron.—J. H. C.

Aluminium, Impts. in Processes for Reducing. F. A. Gooch, New Haven, U.S.A. Eng. Pat. 6151, March 9, 1897.

Sodium fluoride or potassium fluoride is fused with alumina and a haloid compound of aluminium, and an electric current is passed through the fused mass, further quantities of the alumina or haloid compound being added from time to time.—J. H. C.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(A.)—ELECTRO-CHEMISTRY.

Carbon, Electro-chemical Equivalent of. A. Coehn. Zeits. f. Elektrochem. 1897, 3, 424—425.

Carbon anodes, in electrolytes which evolve oxygen at the positive pole, are rapidly destroyed. This is not simply a case of disaggregation—a chemical change takes place as well. The author has examined whether the electrolytic action can be regarded as coming within the scope of Faraday's law. He has endeavoured to determine the electro-chemical equivalent of carbon from the loss of weight of a carbon anode. The electrolyte consisted of equal volumes of sulphuric acid and water, and the electrolysis was carried on at a temperature such as to favour solution of the carbon and not disaggregation. The current was sent at the same time through two copper voltameters. The anodes were arc-lamp carbons 6 cm. long, 1 cm. in diameter, carefully boiled and dried at 200° beforehand. After electrolysis they persistently retained the brown-coloured solution, and required washing for several days. The most probable theoretical value of the electro-chemical equivalent of carbon would be $12/4 = 3$. The first determination quoted gave the value 3·5. This somewhat larger value was probably due to a partial disaggregation of the carbon. Preliminary experiments had shown that this occurred in

concentrated solutions without the electrolyte becoming in any way coloured; in more dilute solutions, however, the colour became a deeper and deeper brown. The new determinations were made with solutions containing from 10 to 500 volumes of water to 1 volume of sulphuric acid. The electrolysis lasted from 36 to 120 hours. The liquid was then filtered and the separated carbon particles washed and dried. Their weight was deducted from the loss of weight of the carbon anode. These determinations gave values varying from 2·7 to 3·0. Thus it appears that if we take the electro-chemical equivalent of hydrogen as H = 1, that of carbon is C = 3.—D. E. J.

Percarbonate of Potassium: Its Preparation and Properties. A. v. Hansen. Zeits. f. Elektrochem. **3**, 1897, 445.

See under VII., *page* 679.

PATENTS.

Electrical Apparatus for the Treatment [Electrification] of Air, Gases, and Vapours, Improved. A. E. Bonna, A. le Royer, and P. van Bercham, Geneva. Eng. Pat. 13,688, June 20, 1896.

Apparatus is shown for electrifying air, gases, and vapours "for certain chemical purposes" not stated.—J. C. R.

Electrolytic Apparatus [Pt Wire Electrodes], Impts. in. C. Kellner, Vienna. Eng. Pat. 16,057, July 20, 1896.

Relates to methods for obtaining high current densities with electrodes of platinum wire secured to supports of dielectric material.—J. C. R.

(*B.*)—ELECTRO-METALLURGY.

Lead: Method of Treatment, rendering it more easily attacked by Chemicals in Preparation of White Lead, Accumulator Plates, &c. J. Walter. Zeits. f. Elektrochem. **3**, 1897, 449—450.

A Ger. Pat. specification by the author, dated June 21, 1887, is quoted. The lead is alloyed with easily oxidisable metals, such as sodium, potassium, magnesium, aluminium, calcium, barium, strontium, manganese, or chromium. The metal alloyed with the lead is converted into oxide by exposure to air or removed by suitable reagents, leaving the lead in a pulverulent form well adapted for such processes as the "formation" of accumulator plates. The alloys with sodium and potassium are especially suitable. For the purposes under consideration the volume ratios, and not the mass ratios, are the determining factors in the composition and behaviour. As lead is ten times as heavy as sodium, 1 per cent. of the latter by weight corresponds to 10 per cent. by volume. The alloys are easily oxidisable, and can be cast on to any lugs or other conductors. The alloy removes any coating of oxide on the latter, and so makes a good electrical connection with it.—D. E. J.

PATENTS.

Ores containing Zinc, Impts. in the Treatment of. A. M. and W. Clark, London. From E. A. Ashcroft, Melbourne, Victoria. Eng. Pat. 11,076, May 21, 1896.

This process was devised to enable zinc blende (alone or mixed with galena) to be so treated as to yield directly an electrolyte from which the zinc may be deposited electrolytically without formation of spongy deposits. This may be accomplished by having oxy salts of zinc in the solution from which the zinc is being separated. The ore is first roasted, to convert the zinc sulphide as completely as possible into oxide or sulphate. A portion of the roasted ore is then leached with aqueous ferric chloride or sulphate solution according to the process described in the amended specification of Eng. Pat. 13,850 of 1894, or is leached according to Eng. Pat. 13,534 of 1895 (this Journal, 1895, 658). Further portions of the roasted ore are finely crushed, mixed with finely divided carbon, and subjected to an even temperature of 650° C. (as nearly as possible) for about two hours. The zinc sulphate present thus becomes oxide ($ZnSO_4 + C = ZnO + SO_2 + CO$). Portions of the resulting zinc oxide are then added to the solution previously described, which should be heated in order to increase its capacity for dissolving the oxide. The resulting electrolyte is rendered available for continuous use by introducing, as required, further portions of the specially roasted product. If desired, portions of the ore may be treated directly without roasting, or roasting and acid treatment may be applied conjointly. When galena is present or the blende is impure, settling boxes should be placed in the path of the liquors to the electrolysis vats, to remove suspended matter. If the process is to be applied to the continuous deposition of zinc, the proportion of roasted ore to be leached will be small as compared with that converted into oxide; but if it be desired to prepare a quantity of the electrolyte the converse will be the case. —W. G. M.

Porous Metals by Electrolysis, An Improved Method for Obtaining. L. Hoepfner, Berlin. Eng. Pat. 17,671, Aug. 10, 1896.

(1.) "A process for obtaining porous metals by electrolysis, mainly consisting in producing, alternately, spongy or porous, and dense metal precipitates, one operation serving to form the pores, and the next, to solidify or strengthen the walls of such pores."

Other claims relate to varying such conditions as the qualitative composition of the bath, the concentration, temperature, pressure on and motion of the electrolyte, and the density of the current; a solution of oxide of lead in caustic alkalis as electrolyte; the employment, as anodes, of impure metals with a view to their refinement; utilisation of the oxides forming at such anodes in obtaining by-products; and employment of porous metals obtained by the process in the preparation of plates for electrodes of secondary batteries.—J. C. R.

XII.—FATS, OILS, AND SOAP.

Castor Oil, Notes on. H. Meyer. Zeits. angew. Chem. 1897, 297—295; Chem. Centr.-Blatt, 1897, [1], 1229.

The author refers to the work of Juillard (Bull. Soc. Chim. **13** [3], 238), and states that the formation of complex polyricinoleic acids, resembling esters, takes place gradually at the ordinary temperature, without the use of condensing agents. A preparation of pure ricinoleic acid, 8 years old, showed on titration 30—40 per cent. less acidity than the freshly-prepared acid. These poly-acids are completely saponified, *i.e.*, reconverted into ricinoleic acid, in 24 hours, by warm alcoholic potash.

On heating ricinoleic acid and glycerin to 280°—300° C., passing in CO_2, and then washing with water to separate the excess of glycerin, the nearly chemically pure triglyceride of ricinoleic acid was obtained, in the form of a nearly colourless, neutral, active purgative oil, similar to natural castor oil. The oil was soluble in 96 per cent. alcohol and in methyl alcohol, had a specific gravity of 0·959—0·984 and $[\alpha]_D + 5·16°$. It does not form, like natural castor oil, a solid ricinelaidin on treating with nitrous acid. On preserving for a year, the molecule of ricinoleic acid triglyceride becomes doubled or tripled, the specific gravity being increased (0·988—1·009), and the iodine number reduced (from 71—84 to 44—57). The author states that the oil obtained by Juillard, said to be a mixture of tri- and diricinolein, must have been the nearly pure diglyceride of ricinoleic acid.—A. S.

Chinese Oil Tree. U.S. Cons. Reps., Aug. 1897, 477—483. (See this Journal, 1897, 195.)

The wood-oil tree, or *Aleurites Cordata*, belongs to a family very common in China, known as the "tung." The wood-oil tree is the ying tzu tung, so called from the shape of its fruit—ying means a jar. It is also known as the yiu tung or oil tung. It is found chiefly in Hunan, Hupeh, and Szechuen. The seeds are large and poisonous, and it is from them that the oil is expressed. The fruit is gathered in August and September. Hankow is the chief place of export. In the first three quarters of 1896, 188,556 piculs (1 picul = 133⅓ lb.) of this oil were exported.

The oil is expressed from the seeds as follows :—

The nuts are gathered and dried, put in a shallow iron basin about 2 feet in diameter, and stirred about over a good fire until parched ; they are then ground to a fine powder, the oil dripping out into a receptacle. When allowed to settle, and strained through coarse grass cloth, the oil is ready for the market. It is sent down in plaited baskets, lined with putty and varnished paper, with large wide mouths. It is usually of a light colour, somewhat resembling linseed oil, and emits a nauseous odour.

This oil is largely used for painting and calking. Mixed with sifted lime it makes excellent glaziers' putty. After the oil is removed, the nut is calcined or burnt, and produces a very valuable soot, from which Chinese ink is made (or India ink, so called). Cement, or chunam, for seams of boats, is made from this oil. It costs about 90 cash (6 cents) per pint in the village where produced, and is sold at double that price in Hankow.

The oil is extremely poisonous when fresh. A simple native remedy (more effective, indeed, than any foreign one) is to boil a quantity of pine shavings in water and bathe the poisoned part repeatedly. It gives immediate relief, and rapidly cures. The curious aspects of wood-oil poisoning are worthy of special study.

The best wood-oil comes to Hankow from Shin-chau Fu, in Hunan. The oil is given as a remedy in insanity and in cases of metallic poisoning. It is emetic, acro-narcotic, and drastic, proving destructive to rats in a very short time. It is applied as a stimulant to carbuncles, ulcers, burns, swellings, and bruises, and is a constant ingredient in native plasters. It was forbidden to be exported during the Taiping rebellion, as it is a necessary article for shipyards.

Chinese Insect White Wax. U.S. Cons. Reps., Aug. 1897, 484—190.

Since the introduction of kerosine oil into China, and its almost universal use in the remotest provinces of the Empire, the demand for white wax has declined considerably, and the supply has decreased in a corresponding ratio. The value, like the demand, has also declined. Not many years ago it was quoted at double the prices realized at present.

Various uses are ascribed to this wax, but in western China its sole use is for coating the exteriors of animal and vegetable tallow candles and for giving a greater consistency to these tallows before they are manufactured into candles. Insect white wax melts at 160° F., whereas animal tallow melts at about 95° F. Vegetable and animal tallow candles are therefore dipped into melted white wax ; a coating is given to them, and prevents them guttering when lighted. It is also said to be used in other parts of China as a sizing for paper and cotton goods, for imparting a gloss to silk, and as a furniture polish. Chemists are likewise declared to utilise it for coating their pills. In the Fukien and Chêkiang provinces it is employed to impart a polish to steatite, or soapstone, ornaments after the carving is completed.

"Mineral Soap." W. C. Knight. Eng. and Mining J. 1897, **63**, 600.

See under XXIV., *page* 705.

Woolcombers' Suds, The Treatment of. Public Health J., July 14, 1897, 219.

See under XVIII. B, *page* 694.

Low Iodine Number of Linseed Cake, Cause of the. G. Fassbender and J. Kern. Zeits. angew. Chem. 1897, [11], 331.

See under XXIII., *page* 702.

Beeswax, The Iodine Value of. R. Glode Guyer. Pharm. J. 1897, **54**, 308.

See under XXIII., *page* 702.

PATENTS.

Solid Paste or Soap Compound for Dyeing all kinds of Textile Materials, Manufacture of a. Carl Uffelmann, Kassel, Germany. Eng. Pat. 17,382, Aug. 6, 1896.

See under VI., *page* 677.

Washing Substance, New, and Process for Making it. A. Schuchards, Mülheim, and T. Otto, Düsseldorf, Germany. Eng. Pat. 14,303, June 27, 1896.

A washing substance consisting of a soap mass obtained from ground-soap and filling mass, with 1 to 1½ times the weight of "dry soda," forming a mixture "of cheesy consistency."—C. A. M.

XIII.—PIGMENTS, PAINTS ; RESINS, VARNISHES ; INDIA-RUBBER, Etc.

(A.)—PIGMENTS, PAINTS, &c.

Driers, Solid or Liquid. H. Amsel. Zeits. angew. Chem. 1897, 303 and 344.

The opening part of this article consists chiefly of a discussion of the results and opinions recorded by Weger (this Journal, 1896, 728). Weger appears to doubt whether soluble resin driers are ever adulterated with free resin (colophony) ; such is the case, however, and although perhaps it is not a matter of great moment if 10 or 20 per cent. of uncombined resin exist in a substance which is added only in amounts of 1·5 to 2·0 per cent. of varnish, yet when the drier itself has to be examined, if part of the resin be in a free state, the sample cannot be returned as a pure resinate.

Combining together some of the suggestions made by Weger with the processes he has already published, the present author puts forward a scheme for the analysis of the solid soluble driers, which enables their constitution and the presence of free basic or acid matter to be recognised, and which it is believed will throw some light on their actual industrial value. He determines (1) the solubility in chloroform, (2) in alcohol, (3) combined and free mineral matter, (4) acid number, (5) saponification number, (6) reaction with ammonia, and (7) moisture. (2) is returned not only as a percentage, but also as the amount taken up by 100 parts of alcohol in the cold. The combined mineral matter is estimated by deducting the quantity insoluble in chloroform from the total inorganic substances ; it is not advisable to obtain this figure simply by weighing the ash of the soluble portion; but, after a preliminary qualitative examination, each ingredient should be separately determined in the ordinary way. The titration for (4) is carried out both with the material partly dissolved in alcohol, and also completely dissolved in chloroform, the latter process naturally giving higher results. It should be noticed that considerable insight into the nature of the acids present in the sample can be gained by observing its behaviour during titration; for in the case of linoleates the phenolphthalein is temporarily reddened before shaking almost by the first drops of alkali, while with resinates the colour only appears shortly before actual neutrality is reached. (6) depends on the fact that free resin is saponified by shaking in the cold with 1 per cent. ammonia; so that if a pure resinate be treated in this manner and the liquid filtered, it should pass through the paper readily, and the filtrate should only give a slight cloudiness on dilution or acidification. Stronger solutions of ammonia are useless, and even the different results shown in the table may possibly be due solely to variations in the quality of the original resin. The moisture (7) is estimated at 90° or 95° C. Illustrative tables are given.—F. H. L.

Lead, Method of Treatment rendering it more easily Attacked by Chemicals in Preparation of White Lead, Accumulator Plates, &c. J. Walter. Zeits. f. Elektrochem, **3**, 1897, 449.

See under XI. B., *page* 684.

PATENTS.

Paint [Black, for Ships' Bottoms], and in the Method of Producing and Applying same, Impts. in. H. W. Aird and C. E. Felch, Montreal. Eng. Pat. 17,078, Aug. 1, 1896.

The vehicle consists of 30 galls. of linseed oil, 60 galls. of turpentine, 20 lb. of litharge, 5 lb. of manganese oxide,

and 100 of kauri-copal. 100 lb. of this are ground with 110 of Canadian graphite, and the whole thinned with 10 galls. of turpentine and 1 gall. of oil. Canadian graphite has, for this purpose, the advantage of containing some 50 per cent. of a "disintegrating substance" resembling felspar; but if the Ceylon product is employed in the manufacture of the paint, a certain quantity of felspar should be added. According to the purpose for which the composition is to be used, it may be diluted with black japan and (or) turpentine—the latter giving the smoothest but least durable coating. It is specially adapted for ships' bottoms. —F. H. L.

Branding Sheep, Fabrics such as Wool Bales and the like. An Improved Composition for; and Applicable as a Damp-resisting Paint. A. L. Potter, St. Kilda. Victoria. Eng. Pat. 11,013, May 3, 1897.

The object of this invention is to provide a material capable of withstanding rain and heat, which can yet be removed by treatment with soda and water, and at the same time will not injure the wool or surface to which it is applied. 3 lb. of glue, $\frac{1}{2}$ to 1 oz. of potassium bichromate, 3 lb. of lamp black or any other pigment, and $\frac{1}{4}$ pint of turpentine are boiled in 1 gall. of water, some Spanish clay being added if desired to increase the tenacity. If necessary the mixture may be thinned with hot water.—F. H. L.

(B.)—RESINS, VARNISHES.

Linseed Oil [for Varnish Making], Valuation of. W. Lippert. Zeits. angew. Chem. 1897, [10], 306.

See under XXIII., *page* 702.

Asafœtida, Composition of. J. Polásek. Archiv. der Pharm. 1897, [2]; through Pharm. Zeit. 1897, **42**, 240.

See under XX., *page* 697.

(C.)—INDIA-RUBBER, &c.

India-Rubber Industry, Impts. in the. A. Henriques. Chem. Zeit. **21**, 415.

Within the last few years nothing of any considerable interest has appeared either regarding the chemistry of india-rubber or concerning the general manufacturing methods employed in the india-rubber industry. But our botanical and statistical information as regards india-rubber plants and commercial brands of crude india-rubber has been not inconsiderably added to. Manoel V. Coutinho describes a new smoking process for Parà rubber, in which the smoking is carried out in a horizontally revolving cylinder, 5 ft. 2 in. in circumference, into which is passed the smoke of burning Urucary nuts (*Athalea excelsa*) or Inajá nuts (*Maximiana regia*). East Indian rubber exports keep decreasing in quantity owing to the reckless exploitation of the ficus forests. Little is heard concerning the india-rubber plantations started at Ceylon, Madras, Trinidad, Togo, and Cameroon, but the export of india-rubber from all parts of Africa is rapidly expanding. According to K. Schumann, the African rubbers are obtained from 14 species of the genus Landolphia. A. Dewèvre in this respect enumerates 21 species of which landolphia kirkii, madagascariensis, owariensis, Petersiana, lucida, Senegalensis, and tomentosa produce the best brands, whereas the very common L. comorensis var. florida, which formerly was considered the most important of African rubber-bearing plants, is now stated to yield a useless, resinous product only. Of African rubber trees, only Ficus Vogelii and F. Kolstii were known, but since 1894 ever increasing exports from Lagos took place of an india-rubber obtained from a tree called Ire (Ere) by the natives, and which is botanically described as Kickxia Africana Benth. The exports from Lagos in 1895 amounted to 5,069,504 lb., to the value of 269,892*l*. It is stated that the same tree also occurs on the Gold Coast, Togo, and Cameroon. It remains, however, to be seen whether this boom does not result in a speedy annihilation of the rubber trees. The output of india-rubber in the Congo basin has also considerably increased. Concerning the chemistry of india-rubber, C. O. Weber investigated the action thereon of bromide and iodide of sulphur. Henriques confirms his former statement that pure india-rubber, on being heated, protected from light, absorbs traces only of oxygen, whereas, after vulcanisation, the same samples absorb oxygen very freely, especially after they have been deprived of their free sulphur. J. Altschul has thrown some light upon the nature of the india-rubber substitutes, by showing that the amount of chloride of sulphur required for the conversion of an unsaturated oil into solid substitute is greatly reduced by first treating the oil with sulphur at from 140° to 160° C. The patent literature of the india-rubber industry has considerably increased. F. G. Kleinsteuber (Ger. Pat. 84,065, 87,673) prepares a product, commercially known as ambroin, by mixing solutions of copal with fibrous materials, and exposing these mixtures to high pressures. A process for vulcanising with electricity has been patented by Bergeon (Fr. Pat. 243,377), and C. Dreyfuss obtained a patent (Ger. Pat. 85,236) for vulcanising with a solution of chloride of sulphur in benzene.—C. O. W.

PATENT.

Impregnating of Materials with India-Rubber, Gutta-Percha, and the like; Impts. in and relating to the. W. J. A. Donald, Glasgow. Eng. Pat. 18,517, Aug. 21, 1896.

The fabric—leather, cotton, canvas, &c.—is dried, then passed through a warm bath of naphtha or carbon bisulphide, dried again, and finally treated with a solution of india-rubber maintained at a temperature of 100° or 110° F. The finished material is specially adapted for the construction of pneumatic tyres.—F. H. L.

XIV.—TANNING, LEATHER, GLUE, SIZE.

Leather, The Testing of. W. Eitner. Der Gerber, **23**, 53.

The test, depending on the observation that leather does or does not become transparent in water, is valueless. Neither well nor badly tanned leather, nor even raw hide, becomes transparent in water. The swelling or gelatinising action of strong acetic acid upon leather is rather important, and is, for an acid of given concentration, the more pronounced the smaller the quantity of tannic acid contained in the leather. A leather which is not affected by acetic acid of a certain concentration, will however begin to swell if subjected to the action of a more concentrated acid. If the concentration of the acetic acid exceed a certain limit, it will swell or even gelatinise even the strongest tanned leather. Unevenly tanned leather is also unevenly acted upon by acetic acid, so that the observation of the action of this acid upon a section of the leather affords valuable information concerning the thoroughness or penetration of the tan. Thus, well tanned leather belting on immersion in acetic acid of 20 per cent. strength should not show a swelling of the fibre to such a degree as to cause a glassy and pellucid appearance, since the tensile strength of leather decreases as the degree of penetration of the tan increases. On the other hand, sole leather allows a much higher degree of penetration, and will therefore withstand the action of an acid of correspondingly higher penetration. The use of this test as a criterion of the quality of a leather requires therefore considerable discrimination.

Another test according to which the leather after half an hour's boiling in distilled water should not, by the conversion of the leather into glue, render the water turbid, is very misleading. The presence in the leather of such an excess of tannin as would be required to satisfy this test would be fatal to the quality of many kinds of leather, and it will be found that especially the various kinds of tough leather would not stand this test. No better is the test in which the leather is boiled in a solution of alum, which, it is said, ought to remain clear.—C. O. W.

Suède or Glove Kid, The Tanning of. Der Gerber, **23**, 41.

The most important item in the production of suède and glove kid consists in the selection of the skins. These should be very firm, of a fine texture, and they should also be of the requisite dimensions. The next important point

is to conduct the tanning in such a manner as to preserve as much as possible the natural structure of the skins, and to avoid swelling. First class qualities of this leather can only be obtained from American young goat skins. The liming of the skins is carried out in very weak milk of lime, with an addition of sodium sulphide. The latter in French tanneries is often used by itself, or in conjunction with only very little lime. It renders the skins smoother, softer, more elastic and durable than the ordinary liming process, and, besides tending towards economy in the quantity of eggs required, yields skins which are not only very easily dyed but also assume very brilliant shades. In the sodium sulphide treatment the skins, though swelling, never become spongy. They at once re-assume their former thinness and toughness on immersion in water, whereas the lime treatment by itself always results in the texture of the skins becoming somewhat loose and rough. The time the skins require in this treatment varies from 7 or 8 to 10 or 12 days according to whether the depilation of the skins is carried out by hand or by machine, the latter of course being much the cheaper. After depilation the skins are sorted, those suitable for suède or glove kid are treated accordingly, the inferior skins are trimmed, dunged, cleared in acid bran water and pressed.—C. O. W.

Shoe Leather, The Tanning of. W. Eitner. Der Gerber, **23**, 42.

The fault most commonly committed in the tanning of shoe leather consists in working the hide too long in too weak liquors. The haired and fleshed hides are for two days treated in hemlock liquor and are taken successively through four liquors made up with two-thirds of hemlock and one-third of oak bark. In each of these liquors the hides remain for four days. They are then passed into three further successive liquors made up with equal parts of hemlock and quebracho (not the extract), remaining a week in each fresh liquor.—C. O. W.

Leather Dressing, Employment of Urine and Dog Excrement for Glove Leather. E. D'Huart. Bull. Assoc. Belge des Chimistes, **11**, [1], 21—39.

As a result of numerous experiments in the works and the laboratory, it appears that the effect of the canine excrement employed for dressing glove leather is due to the action of a microbe present in large numbers in the excrement and acting upon the amorphous organic matter of the cutaneous tissue and on the residue of the epidermis, which it destroys, the fibrous bundles being at first protected but becoming subsequently attacked if the exposure be prolonged.

The author considers that this explains why attempts made to replace this agent by various chemicals have proved unsatisfactory, the true cause of the action having been hitherto ignored, since the only rational substitute would be a bouillon containing the microbe in question, and not merely a mixture of ammonia, ammonium carbonate, and sodium phosphate. The same effect can, it is true, be produced by a complex mechanical treatment of beating, steeping, &c., but this is a much more expensive process than microbial action.—C. S.

XV.—MANURES, Etc.

Nitragin, Experiments with. Standard Agricultural Reps., Aug. 10, 1897.

Director Dickson and Professor Malpeaux, of the Berthonval (Pas-de-Calais) School of Practical Agriculture, describe in the *Journal d'Agriculture Pratique* some experiments which they have carried out with nitragin, Prof. Nobbe's preparation of nitrogenous bacteria. They first tried the preparation on clover grown in pots of sterile sand, to which some superphosphate of lime, sulphate of potash, and powdered lime had been added, cutting the produce in the flowering stage. From a pot to which no nitragin was applied they obtained 109 grms. of green clover; from a corresponding pot without nitragin, but with some nitrogenous manure, the produce was 132 grms.; from a third pot for which the nitragin was mixed with the seed they cut 159 grms.; and from a fourth, for which the nitragin was mixed with the soil, the crop was 168 grms. There were some duplicate pots from which the produce was almost exactly as given above. Lupins grown under like conditions gave 203 grms. in one case, and 205 in another without nitragin, and 250 with it. A trial with clover in the open ground showed a gain from the use of nitragin, but a less striking one than in the pot experiments. In the case of vetches grown on a plot of ground after wheat without nitragin the produce was 219 kilos., while, with nitragin mixed with the seed grain it was 232 kilos., and where the preparation was mixed in the soil it was 238 kilos. In a trial with white lupins no increase was obtained by mixing nitragin with the seed, but a substantial gain was derived from inoculating the soil with it. (This Journal, 1896, 767.)

Guano Deposit, The Utah. Eng. and Mining J. 1897, **63**, 602.

The deposits vary in thickness from 1 to 20 feet, and a recent estimate of the material in sight, after a very thorough examination, gives the total at over 1,000,000 tons. Analyses show that the Utah guano has about half the percentage of the soluble ingredients contained in the standard high grade Peruvian guano.—A. S.

Aluminium Phosphate from the Island of Grand-Connétable, Manurial Experiments with. A. Andouard. Ann. Agron. 1896, **22**, 247.

Experiments were made in which garlic, wheat, rape, beans, and black radish were grown in poor sandy soil to which potash, lime, magnesia, and nitrogen were added, besides 1 per cent. of phosphoric acid in the form of aluminium phosphate, crude bone-meal, basic slag, and superphosphate respectively. In most cases there was clearly assimilation of phosphoric acid applied as aluminium salt. In the case of beans, however, the yield of dry produce under the influence of aluminium was very low, as compared with bone-meal and superphosphate. Similar experiments in which garlic, rape, and radish were grown in garden soil, without manure, and with aluminium phosphate gave very irregular results.

The effect of aluminium phosphate on three kinds of wheat, growing in the field of the Experiment Station of the Lower Loire, was compared with basic slag and with Ardenne phosphorite meal. All three manures gave about the same yields of wheat.—N. H. J. M.

XVI.—SUGAR, STARCH, GUM, Etc.

Sugar Canes, Mode of Sampling and Method of Analysis of. Report on the Agric. Work in the Botan. Gardens, British Guiana, for the Years 1893-4-5; 9—10.

A representative sample, as far as can be judged by the eye, is taken, consisting of at least six ordinary canes, or from three to six seedling canes, according to the number of fully developed canes in the stool. It is stated that by this method of sampling, the errors are insignificant compared with those of cultivation caused by unfavourable conditions of soil and seasons. The large samples varying in weight from 15 to 30 lb. are sampled down in the following manner the same morning as they are cut down. The canes are sorted into pairs according to their physical characteristics. If of medium length, each cane is divided into four equal parts, and the top and third portions of one cane, and the second and butt portions of the other are reserved for the sample. The portions of the three composite canes thus obtained are split in half, one-half of each portion being taken to form the sample for analysis, and the remaining halves forming, if necessary, a reserve sample. Representative samples of 4 to 8 lb. in weight are thus obtained. When very large canes are being examined, they are first grouped in threes, each cane is divided into six parts, and the top and fourth portions of the first cane, the second and fifth portions of the second, and the third and butt portions of the third are taken to form a composite sample cane. The two composite canes thus obtained are further divided as already described.

The longitudinally divided half-canes are passed through the mill at first singly, then the expressed residues or megass from two pieces are placed together and passed through, this being repeated three times. An extraction is thus obtained approximating to that yielded by double crushing, but the juice obtained contains somewhat less of the sap constituents of the cane than that obtained on the large scale, and is consequently richer in sucrose, of a higher density, of a higher quotient of purity and of a lower quotient of non-sugars. The specific gravity of the juice, after straining from the coarser suspended impurities, is taken by a Westphal balance, arranged to show the density as at 84° F., which, as the coefficients of expansion of water and cane juice are practically the same, is identical with the density at 60° F. 50 c.c. of the juice are pipetted off into a 100 c.c. flask, clarified with a little sub-acetate of lead, the excess of lead precipitated by sodium phosphate, then the whole made up to 100 c.c., filtered and polarised in the usual manner. The glucose is determined in the liquid used for polarisation by means of accurately standardised Pavy's ammoniacal copper solution. The ash is determined by evaporating 5 c.c. of the original juice to dryness and burning at a barely visible red heat in a muffle furnace. The total solids, and thus indirectly, the non-sugars, are usually deduced from the specific gravity, but on estimating them by direct evaporation, it was found that the results obtained, in the case of the juice of the Bourbon cane, were 0·2 to 0·3 per cent. lower than those calculated from the density.—A. S.

Sugar Cane, Presence of Levulose in Ripe. H. C. Prinsen Geerligs. Bull. Assoc. Chim. 1897, **14**, 1080—1086.

In reply to the criticism of Pellet (this Journal, 1897, 250), the author shows that precipitation of levulose could not have occurred in his experiments on the juice of young and ripe canes, as he used only a few drops of acetate of lead slightly basic, and the filtered liquor was always left acid to litmus. He contends that the reducing sugars should not be studied in the molasses but in fresh juice, as they undergo great changes during the course of manufacture.

The following experiments made at the suggestion of Pellet show that in an acid medium, levulose is not precipitated by addition of basic acetate of lead; thus, in the author's experiments, all the sugars present passed into the filtrate, which was polarised, and in which the reducing sugars were estimated. Pellet had also found that levulose which was not precipitated by basic lead acetate in a simply aqueous solution, was immediately precipitated in part when a lead precipitate caused by salts, was formed in the solution. These results were fully confirmed by the author. The following solutions were made up to 100 c.c. and filtered before being examined :—

—	Sugar.	Dextrose.	Levulose.	Common Salt.
	Grms.	Grm.		
(1)	15	..	..	1
(2)	15	..	..	1
(3)	15	0·5	0·5	1
(4)	15	0·5	0·5	1
(5)	..	0·5	0·5	1
(6)	..	0·5	0·5	..

—	Basic Acetate of Lead.	Polarisation.	Reducing Sugars found.
	c.c.		
(1)	..	56·9	..
(2)	10	57·0	..
(3)	..	56·1	1·02
(4)	10	56·5	0·82
(5)	..	0·8	1·02
(6)	10	0·7	0·53

It is thus seen that sub-acetate of lead has increased the polarisation of (2) by 0·1 degree on account of the volume of the precipitate, but has considerably increased the polarisation in the cases of (3) and (4) and of (5) and (6), in consequence of the precipitation of levulose, and has diminished the quantity of reducing sugars for the same reason. To prove that the precipitate contained levulose, the precipitates of (4) and (6) were washed with water and dissolved in acidulated water. The solution was levo-rotatory and contained 0·18 grm. of reducing sugars. Neutral acetate, however, changed neither the polarisation nor the glucose found, so that by employing the reagent neutral or using the sub-acetate in so small a dose that the cane juice remains acid, there is no precipitation of levulose.

The author is completely in accord with Pellet that the quantity of dextrose and levulose in cane molasses is about the same as in invert sugar, but he is opposed to the conclusion that it follows that these two bodies are therefore found in the same proportion in ripe cane. This would be absolutely true if the reducing sugars underwent no change during the manufacture. Unfortunately dextrose and levulose do not remain intact during the heatings and re-heatings with lime and salts which they undergo in the factory. Alberda van Ekesteyn and Lobry de Bruyn have shown (this Journal, 1896, 210) that on treating dextrose with small quantities of base it is partially transformed into levulose and mannose. But it is not necessary to employ alkali in excess. The author made a solution containing 10 per cent. of dextrose and sodium acetate equivalent to 2·5 per cent. of Na_2O, and a similar solution containing 2·5 per cent. of K_2O. These two solutions were heated in a Koch's sterilisation cylinder so as to remain at 100° C. without evaporation. After one, two, three, and four hours, portions were withdrawn and tested. The polarisations gradually diminished from 27·3 and 26·9 to 12·6 and 9·8 respectively, whilst the reducing sugars remained practically the same. The quantity of reducing sugars is therefore not affected by this transformation, but merely the quality. A solution of dextrose, after heating with sodium acetate until the rotation was almost zero, was evaporated and mixed with phenylhydrazine acetate and alcohol; a yellow precipitate was gradually formed, and was recognised as the phenylhydrazone of mannose. The remaining solution extracted with ethyl acetate gave a levo-rotatory sugar with all the characters of levulose. The preponderance of dextrose in ripe cane is moreover not astonishing if one considers the formation and fixation of sugars in the cane as worked out by Went, whose microscopical researches give results in perfect agreement with the chemical analyses of the author.—L. J. de W.

Raffinose in Cane Molasses. H. Pellet. Bull. Assoc. Chim. 1897, **14**, 1086—1089.

When raffinose is present in only small quantity, the results of the analysis are not sufficiently affected to be considered abnormal; it is necessary for the percentage of raffinose, as determined by the inversion method, to reach 2 to 3 per cent., or that the residue after fermentation should polarise strongly to the right for the attention of the chemist to be called to the presence of this sugar. It was thus that Pellet and Barbet were led to suspect the presence of raffinose in a sample of cane molasses from Louisiana; and subsequently they adduced evidence that it contained 2 to 3 per cent. on the weight of the molasses. Other samples, notably in Egypt, did not show sufficient to appreciably affect the results, even when examined after the reducing sugars had been removed by fermentation.—L. J. de W.

The Ranson Process. [*Decolorising and Purifying Sugar, Syrups, &c.*] A. Vivien. Bull. Assoc. Chim. 1897, **14**, 1073—1077.

There are two methods of operation in the Ranson process. That preferred in the refinery is to oxidise organic matters by adding oxygenated water, and then to add a substance having a great affinity for oxygen, such as a sulphite, hydrosulphite, or the corresponding acid. The second method is the inverse of this. It commences by treatment with hydrosulphurous acid in the nascent state, and terminates in the use of oxygenated water, to destroy the affinity of hydrosulphurous acid and oxidise the sulphites formed, and any organic matter capable of being destroyed by oxidation. Oxygenated water acts on organic matters through the nascent oxygen as well as by the molecule of water, both in the nascent state, formed when it decomposes. Any

excess would act on sugar and form glucose, lactic acid, &c. By operating methodically no trace of the reagents employed remains in the liquors treated. The zinc powder used in the preparation of the hydrosulphurous acid also combines, in the form of oxide, with certain organic matters and precipitates them. The combined result of the reactions is purification and decolorisation.

In applying the process in the factory, the syrups and drainings which are to form the first runnings masseeuite are treated in the ordinary sulphiting vats with sulphurous acid to neutrality and then filtered. The greater part of the calcium sulphite is thus eliminated, producing an increase of saline quotient and of purity.

The syrups are then passed, at a temperature of 45° C. to sulphiting vats provided with agitators and "Ranson" oxygenated water added in quantity to transform the soluble sulphites into sulphates. The syrups are then sulphited a second time, so as to leave an acidity expressed as sulphurous acid of 0·5 to 5 grm. per litre, according to the nature of the syrup, then 10 to 50 grms. of zinc powder is added per hectolitre of syrup and vigorously agitated for 15 to 20 minutes. Hydrosulphurous acid is formed, which is a very energetic reducing agent. In its nascent state it destroys 85 to 95 per cent. of the colouring matter, precipitates some nitrogenous matters and considerably diminishes the viscosity of the syrups.

After heating to 75° C., if there is any fear for the nature of the syrup, it is neutralised by baryta, or better, by barium sucrate, but with sound beets it may be left slightly acid and the cost of baryta reduced.

The massecuite obtained is pale, fluid, easy to machine, and gives an extra sugar which keeps perfectly, in store.

When it is boiled acid, the drainage syrups are made slightly alkaline, sulphited and treated as before. The first runnings massecuites are very fine and slightly alkaline, the acidity of the syrups due to organic acids and sulphurous acid disappears, during the boiling, in the condense waters and gives a further purification. No uncrystallisable sugar is formed when working under the above conditions. The plant required is very simple and already exists in the greater number of the factories employing the ordinary sulphiting. For the treatment of first runnings syrups it is preferable, though not indispensable to have recourse to double filtration: the sulphiting vats where zinc powder is added should be furnished with refrigerators and stirrers. The cost of materials is about 0·2 franc per 100 kilos. of sugar. The advantages are:—Increased yield, about 5 kilos., and diminution of molasses, about 10 kilos. per 1,000 kilos. of beetroots, with a gain of 4·867 francs.—L. J. de W.

The Ranson Process, Application of, in the Sugar Refinery. Bull. Assoc. Chim. 1897, **14**, 1077–1080.

Very pure sugar solutions are obtained by the Ranson process (see previous abstract), perfectly decolorised and purified; the massecuite is very white, and is subsequently converted, by special mechanical means, forming part of the process, into fine slabs of refined sugar for breaking into lump, which can be delivered for consumption within 48 hours.

When the oxidising treatment is employed, that is when oxygenated water is used first, the syrups (clairce for boiling or fine clairce) are rendered alkaline with baryta, or failing that, with carbonate of soda, Ranson oxygenated water added and mixed for about two hours at 40 to 50° C. The decolorisation and purification are effected during mixing. An excess of oxygen is used to obtain as complete a decolorisation as possible, and this excess is removed immediately afterwards, or the syrups would be attacked and subsequently recoloured, forming fatty acids, butyric, ulmic, propionic, waxy matters, &c. The excess of oxygen is removed by hydrosulphurous acid (H_2SO_2), which at the same time decolorises the syrups and diminishes the viscosity. The hyposulphurous acid is added as barium, aluminium, &c., salt, or in the free state, and in the latter case it is produced in the liquid itself in the nascent condition. A slight excess is used in order to utilise its special action in completing that of the oxygenated water. By operating thus, the colouring matters rich in oxygen are destroyed by reduction, whilst those poor in oxygen have already been destroyed by oxidation. At the same time there is a purification by precipitation of organic matters in the form of organic salts of baryta and of zinc and diminution of viscosity. The excess of hydrosulphurous acid is removed by a fresh addition of oxygenated water exactly determined by titration with iodine and starch. Finally it is heated to from 75 to 80° C., and filtered mechanically.

The process may also be carried out commencing with the reducing treatment with hydrosulphurous acid.

The purity and saline quotient are raised and ¾ths of the colour removed, as shown by the following analyses:—

Per Hectolitre.	Fine Clairce. Initial.	Fine Clairce. After the Ranson Treatment.	Clairce for Boiling. Initial.	Clairce for Boiling. After the Treatment.
Sugar	66·050	66·050	54·000	54·000
Glucose	0·095	0·095	0·150	0·150
Ash	0·164	0·160	0·235	0·220
Organic matter	0·222	0·158	1·820	1·380
Water	33·169	33·537	43·795	44·250
	100·000	100·000	100·000	100·000
Purity	99·28	99·38	96·07	96·87
Saline quotient	402·80	412·81	229·78	245·45
Decolorisation	Type	78·00	Type	75·00
Alkalinity	Trace	Trace	Trace	Trace

The plant necessary for the treatment of syrup in the refinery consists of tanks furnished with stirrers, and of mechanical filters; the treatment is simple, readily acquired and easy to control. The cost of the process is 0·15 franc per 100 kilos. of sugar melted, or 0·20 franc if sugar composed of ⅔rds of No. 3 and ⅓rd brown sugar is used, the drainage syrups being returned, so as to have a melt of 96 per cent. minimum.—L. J. de W.

Diffusion and Scum Washing, Limits of Loss in. H. Pellet. Bull. Assoc. Chim. 1897, **14**, 1089–1093.

The question of the loss of sugar in exhausted beetroot pulp has been studied by Karlson (Bull. Assoc. Chim., March 1897), who concludes that as the purity of molasses may be reckoned at about 60, the weak juices of the last diffusers should not be worked if they have a purity equal or inferior to this, and consequently the loss of sugar in the pulp will be variable, and may be high. For example, if the weak juice of the last diffuser, in which the exhausted pulp contains 0·30, has a purity of 45, this juice will produce only molasses, and will, besides, take up sugar so as to come out in the molasses of 60° of purity. If the last diffuser contain a weak juice of 60° of purity, and the pulp 0·60 per cent. of sugar, it should be stopped at this point, otherwise the production of molasses will be increased, this conclusion being based on the fact universally known and acknowledged that the further the diffusion is pushed the more impure is the product obtained.

Although this is true in a general way, if the pulp be exhausted by increasing the volume of juice drawn off, but if, by special arrangements which have been put into practice, the pulp can be exhausted with a less volume of liquid, the total purity of the product extracted may be higher, and less sugar left in the exhausted pulp.

The purity of the weak juices is falsified, however, in various ways. The water used has a great influence. 3 grms. of sugar dissolved in a litre of ordinary water showed a purity of only 66 on the ordinary instruments, without having been in contact with the pulp. When the impurities brought by the water used for diffusion are taken into account, the impurities coming from pulp more or less exhausted will be found to vary very little, and the last juice extracted as pure as that extracted first.

In concentrating the thin juice, it is best to evaporate in a copper dish to 114° C., when, if the juice has been well purified, the colour is the same as the juice from which it

was produced. In this way several litres may be evaporated in a few hours, care being taken that it does not burn at the sides, as it does in porcelain vessels.

The same considerations apply in washing the scums. No more impurity is obtained, when the washing is complete, with a small quantity of water than with a larger quantity. The impurity is mostly lime, from particles incompletely carbonated, as may be seen from the increase of alkalinity.
—L. J. de W.

Beetroot, the Different Forms of Nitrogen in the, and their Influence on the Relative Proportions of Sugar and Glucose. E. Urbain. Bull. Assoc. Chim. 1897, **14**, 1095—1098.

It is constantly remarked that on beginning the manufacture of sugar the beetroot contains only traces of reducing sugars, but as manufacture proceeds the quantity increases, particularly for beetroots in silos. With the increase of reducing sugars, the nitrogen amides and ammonium salts also increase, at the cost of albuminoids and nitrates, the total nitrogen being sensibly constant. This may well explain the great difficulties presented by purification towards the end of the season, for if the albuminoids are almost entirely precipitated by lime, it would not be the same with the soluble nitrogen compounds. There is evidently disengagement of ammonia during carbonation, but not in sufficient quantity to compensate for the albuminoids which have disappeared. The presence of a larger quantity of nitrogen as ammonium salts may also explain the diminution of alkalinity during evaporation, for there is here a veritable displacement of ammonia, so well known that it is unnecessary to insist upon it.—L. J. de W.

Wheat, The Insoluble Carbohydrates of. H. C. Sherman. J. Amer. Chem. Soc. 1897, **19**, 291—310.

The author divides his communication into three parts—descriptive, analytical, and physiological. In the first part he concludes from his experimental evidence, that the hemicellulose of wheat consists of pentose anhydrides or true pentosans, since, on inversion with dilute sulphuric acid, pentoses only are obtained. The action of ferric chloride and potassium ferricyanide on wheat fibre showed that, like the typical lignocellulose (jute), the amount of cyanide fixed upon the fibre varied roughly with the amount exposed to the action. This reaction is interesting as showing the similarity of the lignocellulose molecule in these tissues. Pursuing Cross and Bevan's directions ("Cellulose," p. 135) the author obtained with wheat fibre a lignone chloride, which, on analysis, gave numbers agreeing with the formula $C_{19}H_{18}Cl_4O_9$. The fact that this compound can be obtained from such widely different tissues as that of the coat of the wheat grain and jute fibre would seem to indicate that this type of lignification may be general among plants of annual growth.

In order to separate and study the properties of the cellulose of the wheat fibre, the methods of Schulze, Cross and Bevan, and Lange (Zeits. angew. Chem. 1895, 561) were employed, the second-mentioned was, however, preferred.

When acted on by strong sulphuric acid according to E. Schulze's directions (Zeits. phys. Chem. **16**, 414), the cellulose, unlike cotton cellulose, did not wholly dissolve, and a very considerable discoloration was produced. The undissolved portion (nearly one-fifth) did not yield a measurable amount of furfural. The osazone formed was that of dextrose. From this it will be seen that the products of hydrolysis of the furfural yielding group do not appear. By the action of cold, dilute, sodium hydroxide, a filtrate and residue were obtained; the former, on neutralisation with hydrochloric acid was found to contain a substance bearing a general resemblance to wood-gum, in that it gave the characteristic reaction with phloroglucin. The residue constituted about 80 per cent. of the amount treated; a determination of furfural gave 3·34 per cent., equal to 2·67 per cent. of the original. Hence less than one-third of the dissolved substance was pentosan. On reviewing his experiments, the author believes that wheat cellulose contains about 10 per cent. of penta-anhydride, which appears to be chemically combined with a part at least of the hexa-anhydride (normal cellulose); also that the furfural yielding constituents of wheat are essentially pentosans in some form; further, that there is no serious error in considering the amount of furfural obtained as a measure of the total pentosans present.

Part II. Analytical.—It is customary in cereal analysis either to be content with the results of the Weende method or to determine in addition, the starch and, perhaps, the furfural obtainable, calculating the latter to pentosan. The author estimates starch, true pentosans, cellulose, lignin, and allied substances, and combines these determinations into the following scheme:—

<table>
<tr><td colspan="5">5 grms. of the sample (previously extracted with ether) are stirred with about 100 c.c. of water for a few minutes, filtered, and washed.</td></tr>
<tr><td rowspan="4">Filtrate.—Add one-tenth its volume of 25 per cent. hydrochloric acid and heat in a reflux condenser for 2½ hours on a water-bath, or boil ½ hour on a sand-bath.
Clarify, if necessary, and determine the dextrose present in an aliquot portion by means of Fehling's solution and calculate to</td><td colspan="4">Residue.—Wash into a beaker with 100 c.c. of water (more if the proportion of starch is high), heat to boiling, cool partially, and invert with malt extract till starch disappears, filter, and wash.</td></tr>
<tr><td rowspan="3">Filtrate.—Treat as in the case of preceding filtrate and calculate the dextrose found to</td><td colspan="3">Residue.—Boil ½ hour with 1·25 per cent. of sulphuric acid, filter, and wash with water and alcohol.</td></tr>
<tr><td rowspan="2">Filtrate.—Add sufficient sulphuric acid to make 2 per cent.; boil gently in reflux condenser for six hours; determine reducing power, calculate to pentose, and thence to</td><td colspan="2">Residue.—Dry and weigh. In a duplicate residue, determine proteid (and ash) and correct the weight accordingly. Treat according to the method of Cross and Bevan (loc. cit.), dry, and weigh the residue.</td></tr>
<tr><td>Loss of weight corrected for proteids (and ash) equals</td><td>Residue, less proteid, if present (and ash), equals</td></tr>
<tr><td>Soluble carbohydrates as dextrin.</td><td>Starch ....</td><td>Free pentosans ....</td><td>Lignin and allied substances.</td><td>Cellulose.</td></tr>
</table>

The reducing powers were estimated by adding 25 c.c. of the solution to be tested to an excess of boiling Fehling's solution under the usual conditions of dilution, boiling exactly 2 minutes, collecting and determining the cuprous oxide by Lavene's method as follows:—The reduced oxide contained in the filtering tube is dissolved with nitric acid, and washed thoroughly into a flask; an excess of sodium carbonate is added to the solution, and the precipitate dissolved with 1 c.c. of ammonia (0·96 sp. gr.). It is then titrated with a recently standardised solution of potassium cyanide until the colour disappears. Experiments showed that varying amounts of sodium nitrate did not affect the results.

The following method for the determination of furfural is essentially that of the Association of Official Agricultural Chemists. From 2 to 5 grms. of the sample are distilled with 100 c.c. of hydrochloric acid (sp. gr. 1·06), the distillate being collected in a graduated receiver. Each 30 c.c. lost by distillation is replaced by an equal amount of hydrochloric acid. This process is continued until a drop of the distillate produces no red coloration with aniline acetate. Eight to twelve distillations are generally necessary. Sufficient water is added to the distillate to bring it to a volume of 400 c.c., and for every 50 c.c. of water added, 10·2 grms. of sodium chloride are also added. The solution is then exactly neutralised with sodium carbonate

and stirred for half an hour with 10 c.c. of phenylhydrazine acetate, made by mixing 12 grms. of phenylhydrazine with 7·5 grms. of glacial acetic acid and diluting to 100 c.c. It is then allowed to stand until the following day, filtered into a glass filtering tube containing glass wool, and dried to constant weight at 55° to 60° C. in a current of dry air. Finally, the precipitate is dissolved in hot alcohol, the tube dried and weighed, and the loss of weight calculated as furfural hydrazone. For reducing to furfural and to pentosans the following formulæ were used :—

Hydrazone × 0·516 + 0·0104.............	Furfural.
Furfural × 1·84..........................	Pentosans.

The factor for pentosans was obtained by averaging those of xylan and araban.

In Part III. the author discusses the digestibility and physiological value of the carbohydrates, and points out that little information is to be derived from the current methods of analysis, which simply divide this group into "nitrogen-free extract" and "crude fibre."—J. L. B.

Sugar Products, Estimation of Lime in, by Soap Solution. H. Pellet and O. Castels. Bull. Assoc. Chim. 14, [11], 1100.

See under XXIII., *page* 702.

PATENTS.

Molasses, Impts. in Extracting Sugar from, by Means of Barium Hydroxysulphide, and Recovering the Same from the Resulting By-Products. W. Feld, Honningen am Rhein, Germany. Eng. Pat. 16,009, July 20, 1896.

In order to avoid interruption of the continuous process for the extraction of sugar from molasses by barium hydroxysulphide, either alone or admixed with alkali hydroxide, the loss of sulphur is made up by mixing the residual liquid, separated from the precipitated barium saccharate, with alkali sulphate.

The barium sulphate is removed, reduced by carbon to barium sulphide, and again converted by water into barium hydroxysulphide to re-enter the cycle of operations. The liquor is then treated with carbonic acid to liberate sulphuretted hydrogen, which is converted into sulphurous or sulphuric acid that can be used for decomposing barium saccharate, whilst alkali carbonate is obtained by calcination of the residue.—L. J. de W.

Evaporating Apparatus, Improved. R. H. F. Finlay, Belfast. Eng. Pat. 16,552, July 27, 1896.

See under I., *page* 661.

Saccharine Solutions, Impts. in Apparatus for Clarifying. L. F. Haubtman, New Orleans, U.S.A. Eng. Pat. 20,434, Sept. 15, 1896.

This is a device by which saccharine solutions from sugar-cane or other sugar-producing material may be rapidly clarified without contact with atmospheric air.

The apparatus comprises a number of connected heating vessels, arranged one above another, each vessel having in one end a receiving chamber and a discharge chamber, and at the opposite end a transferring chamber, with tubes connecting the transferring and discharge chambers, and valves for closing the ends of certain of the tubes, and a partition extended longitudinally in the vessel, and having a space between its end and one of the tube sheets. There are also means for forcing a heating medium, like steam, downwards through the several heating vessels, and for forcing a liquid through in a direction opposite to that of the heating medium ; a tank for receiving the heated liquid, and a cooling vessel similar to the heating vessels, with a tank for crude cold solution.

The heat, as soon as it strikes the liquors to be treated or clarified, separates the pure liquor from the impurities, and, to keep these impurities from settling in the tubes in transit, this liquor must have a rapid circulation through the tubes. To maintain this circulation when a smaller quantity is to be handled, certain of the tubes are closed by the valves provided. The crude cold solution is used for cooling the heated clarified liquor as it passes through the cooler to the settling tank.—L. J. de W.

XVII.—BREWING, WINES, SPIRITS, Etc.

Primary Fermentation and Attenuation [Brewing], Effect of Malt Meal on. F. Cerny. Oesterr. Brauer- u. Hopfen-Zeit. 1897, 119 ; Zeits. f. d. ges. Brauw. 20, [21], 279—282.

From experiments made with samples (1·6 litre each) of 10·25 wort, pitched with 4 grms. of hops, and containing 0, 0·2, 0·5, and 1·0 grm. of malt meal respectively, the author concludes that malt meal or malt extract may render useful aid to the brewer in critical cases when a suitable attenuation cannot be obtained by a change of yeast or by modifications of the brewing process. The yeast strengthening properties attributed to the meal are, however, merely stimulative and not of a permanent character. A minimum quantity suffices to increase the attenuation at the end of the primary fermentation, but for raising the final attenuation, the effect (which is proportional to the amount of meal used) is best attained by adding the meal (or extract) before the secondary fermentation, since the yeast is thereby better protected. The best form of all is a cold aqueous extract of sterilised malt meal, concentrated diastase preparations not being available in sufficient quantity for practical use.

The actual effect of the above-named additions on the apparent attenuation in the primary and secondary fermentation was as follows :—

	Sample with			
—	0·0 Per Cent. of Malt Meal.	0·2 Per Cent. of Malt Meal.	0·5 Per Cent. of Malt Meal.	1·0 Per Cent. of Malt Meal.
Apparent attenuation:	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Primary	59·90	76·39	78·14	78·73
Final	77·85	82·24	84·68	88·58
Increase ..	17·95	5·85	6·54	9·85

—C. S.

Barley and Malt, Sugar and Starch Formation in. I. Gruss. Wochenschr. für Brauerei, 1897, 321—323.

The author, noting that dried malt always contains less sugar than the same malt when green, was led to carefully re-investigate the ferments present in germinating barley. Very careful precautions were taken to avoid all bacterial complications, and the presence of three distinct ferment actions was confirmed, namely, a starch dissolving, a cell-wall dissolving, and an inverting action. The cell-walls of the endosperm which are dissolved, are apparently hemi-cellulose with a pentose structure. Whether all these ferment actions are due to one or three different ferments is doubtful, but the author appears inclined to the former view.—A. L. S.

Saké, the National Drink of Japan. O. Schiewek. Wochenschr. für Brauerei, 1897, 337—339.

The preparation of saké has already been described in this Journal (1895, 670). The author has studied the growth and spore formation of the mould, *Aspergillus oryzæ*, on different substances, and finds it to grow best on rice. It requires a temperature above 40° F. Several varieties of yeast were found with the rice mould, amongst which were an anomalous form and one with round spores.—A. L. S.

Cereal Straws, The Carbohydrates of the. C. F. Cross, E. J. Bevan, and Claud Smith. Proc. Chem. Soc. 1897, [182], 150—152.

This paper deals with the results of further investigations of the products of acid hydrolysis of the cereal straws and of the celluloses isolated from them, including also the closely related esparto-cellulose. The results confirm those previously communicated (this Journal, 1896, 386), that

the furfural-yielding constituents (furfuroids) are selectively attacked, and for the most part (90 per cent.) dissolved; also, from the exceptionally high numbers for cupric reduction, that they must exist in solution in a fully hydrolysed form (monoses).

The solutions when neutralised, ferment with yeast: carbonic acid and alcohol are produced, and a proportionate effect upon the constants of the solution is shown (density, opticity, cupric reduction, and furfural). Under the conditions, the proportion of the celluloses fermented, amounts to 30 per cent. of the total dissolved solids. Similar conclusions are deducible from a recent paper by Tollens (J. für Landw., 1897, 106—107), in which the fate of malt furfuroids in beer-fermentations is discussed. The experimental numbers in this paper show the disappearance of a large proportion of these constituents in the process.

Since the pentoses entirely resist alcoholic fermentation, as shown by Tollens (Kohlenhydrate, ii.), and further confirmed by the authors, as well as by later observations of Tollens privately communicated, it is evident that the group of furfuroids thus fermented is constitutionally distinct from the pentoses.

Incidentally, and with regard to observations on alcoholic fermentation with mixtures of known hexoses and pentoses, the authors find that the latter remain unaffected in presence of hexoses undergoing fermentation. Under certain conditions, however, the pentoses are removed from solution by the yeast organism; the necessary condition appears to be that of "starvation," in the sense, *i.e.*, of the absence of hexoses. The disappearance of the pentose under these conditions is indicated by determinations of furfural and the fall of the furfural numbers. This phenomenon appears to be the simple one of assimilation by the yeast organism, as shown by Bokorny (Dingler's Polyt. J., 1897, v. 303; this Journal, 1897, 345). The pentose undergoes constitutional change in such assimilation, as the yeast shows no increase in its normal small furfural number.

The authors further discuss the question of the constitution of furfuroids thus shown to yield alcohol on fermentation, and conclude that the hypotheses of the existence of methylene ethers of the pentoses, or pentose formals, affords, up to the point arrived at, a consistent view of their differentiation from the pentoses.

Laccase, Intervention of Manganese in the Oxidising Actions of. G. Bertrand. Bull. Soc. Chim. 1897, **17**, 619—624.

The ash of this ferment contains manganese, the quantity of which was determined by conversion into permanganic acid by boiling with nitric acid and lead peroxide, and comparison of the depth of tint in the colorimeter. The amount of oxygen absorbed by a solution of hydroquinone in presence of this laccase is roughly proportional to the percentage of manganese. The laccase extracted from lucerne (*Medicago Sativa L.*) contains a very small proportion of manganese; its oxidising power is much increased by the addition of manganese sulphate. In this experiment, manganese cannot be replaced by any other metal, not even by iron.—A. C. W.

Acetic Bacterium, A New. Zeidler. Zeits. für Spiritusind. 1897, **20**, [21], 164, and [21], 171—172.

The properties of this bacterium have led the author to name it *termobacterium aceti.* It produces acetic acid from alcohol, and in its form and motile property it agrees with Cohn's termobacterium; but, on the other hand, it differs from the latter in that it has a great tendency to assume involution forms. The motile property is retained in beer cultures until the acidity is equal to 30—35 c.c. $\frac{1}{10}$ N soda solution to 10 c.c. of the liquid, whilst in wort cultures the same property is lost when the acidity reaches 4—5 c.c. In addition to acetic the termobacterium also produces a fixed acid (apparently lactic acid) when grown in beer-wort and solutions containing dextrose, the maximum limit of the fixed acid corresponding with 5 c.c. of $\frac{1}{10}$ N soda to 10 c.c. of liquid. In the presence of alcohol, both acids are formed; thus a wort free from alcohol, showed an acidity after a month, equal to 4·4 c.c. $\frac{1}{10}$ N soda, whilst with the addition of alcohol the acidity reached 59·7 c.c., of which 4·5 were due to fixed and 55·2 to volatile acid. The volatile acid is only produced in alcoholic liquids. The termobacterium also has the power to convert acetic acid into carbon dioxide and water, but it loses this property when the acidity exceeds 43 c.c. of $\frac{1}{10}$ N soda. On wort gelatin it gives a weak growth which does not liquefy the gelatin; in stab cultures it shows a distinct growth, which afterwards becomes confined to the surface, showing the bacterium to be aerobic. The most favourable temperature for the growth and activity of the organism appears to be about 25°. Large quantities of alcohol prevent its development, and in a wort to which 16 per cent. or more alcohol was added the organism perished; with 6 per cent. of alcohol and after 10 days' growth the yield of acid was greatest. Termobacterium aceti is much less resistive than the ordinary acetic bacteria; its cultures in beer and wort perish when kept for six months at the ordinary temperature, and in the presence of the true acetic bacteria, it soon becomes suppressed.—A. K. M.

Fermentation, Volatile By-products of. A. C. Chapman. J. Fed. Inst. Brewing, 1897, **3**, 240—254.

After discussing the nature and probable origin of the volatile by-products of fermentation, the author describes the methods which he employed to estimate the higher alcohols, compound ethers, furfural, and aldehyde. A quantity of the beer, varying from four to eight litres, was distilled until the whole of the alcohol was obtained in a small volume (500 to 600 c.c.). The distillate was made up to a definite volume, and aliquot portions were taken for the estimation of the higher alcohols, compound ethers, and furfural. The higher alcohols were determined by extraction with carbon tetrachloride and conversion into the corresponding fatty acids; the compound ethers by saponification,—the results being expressed in terms of acetic ether (ethylic acetate)—the furfural and aldehyde by the aniline acetate and rosaniline sulphite reactions respectively. The following table shows some results obtained with English beers, the percentages being expressed in parts per 100 of proof spirit :—

——	XXXX Beer.	Light Bottled Beer (carbonated).
Higher alcohols (as amyl alcohol) ...	0·250	0·1300
Compound ethers....................	0·036	0·0440
Furfural..............................	0·002	0·0006
Aldehyde..............................	Trace.	Trace.

——	Bitter Beer.	Bottled Beer.	Mild Ale.
Higher alcohols (as amyl alcohol)....	0·140	0·0840	0·0700
Compound ethers....................	0·052	0·0370	0·0240
Furfural..............................	0·002	0·0018	0·0006
Aldehyde..............................	Heavy trace.	Heavy trace.	Trace.

Analyses of some samples of beer which had been in bottle for many years, showed high percentages of compound ethers, to which the marked flavour of the samples was due. The amount of higher alcohols was not greater than in new beers—a result which is opposed to Lindet's supposition that these substances are formed chiefly when the vigorous primary fermentation is over and the yeast is to some extent exhausted. Experimental evidence is brought forward showing that with high-fermentation temperatures, there is a distinct increase in the percentages of the higher alcohols and generally of the compound ethers.—J. L. B.

Malt, Note on the Value of the Soluble Carbohydrates as an Indication of Forcing of. L. Briant. J. Fed. Inst. Brewing, 1897, **3**, 222—228.

It appears to be the opinion of some brewers that a high proportion of soluble carbohydrates is certain evidence of a forced malt; the object of the author's communication is to show that the facts do not justify quite so definite a

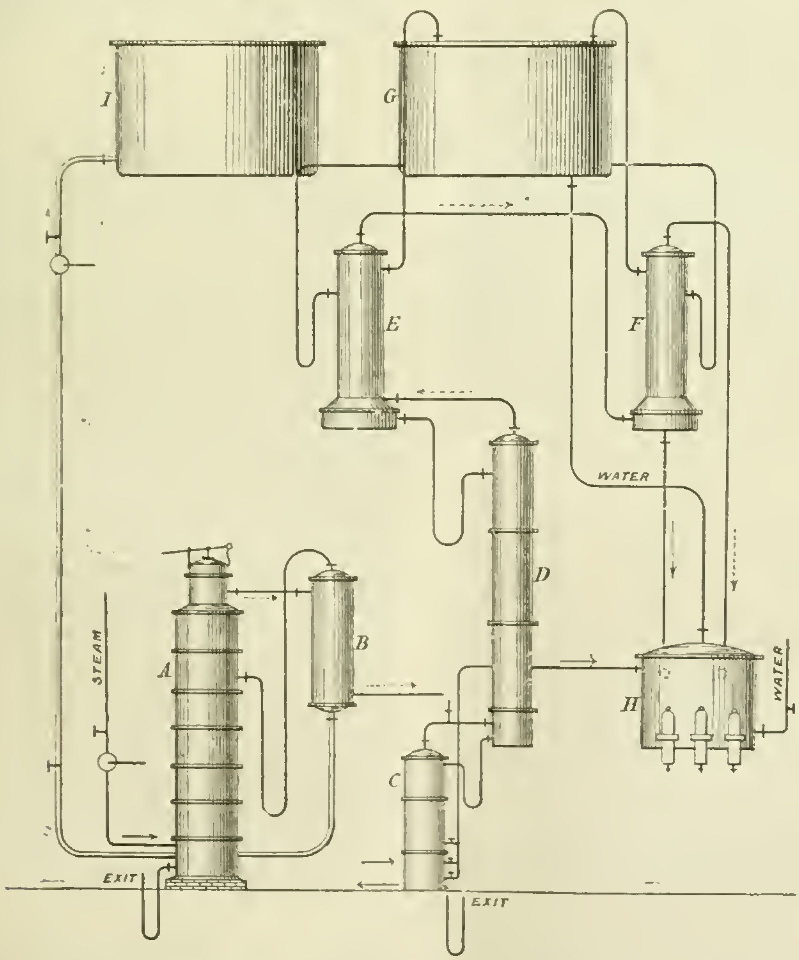
conclusion. It is pointed out that the amount of root produced on the floor has an intimate connection with the quantity of matter, soluble in cold water, yielded by the malt. The author's experiments demonstrate that malts grown with short roots on the cool system contain more matter soluble in cold water than those with long roots. High proportions of soluble matter may be objectionable, but it is contended that they are not necessarily an indication of a forced malt. Malts produced on sound lines may contain as much as 17·5 to 18 per cent. of soluble carbohydrates, and it is suggested that a limit higher than that now commonly employed should be established. Attention is also drawn to the fact that certain types of barley naturally give malts having larger proportions than others of soluble carbohydrates, even when made on the best possible lines, so that a proportion which indicates forcing in one sample, might not do so in another.

On the other hand, the author is of opinion that a small proportion of soluble carbohydrates does not necessarily indicate that a malt has not been forced. The author instances a sample of malt which was "worked up very roughly with about 12 gals. of sprinkling water on the floors at about 68° F., whilst during withering the temperature rose to nearly 90° F., and yet the percentage of soluble carbohydrates was not very abnormally high, being 16·7 per cent." He is inclined to think that these carbohydrates were absorbed by the root, which was unusually large. To detect forcing, the determination of soluble carbohydrates is not sufficient; but if several considerations, such as the diastatic power, the tintometer value, and nitrogenous matter soluble in cold water, be taken into account, the author considers that "forcing" may be detected by analysis.—J. L. B.

Organic Products, Industrial Distillation of. E. Barillot. Bull. Soc. Chim. 1897, **17**, 635—638.

The principle upon which the working of the apparatus is based is that when a complex vapour is led through a bath at a constant temperature, those substances are condensed whose boiling points lie below the bath temperature. Thus, by the use of a series of baths, it is theoretically possible to separate complex mixtures.

In the figure, the liquid to be distilled—wine, must, or wood spirit—is heated, and then enters the column A, from which it proceeds into the heated vessel B. The alcohol vapours are evolved in the column D, and rise into the analyser E, the higher boiling products, mixed with alcohol, run into C, where the alcohol is distilled off and the oils



left conducted into H. The light vapours are further analysed in F. The analysers are kept at a constant temperature by baths of boiling liquid surrounding them, the baths being connected with the reflux condenser G.

—A. C. W.

Wheat, The Insoluble Carbohydrates of. H. C. Sherman. J. Amer. Chem. Soc. 1897, **19**, 291.

See under XVI., *page* 690.

Wood Spirit, Presence of Vinic Alcohol in Raw. G. Arachequesne. Bull. Assoc. Chim. **14**, [11], 1108.

See under III., *page* 667.

Acid Potassium Tartrate in Wine, Determination of. H. Jay. Bull. Soc. Chim. 1897, **17**, 626.

See under XXIII., *page* 704.

PATENTS.

Barrels, Casks, and the like; Impts. in or relating to the Purification or Sweetening of Used. W. J. Engledue, Byfleet, and C. J. Yarnold, Brixton. Eng. Pat. 16,632, July 27, 1896.

The barrel or cask is sweetened by subjecting it to the action of ozone; or its interior is first damped with water or steam, and it is then subjected to the action of ozone, or a mixture of ozone and steam, and finally cleansed by steam or hot water.—L. J. de W.

Condenser for Steam-heated and Similar Pans or Vessels, chiefly applicable to those used in treating Aromatic Substances; A New or Improved. J. F. Henderson, Manchester. Eng. Pat. 7443, March 23, 1897.

See under I., *page* 661.

XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

(*A.*)—FOODS.

Tea, Chinese; On the Relation between the Amount of Caffeine in and Quality of. L. Graf. Forschungs-Ber. 1897, **4**, [4], 88—89.

It has been pointed out by Kellner that in the living tea-plant the amount of caffeine in the leaves decreases with the growth. Since the finer kinds of tea consist of the younger leaves, the author has made experiments to determine whether the percentage of caffeine could give any indication as to the quality of the tea. Two varieties were examined, and the caffeine was determined in the aqueous extract by precipitating the tannin with basic lead acetate, filtering, removing the lead, concentrating the filtrate after the addition of sodium acetate, filtering, and extracting the alkaloid with chloroform. As thus obtained, the caffeine was but slightly coloured, and contained only traces of impurities.

The results, calculated on the air-dried tea, were:—

——	Caffeine.	Price.
	Per Cent.	Marks.
Souchong 206	2·96	1·30
„ 273	3·10	1·80
„ 266	3·53	3·15
Congou 279	2·82	1·80
„ 269	3·70	2·40
„ 268	4·09	3·10

Thus in both kinds of tea the amount of caffeine increases with the quality and commercial value.—C. A. M.

Sausages, Detection of Artificial Colour in. H. Bremer. Forschungs-Ber. 1897, **4**, [2], 45.

See under XXIII., *page* 700.

Cheese, Detection of Margarine in. H. Bremer. Forschungs-Ber. 1897, **4**, [3], 51.

See under XXIII., *page* 700.

Tea, A New Adulterant of. [*Detection.*] J. Delaite and H. Lonay. Bull. Assoc. Belge des Chim. **11**, [1], 13.

See under XVIII A., *page* 700.

Carbohydrates in Foodstuffs, The Quantitative Determination of. W. E. Stone. J. Amer. Chem. Soc. 1897, **19**, 183, 347.

See under XXIII., *page* 703.

PATENTS.

Milk of Cows or other Domesticated Animals, An Improved Treatment, to render it Suitable as Food for Infants and Children. A. Backhaus, Goettingen, Germany. Eng. Pat. 16,016, July 20, 1896.

The milk is separated into cream and skim milk, and the latter made feebly alkaline and treated at a suitable temperature with a ferment, such as trypsin, in sufficient quantity to convert about half the casein into soluble albumin in about 30 minutes, and with a ferment like rennet to precipitate the unacted-upon casein. By raising the temperature to from 70° to 80°, the trypsin and rennet are subsequently rendered inert. The precipitated casein is removed and cream and milk-sugar added to the treated milk, so as to make it resemble human milk. The separated cream may be mixed with water, milk, and milk-sugar, to resemble ordinary milk.—C. A. M.

Butter and other Foodstuffs, An Improved Process for Use in Preserving. Jean, known as Bernard Iribarnégaray, Bayonne, France. Eng. Pat. 3830, Feb. 12, 1897.

The butter or other food material is impregnated with or kept in a solution of 5 to 20 grms. of purified sodium fluoride ("erysoléine") in 1,000 grms. of water.—C. A. M.

(*B.*)—SANITATION; WATER PURIFICATION.

Woolcombers' Suds, The Treatment of. Public Health J., July 14, 1897, 219.

In view of the prominence which has been given in the Bradford Bill, now before Parliament, to the question of the treatment and disposal of "suds" from the woolwashing bowls in combing sheds, considerable interest attaches to a process which has been introduced at the works of Messrs. William Scaife and Co., Ltd., woolcoombers, Laisterdyke. The process is exceedingly simple, and apparently successful. The suds, after being run off from the washing bowl, are allowed to stand for about half an hour in a settling tank, to permit the sand and solid matter to fall to the bottom. The liquor is then pumped into a tank, very much like a wash bowl, in the bottom of which is a system of pipes through which compressed air is forced. About 1 gall. of sulphuric acid to every 700 galls. of suds is added before the "blowing" begins. The violent aëration of the liquor which ensues, quickly brings the grease to the surface in the form of thick foam or froth, and a set of boards, carried on an endless chain, scrape this off and carry it away over one end of the tank. The blowing is continued so long as any froth rises, which is found to be just so long as there is any grease left in the water. The foam, which contains only about 5 per cent. of water, is treated just as the magma obtained in the usual way by precipitation, is dealt with—by pressure in a steam press. The liquor from which the grease has been extracted is treated with lime, in the proportion of about 125—150 grains of slaked lime per gallon of acid water. This neutralises the acid and makes the water quite clear and inodorous, so that it may be turned into the public sewers without being a nuisance. F. W. Richardson estimates the cost for lime at about 3*l.* 10*s.* per million gallons of acid effluent. The apparatus is not costly, and is easily worked; and as the suds can be dealt with—the grease being extracted and the liquor cleared within two hours of their leaving the wash bowl—no nuisance can be created.

PATENTS.

Purifying Air and Separating Matter from Gaseous Fumes, Impts. in Apparatus for. S. Edwards, Manchester. Eng. Pat. 10,942, May 21, 1896.

The gases to be washed or purified are caused to pass through a series of tanks against a stream of water, in which are partially immersed rotating conveyor worms or vanes, against which the gases impinge, and deposit the solid particles.—O. H.

Filtering Sewage, Impts. in Presses for. W. T. and J. Wostenholme, Lancaster. Eng. Pat. 12,735, June 10, 1896.

In presses formed of a number of metal plates having grooved faces covered with sackcloths, trouble is experienced in consequence of the sackcloth becoming forced into the grooves, and seriously reducing the rate of filtration. To prevent this, one or more perforated plates of steel or other metal are interposed between each of the grooved faces and the sackcloths; or two or more of such perforated plates are stayed apart from one another in a cast-iron frame, and held together by bolts, a passage being left in the middle for inlet of the sewage.—L. A.

Furnaces for Consuming, Deodorising, and rendering Innocuous Foul Air, Smoke, and Objectionable and Dangerous Gases generally: Impts. in. S. G. Smith, Plainfield, New Jersey, U.S.A. Eng. Pat. 9905, April 20, 1897.

The gases, ill odours, &c. from gasworks, slaughter-houses, &c., to be consumed, are blown, along with air and steam, into a saddle chamber over the boiler, and pass thence, through apertures ranged around the firebox, into the furnace, where they are ignited. The heat of combustion of these matters is utilised.—C. S.

(C.)—DISINFECTANTS.

PATENTS.

Formic Aldehyde, Impts. in the Production of Vapours of and in Apparatus therefor. J. J. A. Trillat, Paris. Eng. Pat. 17,177, Aug. 1, 1896. (Under Internat. Convention.)

A current of compressed air from a gasometer, pump, or any suitable appliance, is driven through a water-jacketed closed vessel containing methyl alcohol heated to a constant temperature by a gas or spirit flame. The stream of air and alcohol vapour thus produced, is led into the "oxidiser-distributor."

The latter consists of a row or battery of oxidising cylinders, constructed like Bunsen burners, fixed upon a chamber. Each oxidising cylinder contains a tube of retort carbon filled with copper, silver, or platinum wire gauze, or platinised asbestos, &c., and is warmed to start the oxidation, which soon raises the gauze to incandescence; the admission of air is then regulated, so that the incandescence does not exceed a cherry-red.

Several modifications are described, the object in all being to separate the alcohol vaporised from the oxidiser, so that the former may be outside the place to be disinfected, the latter alone being placed inside.—L. A.

Dipping Sheep, An Improved Composition or Mixture for, and for analogous Purposes. R. Mackill, Glasgow. Eng. Pat. 448, Jan. 7, 1897.

Nicotine extracted from tobacco is used instead of tobacco itself. A composition suitable for 1,000 sheep contains 1 lb. of nicotine extract (80 per cent.), 5 galls. of cresol, and 50 galls. of water. This, after thorough mixing, is further diluted with 450 galls. of water.—L. A.

Aldehydes, Impts. in and connected with Apparatus for Producing Vapours of. E. Fournier, Paris. Eng. Pat. 7887, March 26, 1897.

The apparatus consists of a glow-lamp, comprising a reservoir for the reception of the alcohol, an annular wick-tube, a central siphon tube for the admission of air, and a movable screen. Over the wick-tube is a disc of platinum-wire gauze, which can be raised or lowered by suitable means. The lamp is first lighted to raise the gauze to incandescence, and then extinguished, after which flameless combustion is continued by the agency of the platinum gauze. In order to disguise the unpleasant odour, a cup is provided above the flame for the reception of some solid, volatile, aromatic substance; or a disc of asbestos may be used, on which liquid scents may be poured.—L. A.

Grain, Seeds, and other Vegetable Substances; Impts. in the Method of and Means for Disinfecting and Preserving. M. Clajus, Ober-Altwaltersdorf, near Habelswerdt, Germany. Eng. Pat. 11,183, May 8, 1897.

The grain is placed in a revolving wire-gauze drum contained in the upper part of a wooden outer casing, and sulphur is burnt in the lower part. The sulphur dioxide passes up through the grain and escapes through a flue at the top. A very short exposure to the fumes proves a very effective protection against diseases in the grain and the attacks of vermin thereon.—L. A.

XIX.—PAPER, PASTEBOARD, Etc.

Steamers for Wood, Destruction of Flange Bolts in. Papier Zeit. **22**, [58], 2035, July 22, 1897.

In some vertical steamers at the Paper Mills, Neumühle, near Meisbach, it was noticed that the wrought-iron bolts of the upper flanges were badly, and those of the lower flange slightly corroded, whilst the cast-iron plates were almost uninjured. In these steamers the wood is put in at the top, and during the process of steaming, a liquid collects at the bottom, which covers the lower flange. This liquid consists of condensed steam, acids, and other substances extracted from the wood. The authorities of the Chemical Experiment Station at Karlsruhe traced the corrosion to the combined effects of acid, steam, and air. The liquid at the bottom contained 1·65 grms. of acid (calculated as acetic) to 1 litre, 1·21 grms. of which was volatile in steam. That the bolts of the bottom flange were not badly corroded is due to the fact that they were only subjected to the action of a dilute solution of acid, whilst the upper flanges were subjected to the combined action of volatile acid, steam, and air. The plates were only slightly attacked, as cast iron offers more resistance to the action of the acid vapours. The remedy is to carefully examine the screws from time to time and replace the injured ones, but an asphalt varnish might prove of benefit. This corrosion is liable to cause dangerous explosions, and even with copper-lined steamers, the acid vapours might penetrate some defect in the lining.

The authority of the *Papier Zeitung* does not recommend the use of a varnish, but rather the use of phosphor-bronze bolts in place of iron ones.—S. P. E.

Paper Mills, Hot-Water System in. Papier Zeit. **22**, [56], 1962.

As it is detrimental to rags and sulphite pulp to allow the caustic lye to stand in contact with the pulp after boiling, it is preferable to wash it at once in the digester with hot water. If cold water be used, the encrusting or resinous matters are precipitated on the fibres, and are then difficult to remove, and make the pulp difficult to bleach. Since heating the wash water by steam is expensive, and as hot water can be provided in a paper mill without any special expense for fuel, it is better to have a system by which this hot water can be conveyed directly to the digesters and washing engines for washing purposes. By thus using hot water, the temperature of the half-stuff can be regulated so that bleaching can be effected without the use of acid, a stronger fibre being thus obtained, and expense saved. Also in coloured papers, heating by steam is liable to affect the dyes, whilst hot water is not.

The writer noticed that during a flood which caused the paper machines to be run with hot water, though frothing was rather heavy, the pulp did not stick to the top couch roll, as it did when cold water was used.—S. P. E.

Paper, Testing. W. Herzberg. Mitt. k. t. Versuchsanst. zu Berlin, **15**, [1], 85—88.

1. *Permeable Spots in Paper.*—The paper in question—a white writing paper prepared from clean rags and dressed with resin paste—allowed the ink to run through in places and give rise to coloured patches 1 to 3 mm. in diameter on the underside. These places were detected by the white appearance they exhibited on damping the sheet, and from their behaviour towards Congo Red and Methyl Orange in faintly acid solution, revealed the presence of a substance

with an alkaline reaction. On treatment with iodine dissolved in potassium iodide, the initially blue coloration produced by the starch gradually changed to brown from the liberation of iodine, the blue being restored by weak acid, and thus indicating free chlorine (nitrous acid being out of the question), which was found, together with lime, on extracting the paper with water. This behaviour indicated the presence of traces of bleaching powder, probably due to lack of care in cleansing the materials after bleaching.

—C. S.

PATENTS.

Multiple-Pulp Papers, Impts. in the Manufacture of. W. A. Kershaw and the Medway Paper Mills Co., Ltd., Maidstone, Kent. Eng. Pat. 12,682, June 9, 1896.

The two or more differently coloured pulps are contained in separate stuff chests. The second and third layers of pulp are delivered to boxes placed over the wire of the machine at required distances from the end. Each of these boxes is provided with an adjustable opening to deliver the pulp upon the machine wire in an opposite direction to the running of the wire. The vacuum boxes take off the water from the different layers of pulp in the usual manner, forming a single sheet which is passed through the machine and finished in the ordinary way.—S. P. E.

Digesters, Cellulose Boilers, and like Steam Boiling Apparatus; Impts. in. C. Kellner, Vienna, Austria. Eng. Pat. 12,910, June 11, 1896.

The inside of the boiler is coated with shallow hollow iron plates or sections, arranged after the manner of the staves of a barrel, and corresponding to the form of the shell of the boiler. The boiler itself is protected from the effects of the liquid to be boiled, by a layer of refractory material, such as cement, between the shell of the boiler and the before-mentioned plates. These plates are connected by bent pipes at their upper parts, and steam is supplied through a pipe extending through the shell of the boiler. The water of condensation is removed from the lower parts of the sections by pipes connected with a common outlet pipe.—S. P. E.

Fibre, Paper, Tissues, and the like; Impts. in the Method of Waterproofing Fabrics made of. J. Waugh, Bradford, Yorkshire. From Amos and Co., Frankfort a/M., Germany. Eng. Pat. 15,893, July 18, 1896.

The improvements consist in saturating the fabrics with a solution of the waterproofing salts (preferably the oleate or other fatty salts of aluminium) in benzene or other readily evaporating solvent, and subsequently evaporating the solvent.—C. A. M.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

Cerium Oxide, The Purification of. Wyrouboff and Verneuil. Comptes Rend. **124**, 1230—1233.

Besides the lower oxide (formulated by the authors CeO) and the higher one (formulated Ce_2O_4), there exists another, $Ce_6O_7 = Ce_3O_4.3CeO$, very stable, which is formed when cerium compounds are treated in solution with chlorine, according to Mosander, or fused with nitre, according to Debray. Still more stable and more readily formed are compounds of the formula $Ce_3O_4.3MO$, where M = La or Di. These oxides are broken up completely by ammonium nitrate, according to the following procedure:—The oxides obtained by calcining the oxalates are dissolved in warm nitric acid, evaporated to a syrup to remove acid, and then diluted largely with water, so as to have about a 4 per cent. solution of the oxides. Adding now ammonium nitrate till the solution contains some 5 per cent., all the Ce_3O_4 precipitates as basic nitrate, while the oxides MO remain dissolved. The precipitate, well washed with ammonium nitrate, is free from lanthanum and didymium, as well as from oxides of the yttrium group. The solution, precipitated with oxalic acid and the precipitate treated by a repetition of this process, yields another portion of the cerium, and so on. If the mixture of oxides contains as much as 50 per cent. of cerium, it will not dissolve in nitric acid; in this case the oxalates are dissolved in nitric acid, hydrogen peroxide and then ammonia added, and the liquid boiled. The precipitated hydroxide, after washing, is dissolved in nitric acid and the operation conducted as above. The precipitate obtained by adding ammonium nitrate for the first time (75 per cent. of the total cerium), contains all the thorium that may have been present. To remove this, the cerium as oxalate or nitrate as nearly neutral as possible, is treated once or twice with a strong solution of ammonium carbonate containing free ammonia, which dissolves all but a trace of thorium; and this trace is got rid of by crystallisation of the sulphate in neutral solution at 60°. Iron, the only remaining impurity, is now removed by precipitating the chloride or nitrate in warm acid solution with oxalic acid, or by keeping the sulphate for some time at a temperature of 400°—450°.

The atomic weight of such a product is constant; the oxide itself, calcined at a high temperature, is absolutely white.—J. T. D.

Cerium Oxide, Preparation of. H. Moissan. Comptes Rend. **124**, 1233.

A note on the previous paper. Cerium oxide, giving no absorption spectrum in strong solution, is converted into carbide in the electric furnace. This carbide, in fine powder, was treated with weak nitric acid in quantity only sufficient to decompose a portion. A second attack, by a quantity of acid not quite enough to dissolve the whole, gave a solution of pure cerium, the whole of the iron being in the first solution and the whole of the thorium in the unattacked residue.

By simple calcination of the evaporated second portion, a cerium oxide is obtained perfectly white.—J. T. D.

Kola Nuts, Composition of. Report on the Agricult. Work in the Botan. Gardens, British Guiana, 1893-4-5. 121—122.

The composition of the kola nuts from Land of Canaan, Demerara River, is given as follows:—Water, 49·49 per cent.; fat, 0·82 per cent.; albuminoids (containing 0·496 per cent. of nitrogen), 3·1 per cent.; amides, &c. (containing 0·019 per cent. of nitrogen), 0·12 per cent.; caffeïne (containing 0·472 per cent. of nitrogen), 1·69 per cent.; sucrose, 1·93 per cent.; glucose, 0·54 per cent.; pectose, gums, acrid matters, &c., 4·14 per cent.; starch, 24·3 per cent.; digestible fibre, 7·73 per cent.; woody fibre, 4·19 per cent.; iron peroxide, 0·07 per cent.; magnesia, 0·08 per cent.; lime, 0·14 per cent.; potash, 0·69 per cent.; soda, 0·03 per cent.; silica, 0·1 per cent.; sulphuric anhydride, 0·16 per cent.; phosphoric anhydride, 0·34 per cent.; and chlorine, 0·06 per cent. The amount of alkaloids appeared to be somewhat above the average, but the greater part of the caffeïne in fresh kola nuts was found to be in the form of a glucoside and not as the free base. It was found that the acrid taste frequently observed in prepared kola might be avoided to a considerable extent by thinly slicing the fresh nuts and drying them in a current of air at 140°—160° F., finishing the drying at 220° F. The acrid matter appears to become oxidised and there is an increase in the amount of colouring matter in the nuts.—A. S.

Chelidonine. A. J. G. Tyrer. Apoth. Zeit. **12**, 442; Pharm. J. 1897, **59**, 21.

The material operated upon was obtained partly by E. Merck and partly prepared from fresh chelidonium roots. In both cases the base crystallised from alcohol in transparent tables melting at 135° C. Results corresponding to those of Henschke and Gelle (Archiv. Pharm. 1888 and 1890) were obtained with regard to the composition of the base ($C_{20}H_{19}NO_5 + H_2O$) and its behaviour towards reagents. Methyl iodide had very little action on the base at 100° C., but at 130°—140° C., a crystallisable compound was obtained having the composition $C_{20}H_{19}NO_5CH_3I$. By the behaviour of the base towards acetic and benzoic anhydride, it was found that only one of the five oxygen atoms is present in the base as an OH group. The compounds obtained

had the following composition:—mono-acetylchelidonine, $C_{20}H_{18}(C_2H_3O)NO_5$; mono-benzoylchelidonine,—

$$C_{20}H_{18}(C_7H_5O)NO_5.$$

The elimination of water observed by Henschke could not be confirmed. A crystalline oxime, $C_{20}H_{19}NO_4.N.OH$, was obtained by treating the base with hydroxylamine.—A. S.

A Menthoglycol. P. Barbier and G. Leser. Comptes Rend. 1897, **124**, 1308—1311.

When citronellal is shaken with 10 times its weight of 5 per cent. sulphuric acid for 12 hours, it becomes viscous, but remains colourless. The product, smelling strongly of mint, on fractionation under a pressure of 10 mm., yields isopulegol and a body of the formula $C_{10}H_{20}O_2$ boiling at 144°—145° under 10 mm. pressure, and after crystallisation from petroleum spirit, melting at 81°—81°·5 C. The substance appears to be a glycol capable of losing water with formation of isopulegol; the authors give it the name menthoglycol and have re-obtained it from isopulegol.

—A. C. W.

Oil of Celery, Constitution of the Odoriferous Constituents of. G. Ciamician and P. Silber. Ber. 1897, **30**, [11], 1419—1424.

From the high boiling portions of oil of celery, sedanonic and sedanolic acids were isolated (this Journal, 1897, 462). Sedanonic acid, $C_{12}H_{18}O_3$, is a ketone, and from the decomposition products of its oxime the formula—

$$(COOH).C_6H_8.CO.C_4H_9$$

is deduced. It is not changed on heating with dilute sulphuric acid, it is reduced by sodium to the alcohol-acid, $C_{12}H_{22}O_3$, which is stable towards permanganate, and hence is saturated. On oxidation with permanganate, normal valeric, normal glutaric and oxalic acids are obtained.

Sedanolic acid, $C_{12}H_{20}O_3$, is reduced by sodium and alcohol to the same saturated alcohol acid, but by the action of sodium amalgam sedanonic acid gives an oily acid of the same constitution as sedanolic acid, but not identical with it. Sedanolic acid oxidised by permanganate in the cold, is chiefly converted into o-oxyamyl-benzoic acid, the lactone of which also possesses a celery-like smell.

In oil of celery, sedanolic acid is probably contained as the anhydride—

$$C_6H_8\left\langle\begin{matrix}CH.C_4H_9,\\ \quad >O\\ CO\end{matrix}\right.$$

tetrahydrobutyl phthalide. Bodies of similar constitution appear to have the celery odour.—A. C. W.

Caffeïne, Some Properties of. E. Tassilly. Bull. Soc. Chim. 1897, **17**, 596—599.

An examination of certain properties of caffeine which are of importance in its determination.

1. Caffeine hydrate does not lose the whole of its water at 150°, the temperature at which it is partially volatile. At 110° the anhydrous substance does not lose weight, but the hydrate heated to this temperature loses 6·93 per cent.

2. When an aqueous solution of caffeine is evaporated on the water-bath, none is volatilised with the steam.

3. In aqueous solution caffeine is decomposed by lime at 100°, ammonia being slowly evolved. Under the same conditions magnesia causes no evolution of ammonia.

4. The partition of caffeine between its solvents ceases to be normal when the liquids contain extractive substances as in the treatment of natural products.—A. C. W.

Dextro-rotatory Licarhodol. P. Barbier and G. Léser. Bull. Soc. Chim. 1897, **17**, 590—596. See this Journal, 1896, 613.

The existence of this alcohol having been doubted, the authors now give a more detailed account of their work.

Pure licareol, boiling at 86°—88° C. under 10 mm. pressure and with the rotation $a_D = -12°·45$, was heated at 150°—160° for 8 hours with its own weight of acetic anhydride. After removal of acetic acid and distillation under 10 mm. pressure, four fractions were obtained, 50°—80° containing terpenes, 80°—105° containing unaltered licareol, 105°—130° containing the ester, and a higher fraction containing an ether $(C_{10}H_{17})_2O$, boiling at 145°—150° under 10 mm. The ester, which boils at 119°—120° under the same pressure, was saponified by the gradual addition of alcoholic potash, excess being avoided. The alcohol was precipitated by water; after two distillations it boiled at 112°—114° under 9 mm., had the density 0·901 at 0°, the rotation for sodium light $a_D = +1° 8'$. The yield was 800 grms. from 3 kilos. of licareol.—A. C. W.

Asafœtida, Composition of. J. Polásek. Archiv. der Pharm. 1897, [2], through Pharm. Zeit. 1897, **42**, 240.

The percentage composition of pure tears of amygdaloid asafœtida was found to be:—Resin, soluble in ether (ferulic acid ester of asaresinol-tannol), 61·40; resin, insoluble in ether (free asaresinol-tannol), 0·60; gum, 25·1; volatile oil, 6·7; vanillin, 0·06; free ferulic acid, 1·28; moisture, 2·36; and foreign matter, 2·5.

The formula for asaresinol-tannol is $C_{24}H_{34}O_5$. It gives a benzoyl derivative, $C_{24}H_{33}O_5.C_6H_5CO$, and an acetyl derivative, $C_{24}H_{33}O_5.CH_3CO$. Asaresinol-tannol contains therefore an hydroxyl group, $C_{24}H_{33}O_4.OH$. On hydrolysing with sulphuric acid, umbelliferone is obtained.

—J. O. B.

Tolu Balsam, Spurious. J. O. Braithwaite. Pharm. Journ. 1897, **58**, 307.

The price of Tolu balsam being at present very high, and the genuine article apparently but rarely met with in the drug market, it is not surprising that fictitious samples are occasionally imported. A considerable quantity of the balsam under notice has recently reached the port of London.

It differs very markedly in physical characters from the genuine substance, being soft and much more viscid, especially when chewed. Under the microscope but few crystals were to be seen in a thin layer of the substance. With genuine Tolu balsam, the field is crowded with them. Extracted with successive quantities of boiling water, it yielded 1·15 per cent. of crystalline acid on cooling. This acid was fractionally crystallised from boiling water, when each fraction was found to melt sharply at 133° C., and to consist wholly of cinnamic acid. Genuine Tolu balsam, under similar treatment, gave 4·2 per cent. of cinnamic acid. Treated with carbon bisulphide, the doubtful sample gave 61·4 per cent. of soluble matter as a fragrant, brown, transparent, viscid mass, with a total acid number of 274 when saponified with alcoholic potash. Genuine Tolu balsam gives with bisulphide a perfectly white crystalline residue, the total acid number of which should not fall below 300. It is evident that the product referred to cannot be regarded as the true official balsam of Tolu derived from *Myroxylon toluifera*, and therefore it should not be used for medicinal purposes; it may be derived from some nearly allied species, since it differs materially from substituted or factitious samples previously reported on by the author and others.

—J. O. B.

Argentol. Pharm. Zeit. 1897, **42**, 243.

"Argentol" is a synthetic antiseptic, consisting of a combination of silver with quinosol. It forms a yellowish almost odourless powder, sparingly soluble in water or in alcohol. Its composition is $C_9H_5N(OH)SO_3.Ag$.

—J. O. B.

Sodium Tetraborate, So-called, and Magnesium Tetraborate. Pharm. Zeit. 1897, **42**, 191, 201, 223.

Considerable confusion has arisen through the incorrect application of the term tetraborate of sodium to several distinct preparations of borax and boric acid. Chemically the term tetraborate of sodium can only be correctly applied to borax, and yet it has been used by Kafemann to designate a preparation of borax 5·5 parts, with boric acid 10 parts. Conrady states that the same substance should be prepared from two molecular weights of boric acid and one of borax. Janicke-Görlitz again describes the salt as being prepared from equal parts by weight of borax and boric acid dissolved in an equal quantity of water. This last preparation has the property of crystallising out very

slowly from warm saturated solutions, so that when injected into a cavity, such as the aural passage, it slowly solidifies, and thus forms a permanent antiseptic plug.

Magnesium tetraborate is prepared as a solution or as a white powder, soluble in four parts of hot water. Kuchenthal gives the following directions for its preparation:—Mix boric acid, in powder, 25 parts; magnesia, 4 parts; with water, at least, 150 parts; warm until complete solution is effected, filter from any excess of magnesia left insoluble; this solution is intended for application to the diphtheritic membrane or for internal administration; when required in the powdered form, the same ingredients are merely rubbed together with a little water and dried at a low temperature.—J. O. B.

Ether, Aldehyde in, Method for the Determination of. Maurice François. J. Pharm. Chim. 1897, 5, 521.

See under XXIII., *page* 704.

XXII.—EXPLOSIVES, MATCHES, Etc.

Chloride of Nitrogen. W. Hentschel. Ber. 1897, 30, [11], 1434—1437.

Indifferent solvents diminish the explosive nature of chloride of nitrogen. A 10 per cent. benzene solution can be lighted or poured over a red-hot plate without remarkable appearances of decomposition. The substances which cause chloride of nitrogen to explode, react very rapidly on its solutions, but mainly in a controllable manner; the action of phosphorus on a concentrated solution alone assumes an explosive character. The solutions are sulphur-yellow, strongly refractive liquids, quickly decomposing in sunlight, but only slowly in the dark, the rate of decomposition being but little dependent on the nature of the solvent. In the decomposition of the benzene solution, free nitrogen and benzene hexachloride are formed; from the carbon bisulphide solution chloride of sulphur is obtained; from chloroform, chlorine, hydrochloric acid, and ammonium chloride. Chloride of nitrogen dissolved in ether is completely converted into ammonium chloride.—A. C. W.

PATENTS.

Pyroxylin Smokeless Explosives, Impts. in the Manufacture of. H. Maxim, London. Eng. Pat. 16,858, July 29, 1896.

The inventor forms pulped guncotton, of varying degrees of solubility in a mixture of ether and alcohol, into sheets of paper which, after having been treated by the solvent, will adhere when pressed together, and thus a body of any desired thickness can be built up, which it is claimed will not warp or crack in drying. The density of the explosive can be modified by altering the proportions of the "soluble" and "insoluble" nitrocotton.—W. M.

Explosives, Impts. in the Preparation of. L. Roux, Paris, France. Eng. Pat. 4672, Feb. 20, 1897.

The inventor produces explosives for blasting purposes which he calls "belterines," by mixing ammonium cresylate with ammonium nitrate in the proportions 4½ to 22 per cent. of the cresylate to 78 to 95½ per cent. of the nitrate. Five per cent. of potassium chlorate dissolved in water is then introduced into the mixture and thoroughly incorporated with it, and after drying, the composition is compressed into cartridges. Another composition consists of a mixture of the ammonium cresylate and nitrate as above, with about 10 per cent. of nitroglycerin containing 1 per cent. of nitrocotton. Detonators containing 1—1·5 grms. of fulminate should be employed.—W. M.

Explosives, Impts. in. O. F. Carlson, Stockholm, Sweden. Eng. Pat. 10,362, April 26, 1897.

The explosives patented consist of from 40—98 per cent. by weight of ammonium perchlorate, and 60—62 per cent. of inflammable substances such as astatki, naphthalene, paraffin, charcoal, sugar, &c.—W. M.

Explosive Compound, A New. W. Majert, Grünau, near Berlin. Eng. Pat. 13,552, June 1, 1897.

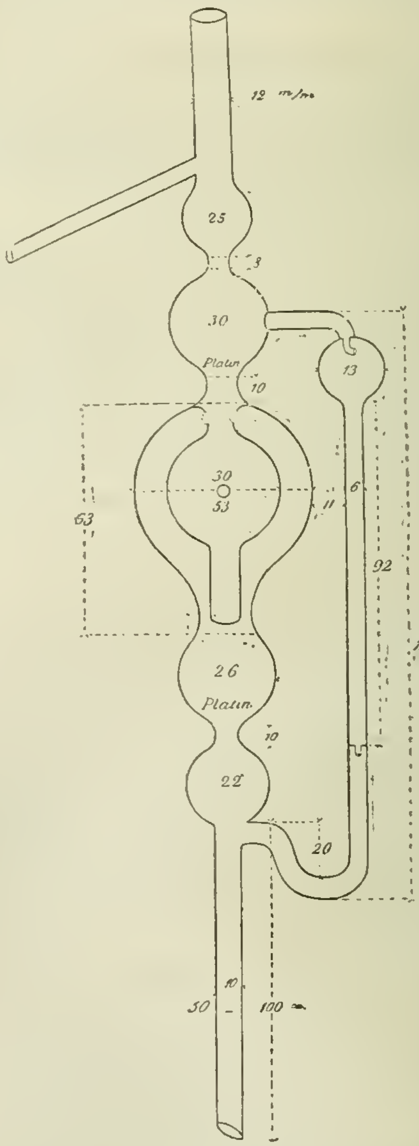
The use of ammonium bichromate is claimed as an explosive agent, the explosion being brought about by a detonator.—W. M.

XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

Fractional Distillation of Benzine [Petroleum Spirit], New Apparatus for the. L. Singer. Chem. Rev. Fett- u. Harz-Ind. 4, [8], 108.

This apparatus was designed by the author to obviate the disadvantage, inherent in the dephlegmators of Glinsky and Lebel, of the excessive height through which the distilled vapours have to ascend, and which has now, in the present



instance, been reduced to about 20 cm. Platinum gauze is employed to close the bulbs, the glass balls being dispensed with, as formerly used. These balls interfered with the distillation, besides being a source of danger of breakage in transit.—C. S.

Gas Analysis, Industrial: Apparatus for. L. Vignon. Comptes Rend. **124**, 1241—1246.

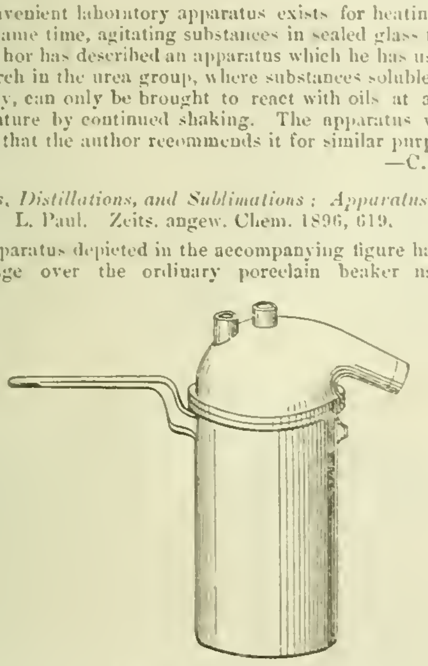
An apparatus similar in general construction to that of Orsat, consisting of a measuring vessel with water reservoir, into which the gas can be aspirated, and from which it can be driven into one or more absorption vessels, or into a mercury eudiometer for explosion. Examples are given of the procedure in an analysis, and of the calculations necessary to allow for the volume of the capillary connecting tube.—J. T. D.

Sealed Glass Tubes, Apparatus for Simultaneously Heating and Shaking in. E. Fischer. Ber. **30**, 1485—1487.

No convenient laboratory apparatus exists for heating and at the same time, agitating substances in sealed glass tubes. The author has described an apparatus which he has used in a research in the urea group, where substances soluble with difficulty, can only be brought to react with oils at a high temperature by continued shaking. The apparatus works so well that the author recommends it for similar purposes. —C. M.

Fusions, Distillations, and Sublimations; Apparatus for. L. Paul. Zeits. angew. Chem. 1896, 619.

The apparatus depicted in the accompanying figure has the advantage over the ordinary porcelain beaker usually



employed in the laboratories of colour factories, &c., in having a handle, by which it is secured, by means of a clamp, in any desired position.—T. A. L.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Arsenic, Notes on the Estimation of, in presence in Copper. G. L. Heath. Eng. and Mining J. 1897, **63**, 663.

The author recommends the following method for the estimation of traces of arsenic:—The precipitate of arsenious sulphide and sulphur obtained in a regular analysis, or by one of the distillation methods, is digested with 20 c.c. of fuming nitric acid until the sulphur is dissolved, and then evaporated with 5 c.c. of pure concentrated sulphuric acid until white fumes are evolved. The liquid is then cooled and diluted with about 2 volumes of water. A Marsh's hydrogen generater is prepared, and 25 grms. of chemically pure zinc (granulated to pass a 20-mesh sieve and be retained by one of 30 meshes to the inch) are placed in the bottle and covered with water to the depth of 1 in. A calcium chloride tube is connected to the bottle and then a piece of combustion tubing ($\frac{1}{4}$ in. internal diameter and about 2 ft. long). 4 ins. of the tube are maintained at a cherry-red heat during the evolution of gas, and this portion of the tube is surrounded with a double layer of coarse brass or copper gauze to prevent overheating and sagging. The latter portion of the tube is constricted at three or four points, and the evolution of hydrogen, which is started by sulphuric acid (diluted with an equal volume of water and one drop of platinic chloride), is maintained at such a rate that the burning jet of hydrogen at the end is only $\frac{1}{4}$ in. high. After lighting the jet, the solution to be tested, after mixing with a solution of 0·5 grm. of chemically pure tin in strong hydrochloric acid, is gradually introduced into the generating bottle. The addition of the solution should take about $1\frac{1}{2}$ hours, and the last traces of arseniuretted hydrogen are driven forward by adding dilute sulphuric acid through the funnel, until nearly all the zinc is dissolved. The tube is then cooled, the short portion containing the film of pure arsenic cut off, weighed, and the weighing repeated after dissolving or volatilising the arsenic film.

The author obtained perfectly concordant results by this method for amounts of arsenic less than 6 mgrms., on making check analyses. The addition of tin chloride is important, as in the absence of it, the zinc retains a certain amount of arsenic.—A. S.

Nitrites, A very Sensitive Reaction for, and Quantitative Estimation of, Colorimetrically. E. Riegler. Zeits. Anal. Chem. **36**, 377—378.

Naphthionic acid, proposed as a test for nitrites (this Journal, 1897, 261), is not always procurable of constant quality, and its value as a reagent is thereby impaired.

A reagent is now prepared which is 20 times as sensitive as naphthionic acid, and not subject to this variability.

2 grms. of chemically pure sodium naphthionate and 1 grm. of pure β-naphthol are added to 200 c.c. of distilled water, vigorously shaken, and filtered. The solution is colourless, and may be kept in the dark without change. 10 c.c. of the solution to be tested are placed in a test tube, 10 drops of the naphthol reagent added and 2 drops of concentrated hydrochloric acid, and the mixture well shaken; 20 drops of ammonia are now allowed to flow down the tube, held in a slanting position, when a red ring appears at the junction of the liquids. On shaking, the whole fluid becomes more or less red or rose-coloured, according to the quantity of nitrous acid present. As dilute solutions of the reagent show a violet-blue fluorescence, the colour must be viewed by transmitted light. The reaction is uncommonly sensitive, showing 1 part of nitrous acid in 100 millions. It may be shown in 1 c.c. of rain water or 1 c.c. of saliva. The reaction depends on the conversion of naphthionic acid by nitrous acid into diazonaphthalene sulphonic acid, which forms with β-naphthol and ammonia a red azo dye.

The standard solution for applying the reaction quantitatively is made by dissolving 0·406 grm. of silver nitrite in hot water, adding some sodium chloride, and, when cool, making up to a litre. 100 c.c. of this solution contains 0·0001 grm. of N_2O_3. 100 c.c. are placed in a flask, 20—30 drops of the reagent added and 4 drops of concentrated hydrochloric acid. It is then repeatedly shaken, and 20 drops of ammonia run in. On shaking, the mixture becomes rose-red in colour, and serves for comparison with the solution to be tested. It must be freshly prepared for each determination.

By this method 0·00005 grm. of N_2O_3 in 1 litre of water may be estimated.

The reagent may be also prepared in the solid form by mixing equal parts of naphthionic acid and β-naphthol intimately in a mortar. A pinch of the powder will serve instead of the 20 drops of reagent. The powder keeps absolutely unchanged, and is as sensitive as the solution. —L. J. de W.

Aluminium Utensils, Analysis of. M. Balland. Comptes Rend. 1897, **124**, 1313—1315.

The author has devised a simple method for the purpose of the examination of the vessels supplied to the French army, the processes already described being considered too long for the purpose (see this Journal, 1896, 136, 830). The aluminium vessels should contain 99—99·5 per cent. of the metal, and those composed of the copper alloy, 2—3 per cent. of copper.

"Pure" Aluminium.—0·5 grm. of the metal cut into thin strips is subjected to the action of 50 c.c. of water and 10 c.c. of pure hydrochloric acid. When the action slackens, the dish is gently warmed until all evolution of gas has ceased. Under these circumstances, carbon, silicon, and copper are unattacked; they are filtered off, washed, ignited, and weighed. To half of the filtrate, 10 per cent. potash is

added in quantity, and the mixture warmed for half an hour; the precipitate is redissolved on the filter by dilute hydrochloric acid, and the solution again treated with potash; the ferric hydrate is now filtered off, washed, ignited, and weighed. In the second half of the filtrate, aluminium and iron are precipitated together by ammonia.

Copper Alloy.—Two quantities of 0·5 grm. are separately attacked by 5 c.c. of hydrochloric acid and 50 c.c. of water, the end of the reaction being accelerated by warming. The copper is filtered off from one solution, ignited, and weighed. In the filtrate, iron and aluminium are determined as before. The copper in the second dish is dissolved by adding 25—30 drops of nitric acid. The residue now contains carbon and silicon; this may be filtered off, ignited, and weighed.
—A. C. W.

Tin in Tin-Plate, Estimation of. H. Masthaum. Zeits. angew. Chem. 1897, 329.

The process suggested by Lunge and Marmier (this Journal, 1895, 822) has several disadvantages, involving, as it does, complicated apparatus, and requiring great care during the heating. The following method is preferable:—25 grms. (or even 100, if a fair sample cannot easily be obtained) of the material are boiled for five minutes with 50 c.c. of 10 per cent. hydrochloric acid, and the liquid poured off into a 250 c.c. flask; the operation being repeated with fresh acid two to four times, until the iron is seen to be completely stripped. The solution does not need filtering, but is at once made up to the mark, and 50 c.c. treated with ammonia in a 100 c.c. flask till stannous hydrate begins to fall, when 10 c.c. of yellow ammonium sulphide are added, the whole shaken, filled up, and filtered. 50 c.c. of the filtrate are diluted with water, all the tin sulphide thrown down by acetic acid, and, after standing over night, the precipitate is brought on to a filter by the aid of 10 per cent. ammonium acetate. As, owing to the certain absence of copper, ammonium instead of potassium sulphide is employed, the washing need only be very slight; in fact, 50 c.c. of the sulphide solution can be directly evaporated and ignited in a porcelain crucible, but the former method is preferable. The precipitate is finally heated with fragments of ammonium carbonate until the ash is white. The results quoted by the author are satisfactory; and he notes that the percentage of tin in tin-plate usually lies between 1·94 and 3·07.—F. H. L.

ORGANIC CHEMISTRY.—QUALITATIVE.

α- and β-Naphthol, A Reaction to distinguish between. E. Léger. Bull. Soc. Chim. **17**, [11], 546—547.

A saturated solution of the naphthol to be tested is made by rubbing in a mortar with water for some time and filtering. To 10 c.c. of this solution are added a couple of drops of a sodium hypobromite solution, made by diluting 30 c.c. of sodium hydroxide solution of sp. gr. 1·33 with 100 c.c. of water and adding 5 c.c. of bromine. With α-naphthol a violet colour is struck or even a precipitate formed, while with β-naphthol a yellow tint is first seen, which becomes greenish, "dead-leaf coloured," and finally yellow again. The reaction with the α-compound is very much more delicate than that with the other, so that by diluting the solution of β-naphthol before applying the test, one can detect in a sample even 1 per cent. of the α-compound. The naphthol solutions, as well as the hypobromite, must be freshly prepared.—J. T. D.

Aldehydes and Ketones, Colour Reactions of. Béla von Bittó. Zeits. Anal. Chem. **36**, 369—376.

Aldehydes and ketones give colour reactions with salts of meta-diamines. A 0·5 to 1·0 per cent. aqueous or alcoholic solution of a chloride of meta-diamine is prepared, and a few c.c. of the solution are poured into an aqueous or alcoholic solution of the substance to be tested. The alcohol must be free from aldehyde and ketone. In a few minutes the reaction takes place with an intense greenish fluorescence, and reaches its maximum intensity in two hours. The colour disappears on adding alkali, is restored on acidifying, but is enfeebled by mineral acids. The reaction occurs if the formyl or carbonyl group is not united to a completely substituted hydrocarbon group.

The author also examined Schiff's fuchsin sulphurous acid reaction, and obtained results differing in many particulars from those of other observers. He also finds that the reagent alone always shows a distinct bluish-violet coloration on cooling after being boiled, so that it is not conclusive of the presence of an aldehyde or ketone if a faint colour appears after boiling. Grape-sugar gives no reaction.—L. J. de W.

Tea, A New Adulterant of. [*Detection.*] J. Delaite and H. Lonay. Bull. Assoc. Belge des Chim. **11**, [1], 13—21.

The sample of (green) tea examined was found to contain 68 per cent. of extraneous leaves rolled into a globular shape. On steeping in water these unrolled less readily than the true tea. A bluish-grey powder separated from the leaves under water, and was found to contain manganese in addition to ordinary ash constituents, but no copper or lead. The microscopic examination of the powder led to the conclusion that earth and indigo had been used to imitate the colour of pure tea. In general form the extraneous leaves resembled *Thea chinensis*, but differed therefrom in microscopic character, as also from *Epilobium*, *Vaccinium arctostaphyllos*, and other known vegetable adulterants, and, in fact, could not be botanically identified. The authors, however, consider that it is sufficient in practice for the chemist to detect the presence of extraneous adulterant leaves in tea, without specifying their nature.—C. S.

Sausages, Detection of Artificial Colour in. H. Bremer. Forschungs-Ber. 1897, **4**, [2], 45—48.

Cases have frequently occurred in the author's experience in which artificial colouring matter could be detected microscopically in the fibres, but could not be extracted by any of the usual solvents, such as alcohol, amyl alcohol, or a mixture of glycerin and alcohol. In such cases a mixture of equal parts of glycerin and water, as recommended by Klinger and Bujard, is the best solvent. The finely divided sausage is heated on the water-bath for several hours with two volumes of this mixture (slightly acidified), the yellow-coloured liquid freed from fat and filtered, and the colouring matter precipitated as lake by the addition of alum and ammonia. On placing the test tube before the spectroscope, the absorption lines of carmine lake, lying between *b* and D, may easily be identified. Since carmine lake gives a red solution with hydrochloric, nitric, and tartaric acids, whilst the acid solution of the sausage colouring matter is yellow, it is suggested that the carmine is present in some other form than lake, possibly combining with the preserving agent to form a compound insoluble in alcohol.—C. A. M.

Cheese, Detection of Margarin in. H. Bremer. Forschungs-Ber. 1897, **4**, [3], 51—53.

The old method of extracting the fat from cheese with ether is, in the author's opinion, open to the objection that impurities are simultaneously extracted. By Henzold's process, in which the fat is extracted by shaking the cheese with 5 per cent. potash, all the free fatty acids, as well as those in combination with the ammonia, are saponified. The method recommended as the most satisfactory is to shake the finely divided cheese with water at 20°—30°, preferably acidified with sulphuric acid (5:200), and to apply centrifugal force, when the fat rises to the surface, whilst the albuminoid substances coagulate at the bottom. In addition to the determination of the usual constants, valuable information may be obtained from the iodine number of the liquid fatty acids separated by the process described by the author (this Journal, 1897, 641). In a number of experiments with pure butter fat, the iodine value of the liquid fatty acids did not exceed 95, even when the cows had been fed on maize oil-cake. A sample of Edamur cheese, when thus examined, yielded a fat the liquid acids of which had an iodine value of 93·35, whilst the values obtained with two samples of margarin cheese (Rodamur and Backstein) were 110·3 and 109·0 respectively.
—C. A. M.

ORGANIC CHEMISTRY.—QUANTITATIVE.

Paraffin in Lignite-Tar Products, Estimation of. H. Eisenlohr. Zeits. angew. Chem. 1897, 300 and 333.

This paper consists of a detailed examination into the processes devised by Zaloziecki and Holand for the determination of paraffin in lignite-tar oils; Holde's method not being dealt with, as it is designed simply for petroleum products which give a residue insoluble in alcohol. As the result of a large number of experiments, which he records at length, the author concludes that all the methods which have hitherto been published are but moderately accurate even when only paraffins of a high melting point have to be determined; where, however, softer paraffins are in question, they become valueless, for the following reasons:—(1) The temperatures (100° or 125° C.) at which it is usually recommended to dry the products are too high, and cause considerable loss by volatilisation; for instance, a paraffin melting at 29·5 may lose 10 per cent., and one melting at 38°, 1·4 per cent. at 100° C.; while at 125°, a 33 paraffin will lose 18·5, and a 48° sample 1·8 per cent. Moreover, the exact amount of these losses is entirely dependent on the time of heating, and also on the size and shape of the vessel containing the paraffin. (2) The solubility of the lower paraffins in ethyl alcohol is greater than that stated by Holand (this Journal, 1894, 286); for both Holde and the present author agree that 100 c.c. of 99·5 per cent. spirit dissolve 0·03 grm. of a specimen melting at 55°—56°. Since lignite tar contains paraffins of a far lower melting point, and since their solubility in alcohol rises as their melting point falls, it is clear that 0 C. is not sufficiently low for the working temperature.

Eisenlohr finds that all the lignite-tar paraffins melting between 31·5° and 60° C. which do not contain more than 14 per cent. of oil, may be accurately estimated by the use of ethyl alcohol of 80° Tr., provided the operation is carried out in the manner indicated. About 0·5 grm. of the substance is dissolved in 100 c.c. of absolute ethylic alcohol, 25 c.c. of water added, and the whole cooled to −18 or −20° by means of a freezing mixture. After a short time it is thrown on a paper (tared under precisely the conditions afterwards adopted in drying the paraffin), which is placed in a filtering apparatus, such as that described by Holde (this Journal, 1897, 362, Fig. 1), but connected with a pump. The precipitate is washed with alcohol of 80° Tr., previously cooled to −18°, until the filtrate is no longer rendered turbid by additions of water, then put into an exhausted desiccator containing sulphuric acid, and heated to 35° or 40° C. for 6 or 8 hours, till its weight remains constant.

Treated in this way, one sample of a pure paraffin melting at 31·5° C., after being mixed with 6·19 per cent. of a paraffin-free oil, gave 93·49 per cent. of true paraffin.

—F. H. L.

Asphalt, Testing Artificial. M. Gary. Mitt. k. t. Versuchsanst. zu Berlin, **15**, [1], 74—80.

The samples examined were made by Zoeller, Wolfers, and Dröge, of Berlin, from coal-tar melted along with 2 to 3 per cent. of sulphur and mixed with about 2·5 per cent. of fine, sifted bleaching powder, the whole being then incorporated by heating and stirring with granulated and ground blast-furnace slag, and finally compressed, under 200 atmos. pressure, into blocks 25 by 12·5 by 5 cm. For the sake of comparison, the samples (A) were tested side by side with similar samples (B) prepared in a knee-lever press and with blocks (C) of Sicilian natural asphalt.

In testing the (heat) conductivity of the sample A, a hole was bored in the longest side, as far as the centre, and filled with mercury in which a thermometer was immersed, and the block heated in a drying oven. At the end of four hours, the temperature of the oven being 71° C., that of the block registered 66° C., and that of a block of wood of equal size and under the same conditions, 61° C. With an oven temperature of 66° C. and the same exposure, the temperatures were 57° C. and 55° C. respectively. In point of radiation on cooling, observed at a room temperature of 17° C., both asphalt and wood receded about 1° C. in five minutes, about 5° C. in 10, and 10° C. in 20 minutes.

For testing the resistance to pressure at high and low temperatures, such as would prevail in practice (street paving), the blocks were sawn in half and heated in an air chamber immersed in a water-bath over a sand-bath, the temperatures being regulated to 80 C., 40 C., and 18 C. respectively. The blocks were embedded in wooden frames to prevent radiation of heat on all sides except the face, which was exposed to the air of the chamber. For the cold test they were kept in a cold chamber containing a Linde refrigerating machine producing an air temperature of about −15 C. In all cases the time of exposure was the same, *viz.*, three hours. The blocks were then placed in an Amsler-Lasson press and pressure applied—to a sharp-edged steel cylinder 4·2 cm. in diameter (13·85 sq. cm. section) and 3 cm. high, resting freely on the block—until breaking resulted.

The average results obtained were as follows:—

Temperature.	Pressure, in Kilos. per Sq. Cm., required to produce (1) a Visible Impression, (2) Breakage.					
	Sample A.		Sample B.		Sample C.	
	1.	2.	1.	2.	1.	2.
−15° C.	153	391	159	403	53	111
+18° C.	45	127	38	67	21	32
+40° C.	38	95	22	44	9	19
+80° C.	25	54	19	27	9	13

It is therefore evident that whilst in the cold the two samples of artificial asphalt, A and B, differ but little in their ability to stand pressure, A is considerably the better in this respect in the warm, and both far surpass the natural asphalt C.

It is considered that the patentee's idea that the process should be worked by municipalities, who could supply the chief materials from the by-products of their own gasworks, might be of importance in districts where stone is scarce.

—C. S.

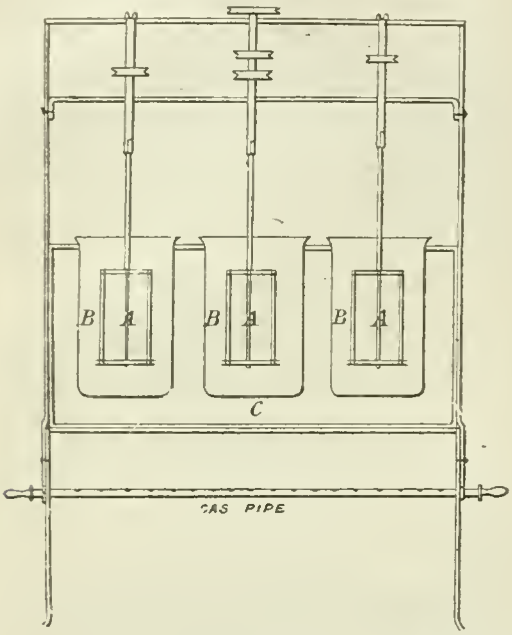
Indigo Testing. J. Grossmann. J. Soc. Dyers and Colourists, 1897, 124—128. (See also this Journal, 1885, 189 and 491; 1887, 455; 1888, 425; 1889, 640; 1891, 488; 1892, 63; 1893, 67; 1894, 426; 1895, 893; and 1896, 475.)

In reviewing the various methods that have been proposed for the assay of indigo, the author concludes that, apart from all other objections which he raised to specific processes, all those which are based on a standard indigotine are unreliable, inasmuch as no two samples of indigotine are of the same strength or composition, owing to the difficulty of preparing this substance in a pure state. Rawson's hydrosulphite process (this Journal, 1885, 489), gives good results, but "requires concentrated attention" and a somewhat complicated apparatus; whereas his permanganate process, whilst giving fair results with high-class indigos, is not to be relied on in the case of lower qualities. Moreover, the indigo red always affects the tinctorial powers of the indigo, and should be taken into account in estimating the value of the latter.

The author has worked out a process based on comparative dye tests, the indigo being first purified and then converted into its sulphonic acid for the purpose. He has also devised an apparatus for carrying out the process, consisting of a reel A, made of glass or porcelain, arranged in such a way that it can be made to revolve in a beaker B, which is placed in a water-bath C. Several of these little machines may be geared together, as shown, and driven by a water turbine or some other motor.

The test is performed as follows. One grm. of the sample to be tested, ground to a fine powder in an agate mortar and mixed with about five times its weight of ground glass, is placed on a dry filter and washed with hot dilute hydrochloric acid (one part of strong acid to four parts of water), until the filtrate is no longer coloured brown. The filtrate is then made up to a definite volume. The sample is next washed with a hot solution of sodium hydrate

(containing 2 per cent. of Na_2O), until the washings are no longer brown, and the filtrate is again made up to a definite volume. The contents of the filter are then washed with



hot water to remove the soda, dried in an air-bath at 100° C., and ground in an agate mortar to a uniformly fine powder. This powder is then transferred in small portions at a time to a porcelain crucible containing 25 c.c. of pure sulphuric acid (sp. gr. 1·85), stirring well with a glass rod after each addition, and is heated for $1\frac{1}{4}$ hours in an air-bath at 95°—100° C., the contents being stirred from time to time. When cold, the mixture is washed into a litre flask, filled up with water to the mark, and filtered.

Five grms. of woollen yarn are now wound on to the reel of the dyeing machine, the beaker is filled with water until the latter just covers the top of the reel, and 1 c.c. of ammonia of sp. gr. 0·880, is added to the same. In this dilute ammonia solution, the yarn is scoured for half an hour at 100° C., the reels being kept revolving. After rinsing, the vessel is again filled with clean water, and 2 c.c. of a 10 per cent. solution of aluminium sulphate and 10 c.c. of the prepared indigo solution are added to the bath. The wool is kept revolving in this until the bath is nearly exhausted (about $1\frac{1}{2}$ hours), taken out, rinsed, and dried. The exhausted bath is kept for reference, and compared with the exhausts of previous and subsequent dye tests.

In testing several samples of different values, quantities representing equal money's worth are taken, in which case the sample giving the fullest shade would be the cheapest. —I. S.

Low Iodine Number of Linseed Cake, Cause of the. G. Fassbender and J. Kern. Zeits. angew. Chem. 1897, 331.

The authors find that by cold pressure, a pure linseed oil may be obtained even from adulterated seed, as the resulting cake retains the greater part of the oil derived from the foreign *Cruciferæ* present. It is impossible, therefore, from a mere determination of the iodine number of the pressed cake, to calculate the amount of impurity existing in the original linseed.—F. H. L.

Beeswax, The Iodine Value of. R. Glode Guyer. Pharm. J. 1897, **58**, 308.

From a number of experiments with genuine English beeswax, the author concludes that the iodine absorption number, obtained in the ordinary manner with Hübl's solution, is a useful factor in the analysis of yellow wax; but is of no service in the case of white wax.

Finding that various authorities differed as to the limits of this figure for pure wax, a number of determinations were made, which point to a mean of 8·5 as the true iodine absorption number, and indicate that the standard should be from 8 to 9, and not a higher figure as stated by others. The test is useless with white wax, since the process of bleaching almost wholly removes, or modifies, the iodine absorbing bodies.—J. O. B.

Linseed Oil [for Varnish-making], Valuation of. W. Lippert. Zeits. angew. Chem. 1897, 306.

The author protests against Amsel's statement (this Journal, 1895, 605), that rosin itself as well as rosin oil and mineral oil can be detected in linseed oil by determination of the saponification number, and by the alcoholic soap solution becoming turbid on dilution with water. It frequently happens that aqueous solutions of pure linseed soaps show a faint opalescence, and, on the contrary, that such liquids even when containing a large excess of rosin, will remain clear for a considerable time; while, moreover, many varieties of colophony which at first give cloudy solutions, after repeated melting, lose this property almost entirely. In a recent pamphlet Amsel says that linseed oil alone should dry on glass in three days, or in 24 hours if 5 per cent. of a drier be added. Apart from the influence of light and temperature on this test, the speed of drying is strictly dependent on the amount of metallic drier employed; the experiment, therefore, must only be carried out in the presence of a definite quantity of a drier of known composition. Amsel's figures also tend to prove that a varnish containing 1 per cent. of a manganese resinate dries more quickly than if it contains 3 per cent., or if it has been boiled with 3 per cent. of red lead; but this appears to be a mistake, and the small proportion of rosin that exists in these products certainly does not hinder their desiccation.

Lippert holds that an oil suitable for the varnish maker must give a good drying test, must bear heating to 300° C. without the formation of any flocculent deposit—remaining perfectly clear—must yield the correct saponification number, as well as a transparent, or only faintly opalescent, solution on diluting the soap with water, and must be tested separately for rosin according to the ordinary methods.—F. H. L.

Sugar Products, Estimation of Lime in, by Soap Solution. H. Pellet and O. Castels. Bull. Assoc. Chim. **14**, [11], 1100—1103.

The use of soap solution in testing for lime salts in products of sugar works appears to give good results, in agreement with those obtained directly by ammonium oxalate, within 10 per cent. more or less. Certain cane-sugar juices, however, which gave no precipitate with ammonium oxalate, showed with soap solution the presence of an alkali combining with soap, which was not lime, and other cases gave great differences in the results obtained by the two methods. This was found to be due to the presence of magnesia derived from the lime used. It is therefore necessary to check each series of tests by a determination with ammonium oxalate and, if necessary, to use a factor for reducing the results. It is also necessary to see if the coefficient to be applied is the same for juices and syrups, for massecuites where the syrups are not returned as for products more highly charged with salts, as molasses.

There is one peculiarity of certain products which affects the exactitude of the soap test. At a given moment an abundant froth may be noticed which would lead one to suppose the operation was complete, but a few moments later the froth disappears and several tenths of a c.c. more soap solution are required to make the froth persist. It is the latter limit which must be taken.—L. J. de W.

Sugar Canes, Mode of Sampling and Method of Analysis of. Report on the Agric. Work in the Botan. Gardens, Brit. Guiana, for the Years 1893-4-5, 1897, 9—10.

See under XVI., *page* 687.

Hops, Estimation of Resins in. L. Briant and C. S. Meacham. J. Fed. Inst. Brewing, 1897, **3**, 233—236.

To determine the soft resins, about 4 grms. of carefully sampled hops are introduced into a thimble of bibulous

paper or glass, and are placed in an 80 grm. Soxhlet apparatus, and extracted for 24 hours with 120 c.c. of petroleum ether having a boiling point of 122° F. When the extraction is complete, the extract is filtered whilst hot into a tared wide-mouthed flask, the filtrate gently evaporated, and the flask and its contents dried in a water-oven until the weight is constant. The residue left in the extractor that contains the hard resins is now extracted for 12 hours with 120 c.c. of ordinary ether, and the extract passed through the same filter as in the previous operation, the subsequent drying and evaporation being carried out in a similar manner. If necessary, the extracted hops may be dried and weighed. The results are expressed on dry hops; but, for commercial purposes, the moisture should be stated as well as the amount of foreign matters. It has been observed that during the evaporation of the extract containing the soft resins, these change to some extent into hard ones, since their solubility in petroleum ether is considerably reduced.—J. L. B.

Carbohydrates in Foodstuffs, The Quantitative Determination of. W. E. Stone. J. Amer. Chem. Soc. 1897, **19**, 183—197; 347—349.

In various articles published by the author during the past 10 years, attention has been called to the character of the composition of the so-called "nitrogen-free extract" of food materials. It is generally recognised that the portion of food material comprised under the above head, consists of a considerable number of definite chemical compounds, mostly of the nature of carbohydrates, but of such evident variation as regards their food value or digestibility, as well as their chemical and physical properties, that it is inconsistent to regard them of homogeneous character or value in food estimation.

The author has devised the following method for the detailed determination of the carbohydrates in vegetable materials. In all the operations linen filters should be used in order to facilitate the subsequent removal of the sample. With very fine starchy materials it is sometimes advisable to fold a paper filter within the linen one.

1. From 50 to 100 grms. of the sample are boiled with 500 c.c. of strong alcohol under a reflux condenser for two hours, or it may be extracted in a Soxhlet's apparatus. The sugars are thus dissolved, and are removed by filtering off the alcoholic extract. This treatment also dissolves waxes and oils; these, however, may be separated from the sugars by evaporating the solution nearly to dryness, and then taking up with water. If the solution contain only cane sugar it may be brought to a given volume and polarised. If several kinds of sugar be present (say, cane and invert sugar), the alcohol is evaporated, the residue dissolved in a known quantity of water, and the sugars estimated by titration with Fehling's solution before and after inversion, according to the ordinary method of treatment.

2. The residue from the alcoholic extraction may contain carbohydrates soluble in water, as for instance, soluble starch and dextrin, which are specially to be considered in materials which have been subjected to the action of heat. 500 c.c. of water are added to this residue and the whole allowed to stand from 18 to 24 hours, with frequent agitations. The solution is filtered, and in the case of ordinary materials, where only dextrin-like substances are to be expected, evaporated to a small volume, inverted with dilute acid, and titrated with Fehling's solution. If, however, the solution contain soluble starch, which may be recognised by the iodine test, a further distinction between this starch and dextrin is necessary. The aqueous solution is brought to a volume of 200 c.c., and of this an aliquot portion is inverted with dilute acid and the total carbohydrates in solution determined by titration with Fehling's solution. Another aliquot portion may be treated with an excess of barium hydroxide solution, the soluble starch being thereby precipitated. In the filtrate from this precipitate, the dextrin may be determined by inversion and titration as before; the difference between this value and the total carbohydrates being considered as soluble starch. The use of barium hydroxide for the direct estimation of soluble starch according to Von Asboth's method, is not permissible in this place, because there are other substances in solution, such as the albuminoids, which combine with the alkali.

3. The residue from the aqueous extract of the material is now brought to an air-dried condition, and weighed, in order to establish its quantitative relation to the sample as originally weighed out. 2 grms. of this material, which now contains the starch and other insoluble carbohydrates, are weighed and boiled for 30 minutes with 100 c.c. of water, in order to convert the starch into a soluble form. When the solution has cooled to 60°, 10 c.c. of malt extract (10 grms. of malt to 50 c.c. of water) are added, and the conversion allowed to proceed at 65 for two to three hours, after which it is filtered and the filter washed with hot water. The residue on the filter should give no iodine reaction. The filtrate is evaporated to 100 c.c., 10 c.c. of strong hydrochloric acid added, and inverted for one hour in a boiling water-bath. The solution is neutralised, and titrated with Fehling's solution, a correction being made for the amount of sugar contained in the added malt extract.

4. The residue from the last process contains the gums, pentosans, hemicelluloses, and cellulose. It is carefully removed from the filter, 100 c.c. of water and 2 c.c. of strong hydrochloric acid added, and boiled under a reflux condenser or in a water-bath for one hour. This treatment converts the gum and pentosans into reducing sugars (xylose). The solution is filtered, neutralised, made up to 200 c.c. and titrated with Fehling's solution.

5. The residue is treated with a 1·25 per cent. solution of sodium hydroxide, filtered, dried, weighed, ignited, and weighed again in order to obtain what is commonly known as crude fibre.

Following this scheme of analysis, the author has tabulated the results obtained with some representative foodstuffs.

In the second paper, attention is drawn to some uncorrected errors in the determination of normal starch. The corrected table is given below:—

—	Cane Sugar.	Invert Sugar.	Dextrin.	Soluble Starch.	Normal Starch.	Pentosans.	Crude Fibre.	Total Carbohydrates.	Nitrogen-free Extract.
Wheat I.	0·52	0·08	0·27	0·00	57·62	4·54	2·68	65·71	77·07
Wheat II.	0·72	0·00	0·41	0·00	56·27	4·37	2·51	64·28	77·66
Flour I.	0·18	0·00	0·90	0·00	67·99	0·00	0·25	69·32	83·54
Flour II.	0·20	0·00	1·06	0·00	67·76	0·00	0·25	69·27	84·54
Maize	0·27	0·00	0·32	0·00	65·45	5·14	1·99	73·17	78·02
Bread (Wheat I.)	0·11	0·10	0·68	1·37	53·54	4·16	2·70	62·59	77·20
Bread (Wheat II.)	0·05	0·32	0·23	2·36	53·62	4·34	2·42	63·34	77·33
Bread (Flour I.)	0·06	0·37	0·27	1·99	64·81	0·00	0·34	67·84	82·94
Bread (Flour II.)	0·15	0·38	0·91	1·74	64·12	0·00	0·17	67·47	85·17
Corn bread (Maize)	0·16	0·19	0·00	2·89	61·74	3·54	2·22	70·75	77·81

The discrepancy existing between the total carbohydrates and nitrogen-free extract may arise from one of two sources of error in the determination of carbohydrates, or the existence of a substance free from nitrogen and which is of a character not usually ascribed to carbohydrates and resistant to the ordinary reactions for such. While the first alternative is not excluded, the author is inclined to the latter conclusion.—J. L. B.

Wheat, The Insoluble Carbohydrates of. H. C. Sherman. J. Amer. Chem. Soc. 1897, **19**, 291.

See under XVI., page 690.

Cacao-Butter, Iodine Number of. D. Holde. Zeits. Anal. Chem. **36**, 381.

HOLDE pointed out (this Journal, 1897, 363) that the upper limit of the iodine number for cacao-butter given by de Negri and Fabris as 51, was incorrectly transcribed from von Hübl's original work, where it is given as 34.

The error is now admitted by Fabris, the number having been copied unnoticed from the 1896 edition of Benedikt's "*Analyse der Fette und Wachsarten.*" The new edition of this work, in which it is given correctly, was not available when de Negri and Fabris published their monograph "Gli Olii."—L. J. de W.

Acid Potassium Tartrate (Cream of Tartar) in Wine, Determination of. H. Jay. Bull. Soc. Chim. 1897, **17**, 626—629.

AN examination of the methods employed for the determination of acid potassium tartrate (cream of tartar) in wine. The following table gives the weights of acid tartrate found by the methods under investigation :—

Solation employed.	Precipitation by Ether-Alcohol.	Crystallisation after Evaporation to $\frac{1}{10}$.
	Grms.	Grms.
1. Alcohol of 9° + 1·35 grms. of tartar.	1·13	1·24
2. Alcohol of 9° + 2·72 grms. of tartar.	2·53	2·60
3. Wine	2·10	2·55
4. Same wine + 1 grm. of tartar..	3·12	3·60
5. Perry	Trace	0·30
6. Same perry + 3 grms. of tartar	2·82	3·24
7. Same perry + 3 grms. of tartar + potash to neutralise 1·375 grms. of tartar.	2·82	3·12
8. Synthetic wine containing 3 grms.	2·86	3·36
9. Synthetic wine containing 1 grm.	0·83	1·21

The higher results given by the crystallisation method are not entirely due to the smaller loss in that method ; the correction deduced from experiments 1 and 2 does not explain the results of experiments 4, 6, 8, and 9. The excess found by this method is due to the crystallisation of another acid salt—the malate—along with the tartrate. It is the malic acid which is neutralised in experiment 7 The author concludes that the results of the precipitation method are more exact, and, with the corrections, more constantly near the truth than those of the crystallisation process. The evaporation in this last appears to destroy the equilibrium existing between acids and bases.—A. C. W.

Ether, Method for the Determination of Aldehyde in. Maurice François. J. Pharm. Chim. 1897, **5**, 521—525.

AN application of the method of Mohler for the determination of aldehyde in alcohol (this Journal, 1897, 265). The reagent contains 220 c.c. of saturated sulphur dioxide solution, 30 c.c. of 0·1 per cent. magenta solution, and 3 c.c. of sulphuric acid of 66°. Solutions of aldehyde in alcohol containing 1 in 1,000 and 1 in 10,000 are employed for comparison.

5 c.c. of each solution and of the ether to be tested, are mixed with 5 c.c. of alcohol in test tubes, 4 c.e. of the reagent are added to each of the three tubes ; these are shaken, corked, and the colour observed after 15 minutes. This preliminary trial shows which standard solution must be employed in the colorimeter. Ether free from aldehyde is obtained by treatment of 1 kilo. of ordinary ether with 200 c.c. of saturated permanganate solution and 20 grms. of caustic soda. After standing 24 hours, with frequent shaking, the treatment is repeated, the ether filtered, and dried over a mixture of 50 grms. of quicklime and 50 grms. of calcium chloride ; it is then filtered and distilled.

—A. C. W.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Strontium Sulphide, Phosphorescence of. J. R. Mourelo. Comptes Rend. **124**, [22], 1237—1238.

PURE strontium sulphide, obtained by reducing the sulphate with carbon, by treating the carbonate at a red heat with hydrogen sulphide, or by heating the carbonate with sulphur, is *not phosphorescent.* If any one of these sulphides, however, be heated for some time in an oxidising flame, so as to form a small amount of sulphate, it becomes phosphorescent.

After prolonged heating in an oxidising atmosphere, so as to convert the greater part of the sulphide into sulphate, the phosphorescence is lost, but is recovered again on reduction by charcoal, if that reduction has left even as much as 0·042 per cent. of sulphate. Strontium sulphide prepared by the author's method, when it is obtained as a hard, granular, semi-polished substance, not at all easily changed in moist air, but still containing a trace of sulphate, exhibits very strong phosphorescence. Both an oxidising substance and a certain structure, then, appear to be influential in determining the phosphorescent power of strontium sulphide.—J. T. D.

Calcium Carbide Residues, Use of, as Antidote to Phylloxera. E. Chuard. Comptes Rend. **124**, 1247—1248.

IN a previous note, the author has shown that among the products of decomposition of calcium carbide by water are small amounts of ammonia, which come off in greater quantity after the whole of the acetylene has been evolved, than before. The residues from acetylene preparation are thus valuable as a fertiliser, and they also serve as an insecticide. Attempts have been made in Spain, and lately by the author at Veyrier (Haute-Savoie), to treat vines with these residues for phylloxera, with considerable success. The toxic properties seem to be due in part to the presence of traces of phosphine in the evolved gas, and experiments made by the author with a specially prepared carbide containing a notable amount of phosphide (not enough to render the evolved gas spontaneously inflammable), have been very successful, the substance having proved a very powerful insecticide, while as a destroyer of phylloxera it seems also, as far as the experiments have yet gone, very hopeful.—J. T. D.

Metallic Salts, New Combinations of, with Pyridine, Piperidine, and Quinoline. R. Varet. Comptes Rend. **124**, [21], 1155.

Pyridine Bromocuprite.—Pure cuprous bromide is dissolved in boiling pyridine to saturation, avoiding all access of air. The solution is filtered through a hot-water funnel, and forms, on cooling, a crop of green crystals. These, when dried rapidly between filter papers, have the composition $Cu_2Br_2(C_5H_5N)_5$. This body is very soluble in pyridine, especially when warmed. When heated, all the pyridine is given off. It is very unstable in the air, turning first green, then brown.

Pyridine Iodozincate.—Zinc iodide combines with pyridine with the evolution of a considerable amount of heat. The compound $ZnI_2(C_5H_5N)_2$ occurs in fine needles.

Pyridine Cyanozincate.—Cyanide of zinc is not appreciably soluble in pyridine. If the two bodies are left in contact in the dark in a cool place, a gelatinous mass is obtained of uncertain composition. This body parts with all its pyridine on warming, at a much lower temperature than that at which the cyanide decomposes.

The author has also prepared combinations of pyridine, of piperidine, and of quinoline with the halogen salts of aluminium and iron ; also with the iodides of barium, strontium, and calcium, and with the bromides and iodides of manganese. The action of piperidine on the chlorides, iodides, and bromides of zinc, cadmium, nickel, and cobalt has also been studied. Quinoline has also been combined with the salts of mercury, of silver, and of copper.—J. O. B.

Moisture by Deliquescent Substances, Further Experiments on the Absorption of. H. Wilson Hake. Proc. Chem. Soc. 1897, [182], 147.

In a preliminary note (Proc. Chem. Soc. 1896, **12**, 33) the author showed that certain deliquescent salts, when exposed to the air, attained a maximum of hydration, and that its maximum corresponded to a definite number of molecules in a large number of cases.

In the preliminary experiments no reference was made to the vapour pressure of water in the air, but in experiments since made, the condition of the atmosphere as regards moisture has been carefully noted, or an artificially saturated atmosphere has been contrived under known conditions of temperature.

Having now experimented with 10 deliquescent chlorides (lithium, magnesium, cadmium, calcium, copper, nickel, cobalt, iron, manganese, and platinum), three nitrates (sodium, magnesium, and manganese), with sulphuric acid and with sodium formate, under various conditions, it was found that (1) they attract quantities of water corresponding in all cases to a definite hydrate, (2) after deliquescing to a maximum, there followed in all cases a decline in weight, and in four instances the salts returned to their original hydration and crystallised out, and that (3) the amount of hydration, though apparently always corresponding to a definite number of molecules of water, is not always the same, but seems to depend, within certain limits, both on the temperature and the relative humidity of the atmosphere and also on the conditions under which the air has access to the salt.

The author suggests that the above experiments demonstrate that the phenomenon of deliquescence is caused by hydration of the deliquescent salt.

"*Mineral Soap.*" W. C. Knight. Eng. and Mining J. 1897, **63**, 600.

"Mineral soap" is the name given to a peculiar kind of clay found in various parts of Wyoming, U.S.A. The clay, when taken from the quarry, has a greenish-yellow colour, which, on exposure to the air, soon changes to a light cream. It forms an emulsion with water, but only a very small percentage dissolves. Thin seams of gypsum and mirabilite (a hydrated sodium sulphate) are found associated with the clay. The following analysis of one of these clays from Rock Creek, Albany Co., Wyoming, is given:—

SiO_2, 59·78; Al_2O_3, 15·10; Fe_2O_3, 2·40; MgO, 4·14; CaO, 0·73; H_2O, 16·26 per cent.—A. S.

Etherification, Study of the Ordinary Process of. L. Prunier. Comptes Rend. **124**, [22], 1239—1242.

The theory of Williamson, based on the successive formation of ethylsulphuric acid and its reaction on alcohol to form ether and regenerate the sulphuric acid originally present, cannot represent the whole truth, for the process does not continue indefinitely, and can go on without the presence of free sulphuric acid.

Possibly the successive reactions—

$$C_2H_5O.SO_2.OH + C_2H_5.OH = H_2O + C_2H_5O.SO_2.OC_2H_5.$$
$$C_2H_5O.SO_2.OC_2H_5 + C_2H_5.OH = (C_2H_5)_2O + C_2H_5O.SO_2.OH.$$

partly explain it, but the existence of sulphonic compounds among the products of the process, already demonstrated by the author, enables a much more complete account to be given. Isethionic acid (as a type of these compounds) may be formed by the splitting up of the ethylsulphuric acid

$$C_2H_5O.SO_2.OH = C_2H_5.OH + SO_3$$

and the immediate action of the trioxide and the alcohol

$$C_2H_5.OH + SO_3 = OH.CH_2.CH_2.SO_2.OH.$$

The ethyl ether of this is then successively formed and decomposed exactly as in the equations given above for ethylsulphuric acid. This view is borne out by the fact that according to Krafft the sulphuric acid of the ordinary etherification process may be substituted by benzenesulphonic acid. It also accounts for the gradual loss of sulphuric acid, which is partly separated and hydrated by the action of the water formed on the ethylsulphuric acid, partly escapes as sulphur dioxide through the dissociation of some of the sulphonic compounds.—J. T. D.

New Books.

The Prospector's Handbook. A Guide for the Prospector and Traveller in search of Metal-bearing or other Valuable Minerals. By J. W. Anderson, M.A., F.R.G.S. Seventh Edition, thoroughly revised and much enlarged. Crosby, Lockwood, and Son, 7, Stationers' Hall Court, Ludgate Hill, London, E.C. Price 3s. 6d. In strong leather wallet with tuck, price 4s. 6d.

This small volume, with rounded corners for convenience of the pocket, contains prefaces to the first and seventh editions, table of contents, and 152 pages of subject-matter. Thereafter follow a glossary of terms used in connection with Prospecting, Mining, Mineralogy, Assaying, &c., and the alphabetical index. The text, which is illustrated with 69 woodcuts, is subdivided as follows:—I. Prospecting. II. Rocks. III. Testing Minerals by the Blow-pipe. IV. Character of Minerals. V. Metals and Metallic Ores: their Characteristics, Testing, Occurrence, &c. VI. Other Useful Minerals and Ores. VII. Composition of Various Rocks. VIII. Testing by the Wet Process. IX. Assay of Gold. X. Treatment of Ores. XI. Surveying. Appendix, with Weights and Measures of England, France, &c. Specific Gravities of Various Rocks and Metallic Ores, &c., &c.

Organic Chemical Manipulation. By J. T. Hewitt, M.A., D.Sc., Ph.D., Professor of Chemistry in the East London Technical College. Whittaker and Co., 2, White Hart Street, Paternoster Square, London, E.C.; 66, Fifth Avenue, New York, U.S.A. 1897. Price 7s. 6d.

Small 8vo volume with preface, table of contents, subject-matter filling 253 pages, illustrated with 63 woodcuts, and terminating in an alphabetical index.

The design of the work will appear in the following brief excerpt of its contents:—

Part I.—i. Purification of Organic Substances. ii. Ultimate Analysis of Organic Compounds. iii. Determination of Equivalent and Molecular Weight. iv. Estimation of Specific Groups of Atoms in Organic Compounds.

Part II.—v. Compounds of the Fatty Series. vi. Aromatic Hydrocarbons and their Derivatives. (i.) Hydrocarbons and their Nitration. (ii.) Reduction of Nitro-Compounds. (iii.) Diazo-Compounds and Hydrazines. (iv.) Sulphonation. (v.) Phenols. (vi.) Aromatic Acids. (vii.) Aromatic Aldehydes and Ketones. (viii.) Quinones. (ix.) Condensations resulting in the Formation of Closed Ring Compounds.

A Course of Practical Chemistry. By M. M. Pattison Muir, M.A., Fellow and Prælector in Chemistry of Gonville and Caius College, Cambridge. Part I. Elementary. Experiments on Chemical Change. Preparations of Various Compounds. Reactions of Acids, Alkalis, and Salts. An Elementary Course of Volumetric Analysis. Qualitative Analysis of Mixtures of Salts containing not more than a single metal in any one group. Longmans, Green, and Co., 39, Paternoster Row, London; also New York and Bombay. 1897. Price 4s. 6d.

This book is intended for use in connection with lectures and demonstrations. It forms Part I. of a work which is to be complete in three parts. It contains subject-matter filling 301 pages, illustrated with 33 woodcuts, and followed by five appendices and an alphabetical index.

The work (Part I.) is divided into three sections:—

Section I. Experiments on Chemical Change. Preparations of Various Compounds. Reactions of Acids, Alkalis, and Salts.

Section II. Volumetric Estimation of Acids, Alkalis, Alkaline Carbonates, and Iodine: the necessary standard solutions being given.

Section III. Qualitative Analysis. There are five Appendices: i. Making Apparatus. ii. Tables. iii. Reagents. iv. Preparation of Standard Solutions. v. Substances suitable for the Various Exercises.

Trade Report.

TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

New Customs Tariff of Canada.

With reference to the note on p. 573 of the June number of the Journal, the following are Schedules A, B, C, of the New Customs Tariff of Canada, therein referred to, which received the Royal assent on June 29 last :—

Schedule A.

Goods subject to Duties.—Ales, Beers, Wines, and Liqueurs.

No.	Article.	Rate of Duty. Dols. Cts.
1	Ale, beer, and porter, when imported in casks or otherwise than in bottle.	Per gal. 0 16
2	Ale, beer, and porter, when imported in bottles (6 quart or 12 pint bottles to be held to contain 1 gallon).	" 0 24
3	Cider, not clarified or refined	" 0 5
4	Cider, clarified or refined	" 0 10
5	Lime juice and fruit juices, fortified with and containing not more than 25 per cent. of proof spirits.	" 0 60
	Do., when containing more than 25 per cent. of proof spirits.	" 2 00
6	Lime juice and other fruit syrups and fruit juices, n.o.p.	20 % *ad val.*
7	Spirituous or alcoholic liquors distilled from any material, or containing or compounded from or with distilled spirits of any kind, and any mixture thereof with water for every gallon thereof of the strength of proof and when of a greater strength than that of proof, at the same rate on the increased quantity that there would be if the liquors were reduced to the strength of proof. When the liquors are of a less strength than that of proof, the duty shall be at a rate herein provided, but computed on a reduced quantity of the liquors in proportion to the lesser degree of strength; provided, however, that no reduction in quantity shall be computed or made on any liquors below the strength of 15 per cent. under proof, but all such liquors shall be computed as of the strength of 15 per cent. under proof, as follows :—	Per gal. 2 40
	(*a.*) Ethyl alcohol, or the substance commonly known as alcohol, hydrated oxide of ethyl or spirits of wine, gin of all kinds, n.e.s., rum, whiskey, and all spirituous or alcoholic liquors, n.o.p.; amyl alcohol or fusel oil, or any substance known as potato spirit or potato oil; methyl alcohol, wood alcohol, wood naphtha, pyroxylic spirit or any substance known as wood spirit or methylated spirits, absinthe, arrack or palm spirit, brandy, including artificial brandy and imitations of brandy, cordials and liqueurs of all kinds, n.e.s.; mescal, pulque, rum, shrub, schiedam, and other schnapps; tafia, angostura and similar alcoholic bitters or beverages.	
	(*b.*) Spirits and strong waters of any kind, mixed with any ingredient or ingredients, as being or known or designated as anodynes, elixirs, essences, extracts, lotions, tinctures, or medicines, or medicinal wines (so called), or ethereal and spirituous fruit essences, n.e.s.	Per gall. 2 40 and 30 % *ad val.*
	(*c.*) Alcoholic perfumes and perfumed spirits, bay rum, cologne and lavender waters, hair, tooth and skin washes, and other toilet preparations containing spirits of any kind, when in bottles or flasks containing not more than 4 oz. each.	50 % *ad val.*
	Do. when in bottles, flasks, or other packages, containing more than 4 oz. each.	Per gall. 2 40 and 40 % *ad val.*
	Spirituous or alcoholic liquors—*cont.*	
	(*d.*) Nitrous ether, sweets spirits of nitre and aromatic spirits of ammonia.	Per gall. 2 40 and 30 % *ad val.*
	(*f.*) Medicinal or medicated wines containing not more than 40 per cent. of proof spirits.	Per gall. 1 50
	Animals, and Agricultural, Animal and Dairy Products.	
18	Lard, lard compound, and similar substances, cottolene, and animal stearine of all kinds, n.e.s.	Per lb. 0 2
19	Tallow and stearic acid	20 % *ad val.*
20	Beeswax	10 % *ad val.*
21	Candles, n.e.s.	25 % *ad val.*
22	Paraffin wax candles	30 % *ad val.*
23	Soap, common or laundry	Per lb. 0 1
24	Castile soap, mottled or white	Per lb. 0 2
25	Soap, n.e.s.	35 % *ad val.*
26	Pearline and other soap powders	30 % *ad val.*
27	Glue, liquid, powdered or sheet, and mucilage, gelatine, and isinglass.	25 % *ad val.*
69	Extract of malt (non-alcoholic) for medicinal and baking purposes.	25 % *ad val.*
72	Yeast cakes and baking powder, the weight of the packages to be included in the weight for duty.	Per lb. 0 6
	Books and Paper.	
123	Albumenized and other papers and films chemically prepared for photographers' use.	30 % *ad val.*
	Chemicals and Drugs.	
141	Acid, acetic acid, and pyroligneous, n.e.s., and vinegar, for each gallon of any strength not exceeding the strength of proof. [And in addition, for each degree of strength in excess of the strength of proof, an additional duty of two cents.] The strength of proof shall be held to be equal to 6 per cent. of absolute acid; and in all cases the strength shall be determined in such manner as is established by the Governor in Council.	Per gall. 0 15
142	Acid, acetic acid crude, and pyroligneous crude, of any strength not exceeding 30 per cent.	25 % *ad val.*
143	Acid, muriatic and nitric, and all mixed or other acids, n.e.s.	20 % *ad val.*
144	Acid, sulphuric	25 % *ad val.*
145	Acid phosphate, n.o.p.	25 % *ad val.*
146	Sulphuric ether, chloroform, and solutions of peroxides of hydrogen.	25 % *ad val.*
147	All medicinal, chemical, and pharmaceutical preparations, when compounded of more than one substance, including patent and proprietary preparations, tinctures, pills, powders, troches, lozenges, syrups, cordials, bitters, anodynes, tonics, plasters, liniments, salves, ointments, pastes, drops, waters, essences, and oils, n.o.p.; provided that drugs, pill-mass, and preparations, not including pills or medicinal plasters, recognised by the British or the United States Pharmacopœia or the French Codex as officinal, shall not be held to be covered by this item; all liquids containing alcohol.	50 % *ad val.*
	And all others, liquid or not	25 % *ad val.*
149	Perfumery, including toilet preparations (non-alcoholic)—viz., hair oils, tooth and other powders and washes, pomatums, pastes, and all other perfumed preparations, n.o.p., used for the hair, mouth, or skin.	30 % *ad val.*
151	Paraffin wax	30 % *ad val.*
152	Antiseptic surgical dressings, such as absorbent cotton, cotton wool, lint, lambswool, tow, jute, gauzes, and oakum, prepared for use as surgical dressings, plain or medicated.	20 % *ad val.*
154	Cod liver oil	20 % *ad val.*
	Colours, Paints, Oils, Varnishes &c.	
158	Dry white and red lead, orange mineral, and zinc white.	5 % *ad val.*
159	Ochres, ochrey earths, raw siennas and colours, dry, n.e.s.	20 % *ad val.*

No.	Article.	Rate of Duty.	Dols.	Cts.
160	Oxides, umbers, burnt siennas, and fireproofs, n.e.s.; laundry bluing of all kinds, rough stuff, and dry and liquid fillers, anti-corrosive and anti-fouling paints commonly used for ships' hulls, and ground and liquid paints, n.e.s.	25 °/o *ad val.*		
161	Paints and colours, ground in spirits, and all spirit varnishes and laquers.	Per gall.	1	12
162	Paris green, dry	10 °/o *ad val.*		
163	Ink for writing	20 °/o *ad val.*		
164	Blacking, shoe, and shoe-makers' ink; shoe, harness, and leather dressing, harness soap, and knife or other polish or composition, n.o.p.	25 °/o *ad val.*		
165	Putty, of all kinds	20 °/o *ad val.*		
166	Turpentine, spirits of	5 °/o *ad val.*		
167	British gum, dextrine, sizing cream and enamel sizing.	10 °/o *ad val.*		
168	Varnishes, lacquers, japans, japan driers, liquid driers, and oil finish, n.e.s.	Per gall.	0	20 and 20 °/o *ad val.*
169	Linseed or flax seed oil, raw or boiled, lard oil, neatsfoot oil, and sesame seed oil.	25 °/o *ad val.*		
170	Illuminating oils composed wholly or in part of the products of petroleum, coal, shale, or lignite, costing more than 30 cents per gallon.	25 °/o *ad val.*		
171	Lubricating oils, composed wholly or in part of petroleum, costing less than 25 cents per gallon.	Per gall.	0	5
172	Crude petroleum, fuel and gas oils (other than naphtha, benzine, or gasoline), when imported by manufacturers (other than oil refiners), for use in their own factories for fuel purposes or for the manufacture of gas.	„	0	2½
173	Oils, coal and kerosene distilled, purified or refined, naphtha and petroleum, and products of petroleum, n.e.s.	„	0	5
174	Barrels, containing petroleum or its products, or any mixture of which petroleum forms a part, when such contents are chargeable with a specific duty.	Each	0	20
175	Lubricating oils, n.e.s., and axle grease	25 °/o *ad val.*		
176	Olive oil, n.e.s.	20 °/o *ad val.*		
177	Essential oils	10 °/o *ad val.*		
178	Vaseline, and all similar preparations of petroleum for toilet, medicinal, or other purposes.	35 °/o *ad val.*		
	Earthenware, Cements, Slate, and Stoneware.			
187	Cement, Portland and hydraulic or water lime, in bags, barrels, or casks, the weight of the package to be included in the weight for duty.	100 lb.	0	12½
188	Plaster of Paris, or gypsum, ground, not calcined.	15 °/o *ad val.*		
189	Plaster of Paris, or gypsum, calcined or manufactured, the weight of the package to be included in the weight for duty.	100 lb.	0	12½
	Glass and Glassware.			
201	Common and colourless window glass, and plain coloured, opaque, stained, or tinted or muffled glass, in sheets.	20 °/o *ad val.*		
202	Ornamental, figured, and enamelled coloured glass, vitrified or painted, chipped, figured, enamelled, and obscured white glass, stained glass windows and memorial, or ornamental window glass, n.o.p., and rough rolled plate glass.	30 °/o *ad val.*		
203	Plate glass, not bevelled, in sheets or panes, not exceeding twenty-five square feet each, n.o.p.	25 °/o *ad val.*		
204	Plate glass, not bevelled, in sheets or panes, n.e.s.	35 °/o *ad val.*		
205	Plate glass, bevelled, in sheets or panes, n.o.p.	35 °/o *ad val.*		
206	Silvered glass, bevelled or not and framed or not.	35 °/o *ad val.*		
207	German looking-glass plate, thin plate, unsilvered or for silvering.	20 °/o *ad val.*		
208	Glass demijohns or carboys, empty or filled, bottles, decanters, phials, glass jars and glass balls, lamp chimneys, glass shades or globes, cut, pressed, or moulded crystal or glass tableware, decorated or not, and blown glass tableware.	30 °/o *ad val.*		
209	Bent plate or other sheet glass, and all other glass, and manufactures of glass, n.o.p.	20 °/o *ad val.*		
	Leather, Rubber, and Manufactures of.			
212	Dongola, cordovan, calf, sheep, lamb, kid or goat, kangaroo, alligator, or other upper leather, and all leather, dressed, waxed, glazed, or further finished than tanned, n.e.s.; harness leather, and chamois skin.		0	17½
213	Skins for morocco leather, tanned, but not further manufactured; sole leather and belting leather of all kinds; tanners' scrap leather; and leather and skins, n.o.p.	15 °/o *ad val.*		
214	Glove leathers, tanned or dressed, coloured or uncoloured, when imported by glove manufacturers for use in their own factories in the manufacture of gloves.	10 °/o *ad val.*		
215	Japanned, patent, or enamelled leather, and morocco leather.	25 °/o *ad val.*		
216	Leather-board, leatheroid, and manufactures thereof, n.o.p.	25 °/o *ad val.*		
220	Manufactures of raw hide, and all manufactures of leather, n.o.p.	25 °/o *ad val.*		
221	India rubber boots and shoes; and all manufactures of india rubber and gutta-percha, n.o.p.	25 °/o *ad val.*		
222	India-rubber clothing, and clothing made waterproof with india-rubber, rubber or gutta-percha hose, and cotton or linen hose lined with rubber, rubber mats or matting, and rubber packing.	35 °/o *ad val.*		
	Metals and Manufactures of.			
225	Ferro-silicon, ferro-manganese, and spiegeleisen.	5 °/o *ad val.*		
235	Chrome steel	15 °/o *ad val.*		
265	Brass wire, plain	10 °/o *ad val.*		
266	Copper wire, plain, tinned or plated	15 °/o *ad val.*		
267	Wire cloth, or woven wire or brass, or copper.	25 °/o *ad val.*		
268	Wire of all metals and kinds, n.o.p.	20 °/o *ad val.*		
272	Lead, old, scrap, pig, and block	15 °/o *ad val.*		
273	Lead, in bars, and in sheets	25 °/o *ad val.*		
274	Lead pipe, lead shot, and lead bullets	35 °/o *ad val.*		
275	Lead, manufacture of, n.o.p.	30 °/o *ad val.*		
276	Brass and copper nails, tacks, rivets and burrs or washers; bells and gongs, n.e.s., and all manufactures of brass or copper, n.o.p.	30 °/o *ad val.*		
277	Zinc, manufactures of, n.o.p.	25 °/o *ad val.*		
278	Nickel anodes	10 °/o *ad val.*		
286	Celluloid, moulded into sizes for handles of knives and forks, not bored nor otherwise manufactured; also, moulded celluloid balls and cylinders, coated with tin foil or not, but not finished or further manufactured, and celluloid lampshade blanks.	10 °/o *ad val.*		
292	Britannia metal, nickel silver, Nevada and German silver, manufactures of, not plated, and manufactures of aluminium, n.o.p.	25 °/o *ad val.*		
293	Sterling or other silver ware, nickel-plated ware, gilt or electro-plated ware, wholly or in part, of all kinds, n.e.s.	30 °/o *ad val.*		
294	Telephone and telegraph instruments, electric and galvanic batteries, electric motors, dynamos, generators, sockets, insulators of all kinds; and electric apparatus, n.e.s.	25 °/o *ad val.*		
295	Electric light carbons and carbon points, of all kinds, n.e.s.	35 °/o *ad val.*		
296	Carbons over six inches in circumference.	15 °/o *ad val.*		
299	Rabbit metal, type metal, phosphor tin, and phosphor bronze in blocks, bars, plates, sheets, and wire.	10 °/o *ad val.*		

(To be continued.)

UNITED STATES.

Customs Regulations.

Sulphite of soda and chloride of magnesium are liable to duty at 25 per cent. *ad valorem*, under paragraph 60 of the Tariff Act of August 28, 1894.

American Drawback on Alcoholic Preparations.

Chem. and Druggist, Aug 9, 1897, 225.

The American Government have made it known that they will in future allow a drawback on certain exported spirituous preparations, such as essences of camphor and peppermint, equal to the amount of duty paid on the alcohol used in the preparations, less 1 per cent.

EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

New Margarine Law in Germany.

Board of Trade. J., Aug. 1897, 204.

A despatch, dated 8th July last, has been received at the Foreign Office from Sir Frank C Lascelles, Her Majesty's Ambassador at Berlin, enclosing memorandum of the new German Margarine Law of 1897, which states that, with reference to those clauses which come into force on October 1st, 1897, the Bill that recently passed the Reichstag and became law, left to the Bundesrath the task of settling the form or method to be employed to render margarine distinguishable from butter when used for trade purposes.

On July 2nd the "Diet" concluded its labours on this subject, and decreed that in order to facilitate its recognition in trade, "sesamol" ("Sesamum orientale") is to be added to it in the preparation of all fats and oils. To 100 parts of the used fats or oils the addition of margarine must be 10 parts of "sesamol," and to margarine cheeses at least 5 parts. For the casks or coverings in which margarine, its cheeses, &c., are to be sold, models are supplied giving the dimensions and position of the lettering, red bands, &c. The name or firm of makers, as also the maker's mark, are to be placed close to the lettering without going over the red bands. The lettering and maker's mark are to be burned or painted on. If done in the latter way they are to be painted in black letters on a white or light yellow ground. The lettering and marks are to appear twice at opposite ends or sides of the barrels or coverings.

Rubber in Tonkin.

A correspondent writes to the *Bulletin de la Société de Géographie Commerciale*, Paris, Vol. XIX., No. 5, that he has discovered, after investigation, that a tree which grows abundantly in Tonkin is the same as the *Ficus elastica*, which is cultivated in Brazil, Bolivia, the Guianas, &c. There is a good opening, he thinks, for a new industry in Tonkin, especially as labour is cheap and easily obtained.

GENERAL TRADE NOTES.

Tunisian Trade in Chemical Products and in Alcohols.

Board of Trade J., Aug. 1897, 221.

The Belgian Consul-General at Tunis has reported specially to his Government on the importation into Tunis of those products which concern chiefly pharmacists, druggists, and wine producers.

These products, the Consul-General states, are not subject to much fluctuation in the market, as are articles of fashion, or those which can be improved by new inventions. It is sufficient to create a connection and revise it occasionally to maintain confidence.

The value of the importation into Tunis of the above-named products amounted in 1896 to over 1,200,000 francs (48,000*l*.). Of this amount alcohol alone stands for 20,000*l*.; it is principally used by wine-growers for fortifying wines.

Mineral Output in U.S.A.

Chem. and Druggist, Aug. 7, 1897, 225.

The annual report for 1896 of Dr. David T. Day, Chief of the Division of Mineral Resources in the United States, gives the following particulars concerning the output of minerals in that country:—"The bromine-industry is chiefly in the hands of a syndicate. In 1896 it showed a slight increase to 546,580 lbs., valued at the works at 114,501 dols., compared with 517,421*l*., worth 134,343 dols., in 1895. The output for 1896 includes the bromine in potassium bromide made directly. The sulphur output rose from 1,800 tons in 1895 to 5,260 tons in 1896. The increase was due to the success attending the working of the Louisiana deposits by Frasch's process. The increased price, due to the Sicilian-sulphur combination, excited search for other deposits, with the result of attracting some attention to the deposits in Texas. The same fact is the cause that, in spite of low prices, search for a more extended supply of pyrites in the United States is quite active in consequence of the higher price of sulphur. The borax output went up to the normal level of 13,508,000 lbs., worth 675,400 dols., all from California and Nevada. In infusorial earth there was a slight decrease in product to a value of 16,792 dols., against 20,514 dols. in 1895. The mineral-waters industry showed an increase in quantity from 21,463,543 galls. in 1895 to 21,668,345 galls. in 1896, but there was a decline in the total value from 4,254,237 dols. in 1895 to 4,005,688 dols. in 1896. There were produced 60,000 flasks (4,590,000 lbs.) of quicksilver in 1882, but 10 years later production had fallen to about one-third of this. Since then the product has gained slowly to 30,765 flasks in 1896. The industry, though still practically confined to California, is slowly extending into Oregon. No development has been made of the deposit recently found in Texas."

The Production of Soda.

Ch. of Com. J., July 1897, 152.

Handels-Museum reports that the production of ammonia soda in Germany again increased last year. Two new factories were built. The total production of soda in the world is now estimated at $1\frac{1}{4}$ million tons. Abroad also this branch of industry is constantly on the increase. North America, hitherto the largest buyer of English Leblanc soda, has increased its production of ammonia soda from 60,000 to 80,000 tons. Russia consumes a total of about two millions of pouds of calcined soda. Of these the Russian factories produce about 1,800,000 pouds, whilst about 200,000 pouds are imported.

Platinum in Russia.

Ch. of Com. J., July 1897, 150.

The Russian Ministry of Finances has recently issued a report on the production of platinum in Russia, according to which that country stands first in the world for this production, 40 times the quantity produced by all other countries together being obtained there. In the year 1880 the quantity produced amounted to 2,946 kilogs.; in 1895 it reached 4,413 kilogs. The production had gone on increasing up to the last year, when it diminished on account of the wet weather in summer. This rare metal is found exclusively in the Southern Ural. The manner of its being worked up is unknown in Russia; this is done in Germany, to which country the platinum is exported in a crude state. Whatever Russia requires of worked-up platinum, it has to buy back from Germany. Of late years the price of this article has run very high; at present it is 900 marks (about stg. 45*l*.) for 1 kilog. of crude platinum in Russia. On mining for the platinum the still rarer metal iridium is also found, but only in very small quantities. Last year the total quantity of iridium obtained did not amount to more than 4·1 kilog., and this was only slightly exceeded in 1894.

BOARD OF TRADE RETURNS.

Summary of Imports.

Articles.	Month ending 31st July	
	1896.	1897.
	£	£
Metals	1,820,622	1,983,600
Chemicals and dyestuffs	534,570	430,792
Oils	718,230	759,037
Raw materials for non-textile industries.	5,106,149	5,985,226
Total value of all imports	34,378,158	36,123,523

Summary of Exports.

Articles.	Month ending 31st July	
	1896.	1897.
	£	£
Metals (other than machinery)	2,964,036	3,023,282
Chemicals and medicines...........	622,017	629,405
Miscellaneous articles..............	2,835,046	2,887,761
Total value of all exports.....	21,359,139	21,501,452

Imports of Metals for Month ending 31st July.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
Copper :—			£	£
Ore .... Tons	15,203	12,209	84,718	41,728
Regulus .... „	8,198	7,669	205,051	213,808
Unwrought..... „	4,680	7,939	228,559	390,557
Iron :—				
Ore............. „	599,431	576,337	416,201	432,879
Bolt, bar, &c. ... „	6,144	6,878	49,397	56,175
Steel, unwrought.. „	1,504	2,589	15,501	14,896
Lead, pig and sheet „	15,003	13,346	165,086	155,702
Pyrites .... „	47,820	51,829	78,640	83,799
Quicksilver .... Lb.	53,890	59,125	3,176	5,551
Silver ore....... Value £	..	..	102,737	130,202
Tin .... Cwt.	49,044	26,406	147,581	80,185
Zinc .... Tons	5,618	5,854	95,834	93,455
Other articles ...Value £	..	..	228,141	284,663
Total value of metals	..	..	1,820,622	1,983,600

Imports of Chemicals and Dyestuffs for Month ending 31st July.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Alkali............ Cwt.	8,477	18,658	6,329	8,470
Bark (tanners', &c.) „	35,957	52,455	13,218	21,837
Brimstone .... „	51,820	35,880	11,891	7,897
Chemicals...... Value £	..	..	129,836	111,237
Cochineal .... Cwt.	451	482	2,860	2,991
Cutch and gambier Tons	1,643	2,800	36,879	42,807
Dyes :—				
Alizarin ....Value£	..	..	20,100	14,653
Anilin and other „	..	..	44,383	37,491
Indigo .... Cwt.	3,737	1,684	61,976	25,013
Nitrate of potash . „	40,872	25,950	31,807	19,149
Valonia .... Tons	1,492	3,580	15,018	36,006
Other articles... Value £	..	..	160,273	103,151
Total value of chemicals	..	..	534,570	430,792

Imports of Oils for Month ending 31st July.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Cocoa-nut......... Cwt.	9,239	12,892	10,041	13,211
Olive.............. Tuns	1,005	1,245	34,978	42,366
Palm .... Cwt.	101,862	95,477	104,842	95,446
Petroleum .... Gall.	13,558,795	21,096,814	279,008	343,889
Seed .... Tons	2,217	2,998	42,949	51,647
Train, &c. .... Tuns	3,956	2,255	66,778	38,083
Turpentine .... Cwt.	93,141	101,102	88,739	99,632
Other articles .. Value £	..	..	90,895	75,263
Total value of oils...	..	..	718,230	759,037

Imports of Raw Material for Non-Textile Industries for Month ending 31st July.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Bark, Peruvian .. Cwt.	2,406	1,710	5,546	3,322
Bristles.......... Lb.	426,894	362,732	57,515	43,389
Caoutchouc ...... Cwt.	32,642	23,280	340,820	273,561
Gum :—				
Arabic......... „	5,828	6,261	13,947	10,872
Lac, &c. .... „	20,293	14,918	99,842	58,273
Gutta-percha .... „	4,343	2,411	36,271	22,512
Hides, raw :—				
Dry............ „	28,623	46,071	72,593	116,846
Wet .... „	48,180	66,159	108,379	146,569
Ivory............ „	1,178	1,308	46,831	49,252
Manure :—				
Guano .... Tons	324	421	1,322	2,339
Bones .... „	5,480	4,069	23,220	13,122
Nitrate of soda... „	9,051	3,249	71,568	24,083
Phosphate of lime „	22,947	21,414	34,190	30,545
Paraffin.......... Cwt.	52,081	47,105	50,308	39,470
Linen rags....... Tons	1,259	1,879	10,829	18,316
Esparto.......... „	17,346	15,121	73,192	60,707
Pulp of wood .... „	35,379	28,510	175,437	149,390
Rosin............ Cwt.	186,358	140,504	45,299	35,551
Tallow and stearin „	194,584	264,983	199,557	249,060
Tar ....Barrels	16,895	13,867	10,639	8,709
Wood :—				
Hewn .... Loads	293,245	364,431	595,087	774,207
Sawn .... „	951,420	1,191,023	2,099,371	2,851,175
Staves......... „	14,514	14,615	71,648	45,817
Mahogany ..... Tons	4,586	7,297	36,717	62,786
Other articles....Value £	..	..	826,012	895,353
Total value	..	..	5,106,149	5,985,226

Besides the above, drugs to the value of 88,509*l*. were imported, as against 82,566*l*. in July 1896.

Exports of Metals (other than Machinery) for Month ending 31st July.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Brass............ Cwt.	9,513	8,732	37,812	38,522
Copper :—				
Unwrought.... „	39,139	33,487	99,610	87,063
Wrought ...... „	18,128	24,533	56,513	73,565
Mixed metal... „	7,285	18,541	18,444	43,898
Hardware...... Value £	..	..	172,555	173,918
Implements ..... „	..	..	128,330	124,268
Iron and steel ... Tons	326,991	331,553	2,213,696	2,136,401
Lead .... „	3,994	4,735	47,682	60,791
Plated wares ... Value £	..	..	30,579	34,266
Telegraph wires . „	..	..	53,139	136,547
Tin .... Cwt.	7,485	7,725	24,607	25,561
Zinc .... „	10,430	12,835	7,738	10,446
Other articles .. Value £	..	..	73,331	78,036
Total value	..	..	2,964,036	3,023,282

Exports of Drugs and Chemicals for Month ending 31st July.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Alkali........... Cwt.	351,753	371,991	95,712	104,509
Bleaching materials „	86,644	81,468	28,885	27,013
Chemical manures Tons	37,228	41,517	140,788	158,874
Medicines....... Value £		..	87,002	91,177
Other articles... „	..	..	269,630	247,832
Total value	..	..	622,017	629,405

Exports of Miscellaneous Articles for Month ending 31st July.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Gunpowder...... Lb.	769,500	953,700	16,420	20,061
Military stores.. Value £	..	..	215,094	136,461
Candles.......... Lb.	1,611,900	2,022,600	24,217	27,177
Caoutchouc..... Value £	..	..	113,652	99,153
Cement.......... Tons	32,600	38,039	53,911	62,387
Products of coal Value £	..	..	111,962	121,458
Earthenware ... „	..	..	159,617	150,761
Stoneware...... „	..	..	17,400	13,392
Glass:—				
Plate........... Sq. Ft.	170,324	140,196	10,837	7,360
Flint........... Cwt.	7,740	9,285	17,833	20,892
Bottles......... „	62,856	69,676	30,135	33,067
Other kinds.... „	21,599	18,322	17,698	14,433
Leather:—				
Unwrought .... „	11,040	13,350	99,519	123,292
Wrought ..... Value £	..	..	33,280	35,043
Seed oil.......... Tons	4,285	3,688	81,088	64,017
Floorcloth ..... Sq. Yds.	1,931,800	2,127,300	82,625	90,110
Painters' materials Val. £	..	..	153,893	148,985
Paper .... Cwt.	87,183	87,712	130,349	129,548
Rags............ Tons	4,602	5,533	23,850	28,238
Soap............ Cwt.	59,739	57,338	63,422	56,239
Total value	..	..	2,835,046	2,887,761

Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—PLANT, APPARATUS, and MACHINERY.

Applications.

16,989. J. E. Slack and D. Rushworth. Improvements in the manufacture of tubes in apparatus for heating and cooling fluids. July 19.

17,099. O. H. Pieper and A. F. Pieper. Rheostats. Complete Specification. July 20.

17,356. W. G. Heys.—From J. Klein, of the Maschinen und Armaturfabrik vormals Klein, Schanzlin, and Becker, Germany. Improvements in apparatus for cooling liquids. Complete Specification. July 23.

17,475. Forbes, Abbott, and Lennard, Ltd., and L. Gaster. Improvements in and apparatus for use in subjecting air or other gases or liquids or solids to the action of air, gases, or liquids; applicable also for mixing, cooling, heating, and other analogous operations. July 24.

17,712. J. Lehmann and A. Serigiers. Apparatus for compressing gas. July 28.

17,714. J. Buchanan, jun. Improvements in appliances for discharging coke and like ovens. July 28.

18,189. O. Guttmann. *See* Class VII.

18,311. L. A. Chevalet and W. Boby. Improvements in apparatus for heating and purifying water. Aug. 6.

18,454. D. W. Forbes and W. Kemp. Conical vaporiser. Aug. 9.

18,460. M. J. Kelly. An improved composition for removing and preventing incrustation and corrosion in steam and water boilers and cisterns. Aug. 9.

18,578. H. Hirzel. An improved absorption apparatus for absorbing gases in liquids. Complete Specification. Aug. 10.

18,655. J. C. Montgomerie. Improvements in filter presses and in cloths therefor. Aug. 11.

18,816. L. Fremy. An improved apparatus for sterilising liquids and for use as a receptacle for containing the same. Date applied for March 18, 1897, being date of application in France. Complete Specification. Aug. 13.

18,819. W. R. Renshaw. Improvements in apparatus suitable for cooling liquids and condensing. Aug. 13.

Complete Specifications Accepted.*

1896.

22,777. T. Levoz. Converters. Aug. 18.

22,811. F. N. Cockson and J. Stewart. Means for providing for the collection and removal of saline and other deposits from steam boilers, evaporators, and the like. Aug. 18.

1897.

5823. Maigen's "Filtre-Rapide and Anti-Calcaire" Co., Ltd., and A. A. Ellis. Treatment of water or other liquids with powdered material. Aug. 4.

16,035. A. J. Boult.—From A. G. Waterhouse and A. W. Case, United States. Heating fluids and distilling. Complete Specification. Aug. 11.

16,237. P. Gerlach. Cooling apparatus for beer and other liquids. Complete Specification. Aug. 18.

16,398. R. Haddan.—From H. Colberg, A. Serret, and F. Amigo. Improved furnace for the production of carbide of calcium. Complete Specification. Aug. 18.

II.—FUEL, GAS, and LIGHT.

Applications.

17,021. E. Godin. Improvements in acetylene gas generators. Complete Specification. July 19.

17,090. E. Barnard. An improved apparatus for the generation of acetylene gas or other gases. July 20.

17,327. W. B. Hartridge. An improvement in fuel blocks. July 22.

17,448. R. F. Carter. Improved apparatus for the production and storage of acetylene gas. Complete Specification. July 24.

17,482. N. Reggiani and A. Chrisini. Improvements in acetylene gas generators. Complete Specification. July 24.

17,635. H. C. B. Forester and The Patent Agglomerated Fuel Syndicate, Ltd. Improvements in or relating to the manufacture of artificial fuel. July 27.

17,644. H. H. Lake.—From Fellner and Ziegler, Germany. Improvements in apparatus for purifying gas by washing or scrubbing. July 27.

17,749. V. B. Lewes. Improvements in and apparatus for the manufacture of illuminating gas. July 28.

17,793. J. G. A. Kitchen. Improvements in or connected with acetylene generators. July 29.

17,813. P. Dumont. Improvements in acetylene apparatus. July 29.

17,884. H. Muratet and J. Bessac. Improvements in acetylene gas generators. July 29.

17,883. R. Rousselet and A. de Labrousse. Improvements in acetylene gas generators. July 30.

17,927. M. Bernstein. Improvements in petroleum blue flame burners for incandescent lighting and heating purposes. Complete Specification. July 30.

17,938. A. Rieffel. Improvements in apparatus for the production of acetylene gas. Complete Specification. July 30.

17,976. E. Gobbe. Improvements in and apparatus for the manufacture of gas and the obtainment of bye-products therefrom. July 31.

18,005. W. P. Thompson.—From A. Julien, Belgium. Improvements in and relating to apparatus for producing heating or lighting gas by the aid of volatile hydrocarbons. Complete Specification. July 31.

18,056. P. Imbert-Rolland. Improvements in acetylene gas generators. Aug. 3.

18,085. W. Young and A. Bell. *See* Class III.

18,128. F. Goulding. Improvements in apparatus employed in the production of acetylene gas. Aug. 3.

18,218. F. H. Smith. Improvements in the preparation of calcium carbide and the like for use in gas generators. Aug. 5.

18,315. G. Webb, jun., J. W. Kelly, and H. Rountree. Improvements in apparatus for the production of carbide of calcium. Aug. 6.

18,355. J. C. Bull.—From Jossé and Defays, France. Improvements in and relating to the manufacture of acetylene gas. Aug. 6.

18,360. G. Daubenspeck. Improvements in and relating to refractory incandescent mantles or hoods. Aug. 6.

18,481. G. F. Dinsmore. Improvements relating to the transmission of heat for making water-gas, generating steam, melting refractory material, and the like, and to apparatus therefor. Date applied for Jan. 11, 1897, being date of application in United States. Aug. 9.

18,528. J. B. Lavaur. Improvements in acetylene gas generators. Aug. 10.

18,565. E. J. Clubbe, A. W. Southey, and The Electric Motive Power Company, Ltd. Improvements in the generation of oil-gas. Aug. 10.

18,603. T. Mitchell. Improved apparatus for the generation and storage of acetylene and other gases. Aug. 11.

18,639. F. J. Collin. Improvements in coke-ovens. Aug. 11.

18,744. Sir C. S. Forbes, Baronet. Improvements in or connected with acetylene lamps. Aug. 12.

18,745. Sir C. S. Forbes, Baronet. Improvements in generators for acetylene gas. Aug. 12.

Complete Specifications Accepted.

1896.

13,511. A. F. Bowers. Regulators and gas-holders for acetylene gas. July 28.

17,209. G. C. Fowler and M. B. Fowler. Manufacture of night lights. Aug. 4.

17,904. H. H. Lake.—From O. Grenier and J. Grand. Apparatus for generating and utilising acetylene gas for lighting purposes. July 28.

18,915. B. Kosmann. Separation of certain rare earths, and the manufacture therefrom of fabrics for use in incandescent gas-lighting. July 28.

20,074. G. W. Gaskell and R. R. Gibbs. Generation and storage of acetylene. Aug. 4.

20,125. W. Young, S. Glover, and T. Glover. Manufacture of illuminating gas. Aug. 18.

20,225. P. Fritzsche. Obtaining ether from gases containing ethylene and apparatus therefor. Aug. 11.

20,895. D. D. Esson. Combustible compound or fuel. July 28.

21,351. A. Kay. Apparatus for the generation and storage of acetylene gas. Aug. 4.

21,374. J. D. Palmer, H. Palmer, and H. E. Trestrail. Night lights. Aug. 11.

21,695. J. B. Fournier. Apparatus for containing and supplying liquefied gas for lighting and other purposes. Aug. 4.

21,698. A. H. Barker. Generators for acetylene gas. Aug. 4.

21,758. E. C. L. Pillion and C. Bertolus. Apparatus for generating and storing acetylene gas. July 28.

22,359. E. H. Maddock and W. Jones. Acetylene gas generators and gasometers. Aug. 18.

22,526. L. T. FitzGibbon. Production of acetylene gas. Aug. 18.

23,289. C. F. J. B. Becherel. Apparatus for the manufacture of acetylene gas. Aug. 18.

23,290. C. F. J. B. Becherel. Apparatus for the manufacture of acetylene gas. Aug. 18.

23,669. H. E. Oving. Process and apparatus for the production and treatment of acetylene. July 28.

23,670. H. E. Oving. Treatment of acetylene or other gases for lighting, heating, or other purposes. Aug. 4.

24,114. A. J. Smith and A. G. Smith. Automatic acetylene gas generator. Aug. 11.

24,140. P. de Resener and H. L. A. Luchaire. Apparatus for the manufacture of acetylene. Aug. 11.

29,551. H. K. Spence, A. Beveridge, W. G. Beveridge, and S. M. Beveridge. Acetylene gas generators.

1897.

9996. W. Bueddeus. Process for obtaining thorium oxide and for enriching monazite sands. Aug. 18.

13,103. F. H. Smith. Manufacture of calcium carbide and the like, and method of using the same in lamps or generators. Aug. 18.

14,171. H. A. Jones. Gas apparatus. Aug. 18.

14,814. S. Pitt.—From The Compagnie Internationale des Procédés A. Siegle, France. Apparatus for heating by means of liquid hydrocarbons. Aug. 4.

15,509. L. V. Pratis and P. Marengo. Apparatus for the manufacture or production of hydrogen gas. Aug. 4.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Application.

18,085. W. Young and A. Bell. Improvements in the carbonisation or destructive distillation of coal, and in the production of carburetted water gas, and in apparatus therefor. Aug. 3.

IV.—COLOURING MATTERS and DYES.

Applications.

17,018. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of dyestuffs for cotton. July 19.

17,328. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of yellow azo dyestuffs fast to washing and light. July 22.

17,529. H. H. Lake.—From L. Dufour, Italy. *See* Class XIV.

17,729. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of new anthraquinone dyestuffs. July 28.

18,002. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, France. Manufacture of green and blue-green colouring matters from triphenylmethan. July 31.

18,017. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement Gilliard P. Monnet et Cartier, France. Improvements in and relating to the manufacture of dyestuffs. July 31.

18,376. I. Levinstein and Levenstein, Ltd. The manufacture or production of new colouring matters. Aug. 7.

18,494. J. Y. Johnson.—From C. F. Boehringer and Soehne, Germany. Improvements in the manufacture or preparation of amino- and hydrazino-derivatives of purine. Aug. 9.

18,762. A. Ashworth. Improvements in the manufacture of colouring matters suitable for dying unmordanted cotton. Complete Specification. Aug. 13.

Complete Specifications Accepted.

1896.

19,946. R. Blank. Manufacture of compounds of the series of the indoxylic acids and of dyestuffs of the indigo series. Aug. 4.

19,976. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Basic disazo colouring matters. July 28.

21,196. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier. Production of colouring matters. Aug. 4.

21,198. G. B. Ellis.—From La Société Chemique des Usines du Rhône anciennement G. P. Monnet et Cartier. Manufacture of colouring matters. Aug. 11.

22,041. O. Imray.—From The Farbwerke vormals F. Bayer and Co. Ortho- and para-nitrobenzyl aniline and its homologues. Aug. 11.

23,175. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. New violet mono-azo dyestuffs for wool, dyeing a fast brown by oxidation with chrome. Aug. 4.

25,725. I. Levenstein and Levenstein, Ltd. Production of a new colour base and of colouring matters therefrom. Aug. 4.

1897.

12,963. L. Lederer. Production of indifferent (neutral) compounds of alkoxylated phenols. Aug. 18.

15,009. R. Barge. Separating ortho-anhydro-sulphamine-benzoic acid from para-sulphamine-benzoic acid. Aug. 4.

V.—TEXTILES, COTTON, WOOL, SILK, Etc.

Applications.

17,302. J. Wetter.—From Herberlien and Co., Switzerland. Improvements in the treatment of cotton yarn and other vegetable textile material for imparting to it a silk-like gloss. July 22.

17,397. H. A. Lowe. Improvements in the treatment of cotton or other vegetable fibres. July 23.

17,901. W. P. Dreaper and H. K. Tompkins. Improvements in the manufacture of artificial silk from suitable forms of cellulose. July 30.

17,959. G. Wendler and L. E. Wendler. Improvements in the method of and apparatus for treating textile fabrics having a vegetable origin. July 31.

18,222. W. Clegg. An improved method of treating animal fibres. Aug. 5.

18,471. T. H. Thate. Process for imparting to cotton and other vegetable fibres a silk-like appearance. Aug. 9.

18,472. S. Wolf. Manufacture of cloth felt and wadding from cellulose. Aug. 9.

18,473. S. Wolf. *See* Class XIX.

18,728. A. L. C. Nodon and L. A. Bretonneau. Improved process for decorticating and degumming Ramie and other like fibrous materials. Complete Specification. Aug. 12.

18,733. H. Ferguson. Improvements in the process and machinery for breaking and scutching fibrous plants and stems, such as brown rhea, ramie, hemp, jute, and the like. Aug. 12.

Complete Specifications Accepted.

1896.

19,217. A. Monforts. Machines for finishing yarns in the form of hanks or skeins. Aug. 4.

22,605. S. Whittaker. Machinery for preparing or spinning cotton or other fibrous materials. Aug. 4.

28,376. H. Giesler. Manufacture of mixed or particoloured fabrics. Aug. 4.

28,870. O. Seyfert. Process for giving a silk appearance to cotton. July 28.

1897.

11,804. A. H. Prinz. Treatment of jute, bast, chinagrass, rhea, and like plants for use in textile industries. Aug. 11.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

Applications.

17,173. H. H. Lake.—From D. Mattei, Italy. Improvements in apparatus for dyeing textile fabrics. Complete Specification. July 20.

17,452. J. Smith. Improvements in or connected with dyeing textile materials aniline black, partly applicable also to dyeing textile materials other colours. July 24.

18,487. G. B. Ellis.—From J. Heilmann et Cie., Alsace. Improvements in the manufacture and in the production of coloured designs on woven fabrics. Aug. 9.

18,527. J. Grossmann. Improved method of dyeing cotton piece goods. August 10.

Complete Specifications Accepted.

1896.

19,428. J. Schneider. Improvements in the treatment of fibres and fibrous or textile materials to improve the appearance or finish and facilitate the operation of dyeing. July 28.

20,846. B. Lee. Vessels for dyeing purposes. July 28.

21,492. F. Davies and A. Liebmann. Machine for dyeing, finishing, sizing, washing, or similarly treating yarn. Aug. 4.

1897.

14,831. M. L. Kellogg. Improvements in dyeing hair or fur. Aug. 4.

VII.—ACIDS, ALKALIS, and SALTS.

Applications.

17,313. H. J. Krebs. Improvements in process of and apparatus for distilling ammonia. Complete Specification. July 22.

17,476. R. H. Davidson and The United Alkali Company, Ltd. Improvements in and apparatus for the manufacture of flowers of sulphur. July 24.

17,609. I. L. Roberts. Process of and apparatus for the manufacture of metallic carbides. Date applied for Dec. 29, 1896, being date of application in United States. Complete Specification. July 27.

17,683. F. H. Bowman and F. E. Bowman. Improved means and apparatus for electrolytically decomposing salts of the alkaline and earthy metals for the manufacture of caustic bleaching powder, chlorine, and other products contained in them. July 28.

17,834. E. F. Turner. Improvements in the treatment of metallic chlorides such as those produced from sulphide ores. Complete Specification. July 29.

17,930. C. J. Yarnold. Improvements in or relating to the manufacture of hydrogen peroxide. July 30.

17,931. J. C. Butterfield. Improvements in the oxidation of ferrous salts. July 30.

18,189. O. Guttmann. Improvements in apparatus for converting nitric peroxide into nitric acid. Complete Specification. Aug. 4.

18,271. E. L. Lalbin. An improved process for producing alkaline prussiates and cyanides and reproducing the reagents employed. Aug. 5.

18,417. O. Luppe. An improved process for the recovery of perchloride of iron from the solutions of perchloride of iron used for etching purposes. Aug. 7.

18,614. J. S. Rigby. New or improved applications of "waste" sulphuric acid, and apparatus for the utilisation thereof and the production of sulphates therefrom. Aug. 11.

Complete Specifications Accepted.

1896.

15,977. L. L. Billandot. Process of manufacturing phosphorus. July 28.

20,068. W. R. Clarke and The United Alkali Company, Ltd. Means for effecting the absorption of chlorine gas, applicable in the manufacture of chlorates and bleaching liquor. Aug. 11.

20,883. G. P. Lewis and R. A. Cripps. Improved means for manufacturing and recovering cyanides, and apparatus therefor. Aug. 4.

21,178. A. Schmitz and E. C. W. Toerges. Obtaining tartaric acid and tartrates. Aug. 11.

21,953. T. Savage. Production of copper sulphate. July 28.

22,718. G. Kynoch and Co., Ltd., and A. T. Cocking. Process for denitrating acid which has been used in the nitro-glycerine manufacture. Aug. 18.

1897.

12,266. The Compagnie Generale l'Alumine. Manufacture of double chloride of aluminium and soda. July 28.

VIII.—GLASS, POTTERY, and ENAMELS.

Complete Specifications Accepted.

1897.

11,630. C. C. Schirm and O. Lessing. Ceramic production of grinding agents. Aug. 18.

14,481. C. E. Blue, jun. Machines for the manufacture of glassware. Aug. 4.

15,618. T. Küpper. Apparatus for glass-blowing. Aug. 11.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, and CEMENTS.

Applications.

17,419. N. T. Ashton and J. Crompton. A new or improved manufacture of hydraulic cement. July 23.

18,046. J. Foster. Improvements in the construction of kilns for burning lime and other similar material. Aug. 3.

18,246. F. D. Cummer. Improved means applicable for use in the manufacture of cement. Aug. 5.

18,391. J. Parnall. Improvements in the manufacture of plaster or cement. Aug. 7.

18,672. E. Robbins. An improved compound concrete and means and methods of producing the same, and architectural and other works thereof, including shipbuilding and maritime works of concretion in general. Aug. 12.

18,779. T. Holden and C. Major. Improvements in apparatus for the manufacture of cement. Aug. 13.

Complete Specifications Accepted.

1896.

20,603. G. G. M. Hardingham.—From F. D. Cummer. Means applicable for use in drying cement, slurry, liquid, clay, and other substances in like condition. July 28.

20,612. P. Kleber. Manufacture of artificial stone. Aug. 18.

1897.

9316. L. S. Kassoo. Treating and assembling the ingredients for the manufacture of paving compositions. Aug. 18.

12,024. J. C. Rombach and E. S. Restieaux. Improvements in cements for resisting the action of acids, acid vapours, and heat. July 28.

15,050. P. Rüchert. Artificial stones. Aug. 18.

X.—METALLURGY, MINING, Etc.

Applications.

17,016. H. Birbeck. — From T. B. Birbeck, Western Australia. Improvements in or relating to extracting precious metals from their ores and apparatus therefor. July 19.

17,040. B. H. Springett.—From A. L. Lanseigne and A. H. V. Leblanc, France. Improvements in the process of gilding, silvering, nickeling, bronzing, plating, oxydising, and the like, on aluminium and its derivatives. July 19.

17,401. J. Armstrong. Improvements in the treatment of complex ores containing zinc. July 23.

17,566. L. Davies. Improvements in or connected with piles for making bar iron. July 27.

17,600. C. F. Claus. Improvements in the production of metallic zinc from blende. July 27.

17,624. H. E. Fry and R. H. Everitt. Improvements in the method of and apparatus for extracting lead with other metals from ores. July 27.

18,007. H. L. Sulman and H. F. K. Picard. Improvements in the recovery of gold from certain metallic products obtained in the extraction of precious ores. July 31.

18,135. T. Doherty. Method and means for manufacturing steel direct in a cupola. Aug. 3.

18,217. W. J. Wigg and E. G. Ballard. Improvements in the separation and recovery of zinc from liquors containing zinc in solution with iron and other metals. Aug. 5.

18,252. T. B. McGhie. Improvements in the recovery of precious metals from their solutions. Aug. 5.

18,339. F. B. Aspinall and E. C. Ekstromer. Improvements relating to the extraction of precious metals from their ores. Aug. 6.

18,607. W. Allen. A solder for uniting aluminium. Aug. 11.

18,640. J. Landin. Wet process for extracting gold from gold ores or gold ore waste. Complete Specification. Aug. 11.

18,712. A. Stein. Improvements in annealing or heating furnaces. Aug. 12.

18,731. F. George. An improved process of soldering aluminium. Complete Specification. Aug. 12.

18,787. J. B. Hannay and A. Cruikshank. Improvements in extracting gold and other metals from ores or minerals containing them, and apparatus therefor. Aug. 13.

18,802. J. Hall. Improvements in the manufacture of mild steel. Aug. 13.

Complete Specifications Accepted.

1896.

17,941. B. Thomas and J. W. Thomas. Elimination of arsenic from metallic copper. Aug. 11.

23,597. B. H. Thwaite and F. L. Gardner. Method of working iron blast furnaces as to utilise the gases evolved for producing motive power. Aug. 11.

1897.

8763. C. H. Foote, W. R. Walker, and E. A. S. Clarke. Manufacture of steel by the Bessemer or open-hearth process. Aug. 18.

11,996. G. Deer. Smelting of copper, and apparatus for that purpose. July 28.

XI.—ELECTRO-CHEMISTRY and ELECTRO-METALLURGY.

Applications.

17,026. C. D. Abel.—From C. N. Seduff, Russia. A new galvanic element. Complete Specification. July 19.

17,081. F. J. Patten. Improvements in and connected with electrical furnaces. July 20.

17,105. W. J. L. Sandy. Improvements in primary batteries. July 20.

17,158. W. Rowbotham. Improvements in electric batteries. July 20.

17,161. J. Hargreaves. Improved means applicable for use in the electrolysis of salts. July 20.

17,315. C. A. Meygret. An improved process for electrolytically purifying and filtering saccharine liquids or solutions. July 22.

17,316. L. G. Garcia. Improvements in accumulators. Complete Specification. July 22.

17,319. W. J. S. Barber-Starkey. Improvements in plates for secondary batteries. July 22.

17,471. C. D. Abel. From Siemens and Halske, Germany. Improvements in electric furnaces. July 24.

17,579. A. W. Southey. Improvements in secondary batteries. July 27.

17,837. J. Holloway. Improvements in apparatus for the electro-deposition of metals. July 29.

18,029. C. Marschner. An improved process for the manufacture of electrode plates for storage batteries. July 31.

18,176. A. Heil. Improvements in or relating to electrical accumulators or storage batteries. Aug. 4.

18,430. F. Pescetto. Improvements in the manufacture of electric accumulators. Complete Specification. Aug. 7.

18,564. E. J. Clubbe, A. W. Southey, and The Electric Motive Power Company, Ltd. Improvements in secondary batteries. Aug. 10.

18,566. E. J. Clubbe, A. W. Southey, and The Electric Motive Power Company, Ltd. Improvements in and connected with secondary batteries. Aug. 10.

18,616. R. Hurley, H. Cooney, J. Glover, and G. P. Campbell. Improvements in or connected with electrolytes for primary electric batteries. Aug. 11.

18,628. C. H. Cole. Improvements in portable electric primary batteries. Complete Specification. Aug. 11.

18,635. G. P. Torelli. Improvements in or relating to primary batteries. Aug. 11.

18,658. A. Nodon. Improvements in porous diaphragms and vessels for cells and accumulators. Aug. 11.

18,796. W. M. McDougall. Improvements in and connected with secondary or storage batteries. Aug. 13.

18,835. S. Ziani de Ferranti. Improvements in electrolytic cells. Aug. 14.

18,884. C. Zimmer. Improvements in and relating to the manufacture of plates for electric accumulators. Complete Specification. Aug. 14.

Complete Specifications Accepted.

1896.

16,597. R. M. J. Heartey and B. Germain. Primary batteries. July 28.

18,046. M. M. Brophy and J. D. F. Andrews. Electric accumulators or secondary batteries. Aug. 18.

18,951. A. Conder and E. P. Michelot. Improvements in electrical accumulators. July 28.

21,027. W. L. Wise.—From The Aluminium Industrie Aktien Gesellschaft. Electrolytic apparatus. July 28.

21,509. J. G. A. Rhodin. Improved electrolyser or electrolytic apparatus. July 28.

21,749. The Sussmann Electric Miners' Lamp Co., Ltd., and S. A. Rosenthal. Active material for secondary battery plates. Aug. 11.

22,595. A. G. New. Electric accumulators. Aug. 18.

24,473. H. Leitner. Improvements in primary and secondary galvanic batteries. Aug. 4.

1897.

12,519. W. Majert. Plates for accumulators. Aug. 18.

12,827. V. Jeanty. Improvements in electric primary and secondary batteries. Aug. 11.

15,013. S. Pitt.—From L. Bomel and Société Bisson, Bergès, et Cie., France. Negative electrode for zinc accumulators. July 28.

15,510. J. Y. Johnson.—From J. P. Clare, G. E. Hatch, J. H. Taylor, United States. Improvements in secondary or storage batteries or accumulators and in the manufacture of the same. Aug. 4.

15,513. C. A. Jensen.—From P. Jebsen, Norway. Producing peat charcoal by means of an electrical current. Aug. 4.

16,069. L. W. Pullen. Electric batteries. Aug. 11.

16,361. E. W. Junger. Accumulator plates for secondary batteries. Aug. 18.

16,362. E. W. Junger. Electrodes of electric accumulators. Aug. 18.

XII.—FATS, OILS, and SOAP.

Applications.

17,405. A. J. Boult.—From La Société "Meurice-Save-Simon," Belgium. Improvements in or relating to preparations for laundry purposes. July 23.

17,738. R. Gesell. Improved process for making soap. Complete Specification. July 28.

18,251. J. Hopkinson. Improvements in the treatment of wool grease, raw or distilled, and its derivatives. Aug. 5.

18,550. A. Euston. Improvements in apparatus for treating flax and other oleaginous seeds for the extraction of oil therefrom. Complete Specification. Aug. 10.

Complete Specifications Accepted.

1896.

17,118. F. V. Klever. Lubricants. July 28.

19,903. H. Hadfield. Manufacture of soap and in the frames used in the manufacture of soap and method of cutting or slabbing soap. Aug. 18.

21,048. E. S. Wilson and E. Stewart. Manufacture of soap. July 28.

1897.

15,156. A. Jolles. Manufacture of soaps. Aug. 4.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

17,674. E. Parkin. A new or improved process for treating vulcanised rubber to recover the india-rubber therefrom. July 28.

17,774. J. Noad. An improvement in the manufacture of white lead. July 29.

17,775. J. Noad. An improvement in the manufacture of white lead. July 29.

17,897. J. Livingstone and A. F. Crawford. An improved imperishable carbon paint. July 30.

17,936. W. Ramsay. Improvements in extracting and purifying gutta-percha. July 30.

18,331. W. Ramsay. Improvements in and relating to the treatment of gutta-percha and the like. Aug. 6.

18,519. I. Goldblum. Improvements in or connected with the manufacture of varnishes, paints, and the like. Aug. 10.

18,533. D. J. Ogilvy. Improvements in the method or process of making printing inks. Complete Specification. Aug. 10.

Complete Specifications Accepted.

1896.

19,310. J. Wetter.—From M. Weinrich. Revivifying bone black or animal charcoal. July 28.

19,788. A. C. J. Charlier. Manufacture of white lead. Aug. 18.

23,716. H. H. Lake.—From G. Hermeguies. Manufacture of zinc white. Aug. 18.

1897.

2108. G. L. Burnham. Paint for ships' bottoms. Aug. 18.

15,180. P. S. Watkins and P. J. Nunn. Manufacture of sealing wax. Aug. 4.

15,330. B. J. B. Mills.—From La Granitine Société Anonyme, Belgium. Manufacture of agglutinant enamels.

XIV.—TANNING, LEATHER, GLUE, AND SIZE.

Applications.

17,529. H. H. Lake.—From L. Dufour, Italy. *See* Class XX.

18,332. E. J. Thibaut. Improved means to be used in tanning hides and skins. Aug. 6.

Complete Specifications Accepted.

1896.

22,103. J. Forster. Method of and means for tanning hides and the like. Aug. 18.

1897.

12,099. C. S. Dolley. Processes of tanning. July 28.

12,449. P. G. Sanford. Treatment of blood and other albuminous matter, both animal and vegetable, for use in manufactures and the arts. July 28.

XV.—AGRICULTURE AND MANURES, Etc.

Applications

17,926. W. E. Rowlands. Manufacture of manure from waste leather and phosphate of lime. Complete Specification. July 30.

18,191. J. Hudson. Improvements in the manufacture of manure from wool refuse. Aug. 9.

XVI.—SUGARS, STARCHES, GUMS, Etc

Applications.

17,444. J. Keil. Improved process and apparatus for the production of starch and gluten out of cerealia and legumines. Complete Specification. July 24.

18,429. L. Sternberg. Improvements in and connected with the extraction of sugar from molasses by the use of barium compounds. Complete Specification. Aug. 7.

Complete Specifications Accepted.

1896.

19,815. G. Ranson. Process for refining and purifying sugar. Aug. 18.

1897.

1467. E. R. Savigny. Sweetening substance and a process for its manufacture. Aug. 4.

15,295. L. P. Bauer. Manufacture of glucose or sugar. Aug. 4.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

17,816. W. J. Glennon and A. Y. Oastler. An improved apparatus for aerating and sterilising beer. July 29.

18,112. H. H. Lake.—From L. S. Langville and H. Tanszky, United States. Improvements in and relating to the manufacture of yeast. Complete Specification. Aug. 3.

18,256. J. E. Carroll. Process and apparatus for maturing or treating wine and spirits. Aug. 5.

18,645. F. S. Masters and J. C. Shears. Improvements in the manufacture of beer and other liquids and in apparatus therefor. Aug. 11.

18,713. D. J. Etty and C. R. Long. An improved process of and apparatus for ageing whisky. Complete Specification. Aug. 12.

18,793. R. D. Bailey and L. P. Ford. An improved combined mashing vessel, raw grain conversion vessel, brewing copper, cooling apparatus, and hop back for boilers' and distillers' use. Aug. 13.

Complete Specifications Accepted.

1896.

21,011. G. E. Jacquemin. Improved method of and apparatus for the manufacture of pure yeasts. Aug. 18.

1897.

973. H. Muller-Thurgau. Process for production of non-alcoholic or feebly alcoholic fruit juices (fruit and grape wines) which may be kept or preserved without change. Aug. 18.

12,958. H. Krawsz and L. Vass. Process for utilising the waste of hops and malt of breweries. Aug. 4.

XVIII.—FOODS, SANITATION, Etc., AND DISINFECTANTS.

Applications.

A.—Foods.

17,361. H. H. Christiansen. Improved process of treating margarine. July 23.

17,512. A. Searl. *See* Class XX.

17,915. S. Pitt.—From The Chemische Fabrik auf Actien vormals E. Schering, Germany. The production of food preparations containing casein. July 30.

18,072. J. Simmons. Food product. Aug. 3.

18,714. E. C. L. Kressel and T. Hill-Jones. The manufacture of an improved alimentary extract. Aug. 12.

B.—Sanitation.

17,261. J. B. Petrie. An improved method of purifying sewage and other foul waters by means of a secondary process following other primary processes. July 22.

18,555. W. D. Scott-Moncrieff. Improvements in or relating to the purification of sewage, and apparatus therefor. Aug. 10.

18,839. B. C. Farmer. Improvements in the construction of precipitation tanks used in the filtration and treatment of sewage and other foul waters. Aug. 12.

C.—Disinfectants.

17,464. W. Loebinger. Improvements in or relating to disinfection by means of formaldehyde. Complete Specification. July 24.

18,178. W. L. Wise.—From Knoll and Co., Germany. Improvements in the manufacture of antiseptic substances. Aug. 4.

18,250. H. Oppermann. Process for the preparation of easily soluble compounds of formic aldehyde for preserving, disinfecting, and therapeutic purposes. Complete Specification. Aug. 5.

18,656. E. T. C. Weihers. Improved mixture for the destruction of microbes of the contagious diseases of animals, also applicable for the treatment of animal skins. Aug. 11.

Complete Specifications Accepted.

A.—Foods.

1896.

23,126. W. P. Thompson.—From Kietz and Co. Manufacture of strengthening and flavouring food extracts. Aug. 18.

1897.

7649. K. Franz. Improved food preparation and process for the manufacture thereof. Aug. 18.

B.—Sanitation.

1896.

23,042. D. Cameron, F. J. Commin, and A. J. Martin. Treatment of sewage or other liquids. Aug 4.

XIX.—PAPER, PASTEBOARD, Etc.

Applications.

17,736. T. Schiffer. Process for producing waterproof and washable glazed chromo-paper. Complete Specification. July 28.

18,473. S. Wolf. Improved manufacture of blotting paper, filter paper, and soft fibre-felt. Aug. 9.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

Applications.

17,013. P. Sylva. A new or improved process for manufacturing chloride of ethyl. July 19.

17,169. A. F. Llobet. Improvements in the manufacture of milky juice of cereals. Complete Specification. July 20.

17,246. J. E. Lyndall and L. R. Orr. A liquid essence, and process for its manufacture. July 21.

17,409. O. Imray.—From the Society of Chemical Industry in Basle, Switzerland. Manufacture of new therapeutical compounds. Complete Specification. July 23.

17,512. A. Searl. Improvements in processes for the preparation of soluble and highly concentrated extracts of tea and coffee. July 26.

17,529. H. H. Lake.—From L. Dufour, Italy. An improved process for the preparation of extracts used in tanning and as colouring agents. July 26.

17,692. L. Lederer. Improvements in the process for preparing the halogen derivatives of acetone. Complete Specification. July 28.

17,693. L. Lederer. Improvements in the process for the preparation of hexymethylen-tetramin and its derivatives. Complete Specification. July 23.

18,478. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of pharmaceutical compounds. Aug. 9.

18,698. T. Bird. A process for the treatment of artificial camphor either in the crude state or after resublimation. Aug. 12.

Complete Specifications Accepted.

1896.

16,860. H. Higgins. Separation of caseinogen or casein from separated milk, and its preservation in a dry state. July 28.

18,981. A. Koelliker. Acid salts of piperazine and fatty acids, and the production therefrom of double salts of lithia and piperazine. July 28.

19,744. J. J. A. Trillat. Production of formic aldehyde vapours. Aug. 18.

20,622. G. B. Ellis. — From La Société Chimique des Usines du Rhône, anciennement G. P. Monnet et Cartier. Distributing formic aldehyde vapours. Aug. 4.

20,697. A. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Schering. Production of a vinyldiacetonalkamine, and of anæsthetics therefrom. July 28.

20,773. J. J. A. Trillat. Production of vapours of formic aldehyde, and apparatus for the same. Aug. 4.

21,106. G. B. Ellis.—From La Société Chimique des Usines du Rhône, anciennement G. P. Monnet et Cartier. Manufacture of vanillin. Aug. 4.

29,447. O. Imray.—From F. Hoffmann, La Roche, and Co. Pure acetosulphanilate salts. Aug. 18.

1897.

14,610. B. Willcox. — From C. F. Boehringer and Soehne, Germany. Manufacture or preparation of theobromine. July 28.

14,611. B. Willcox. — From C. F. Boehringer and Soehne, Germany. Manufacture of iso-eugenol and derivatives thereof. July 28.

14,612. B. Willcox. — From C. F. Boehringer and Soehne, Germany. Production of trichloropurine. July 28.

XXI.—PHOTOGRAPHY.

Application.

17,122. G. J. Sershall and J. H. Kirk. An improvement or improvements in the production of coloured photographs. July 20.

Complete Specification Accepted.

1897.

11,133. G. Selle. Photographic dark backs for successive exposures for the purpose of colour photography. Aug. 4.

XXII.—EXPLOSIVES, MATCHES, Etc.

Applications.

17,416. W. Hope. Improvements in pellets and grains of gunpowder of any kind. July 23.

17,417. W. Hope. Improvements in gunpowders and other explosives. July 23.

18,563. J. Caveri. Improvements in the manufacture of matches. Complete Specification. Aug. 10.

18,642. F. M. Hale, J. Deas, and J. B. Reavil. Improvements in percussion fuzes for explosive projectiles or shells. Aug. 11.

Complete Specifications Accepted.

1896.

17,307. C. E. Bichel. Manufacture of nitro-glycerine explosives. Aug. 11.

18,034. M. Bielefeldt. Manufacture of explosives. Aug. 18.

22,162. J. C. Hamilton. Explosive compounds for blasting purposes. Aug. 11.

22,190. G. Kynoch and Co., Ltd., and A. T. Cocking. Manufacture of explosives. July 28.

22,718. G. Kynoch and Co., Ltd., and A. T. Cocking. *See* Class VII.

1897.

14,196. F. Volpert. Manufacture of explosives. Aug. 4.

14,989. J. T. Maguire.—From The Diamond Match Company, United States. Improvements in matches. July 28.

15,499. H. Maxim. Manufacture of explosive compounds. Aug. 4.

PATENTS UNCLASSIFIABLE.

Application.

17,610. J. Carnrick. A process of preparing protonuclein. Complete Specification. July 27.

Complete Specification Accepted.

1896.

17,717. F. J. Jones and G. W. Jones. A chemical compound. Aug. 11.

Printed and Published by Eyre and Spottiswoode, East Harding Street, London E.C., for the Society of Chemical Industry.

THE JOURNAL

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NOTICES.

Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, part of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

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CHANGES OF ADDRESS.

Ablett, A. W., l/o Johannesburg; I.X.L. Cyanide Works, Sheba G. M. Co., Eureka City, Barberton, S.A.R.

Bamber, H. K. G.; Journals to Hillside, Greenhithe, Kent.

Bischof, G., l/o Bloomsbury; 9, Hythe Road, Willesden Junction, N.W.

Bowman, R., l/o Elabouga; Victoria Chemical Works, Widnes.

Briggs, J. F., l/o India; 70, St. Ann's Hill, Wandsworth, S.W.

Church, E. D., jun., l/o Trenton; 65, Wall Street, New York, U.S.A.

Cooke, A. W., l/o Johannesburg; c/o Brotherton and Co., Holmes Street, Dewsbury Road, Leeds.

Crow, H. W.; Journals to 11, Norwich Road, Forest Gate, E.

Douglas, Wm., l/o Stamford Hill; Diamond Plantation, Demerara.

Elworthy, H. S., l/o India; c/o Henry S. King and Co., Cornhill, E.C.

Farrington, T., l/o Waterloo Place; 5, Summerhill Terrace, Cork, Ireland.

Garton, R. S., l/o Smethwick: retain Journals until further notice.

Griffith, D., Agnew, l/o Isle of Man; c/o Mitchell and Co., 27, King Street, Liverpool.

Horsmar, F., l/o Forest Gate; 45, Hyde Vale, Greenwich, S.E.

Jarvis, Talbot, l/o Burton; Castle Lodge, Bedford.

Langer, Dr. C., l/o Hampton House; Upmore, Milverton, Leamington.

McEwen, Atholl F., l/o Laurie; retain Journals until further notice.

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Queeny, John, F., l/o New York; c/o Meyer Bros. Drug Co., St. Louis, Mo., U.S.A.

Raymond, C. W., l/o Forest Gate; 32, Campbell Road, Bromley-by-Bow, E.

Russell, Dr. W. J.; Journals to 34, Upper Hamilton Terrace, N.W.

Sedding, G. H. P., l/o Anita; River Junction, Gadsden Co., Florida, U.S.A.

Shepherd, H. H. B., l/o Victoria Docks; Northcote, Mount Pleasant Lane, Upper Clapton, N.E.

Smith, H. Wood, l/o Battersea; 22, Great Winchester Street, E.C.

Thornton, H., l/o London; Redbourn House, Ford Road, Ashford, Middlesex.

Twitchell, E., l/o Mount Auburn; 9, Cumberland Building, Avondale, Cincinnati, Ohio, U.S.A.

Welch, J. Cuthbert, l/o Kaslo: Trail, British Columbia.

Wilson, Jno., l/o Stratford; 74, Bearwood Road, Smethwick, near Birmingham.

Zinkeisen, Dr. W., l/o George Street; 66, Virginia Street, Glasgow; Journals as before.

GOVERNMENT LABORATORY.

After October 4th, 1897, the address of the Government Laboratory will be Clement's Inn Passage, Strand, W.C., instead of Somerset House. The change affects the following members:—

	*Thorpe, Dr. T. E.	
Bannister, R.	*Holmes, J.	*Proctor, C.
Davis, H. W.	Hooper, E. Grant.	Stoker, G. N.
*Helm H. J.	Lucas, A.	*Woodward, J.

The Journals of those marked with an asterisk are to be addressed as heretofore.

Deaths.

Alsberg, Dr. M., Messrs. Sondheim Alsberg and Co., New York, U.S.A.

Fletcher, G., Poplar Ironworks, King Street, Poplar, E.

Journal and Patent* Literature.

I.—PLANT, APPARATUS, AND MACHINERY.

Zinc Plates as Covering for Steam Pipes. Russner. Mitt. aus d. Praxis des Dampfk. u. Dampfm.-Betr. 1896, 334; Proc. Inst. Civil Eng. 1897, **128**, [2], 49.

The author found that, compared with the naked pipe, a covering of one zinc plate (leaving a layer of air between plate covering and pipe) caused a saving of 83 per cent. of condensed water; a covering consisting of two plates, 86·6 per cent.; whilst a felt covering 0·6 in. thick, caused a saving of 91·4 per cent. The ordinary non-conducting coverings in use, effect the following savings in condensed water:—Kieselguhr, 0·8—0·9 in. thick, 70 to 75 per cent.; cork fibre, 2 ins. thick, 85 per cent.; soap waste, 1 in. thick, 85·6 per cent. Non-conducting compositions applied in a plastic state must be dried by the heat supplied of the steam pipe, and the cost of this drying is generally overlooked; further, owing to their greater thermal capacities, the cooling-down losses at the end of each day are much greater for the plastic coverings than for zinc plate coverings, while the latter compare favourably with the former in regard to first cost.—A. S.

Steam, Superheated. W. Ripper. Proc. Inst. Civil Eng. 1897, **128**, [2], 5—46.

The author concludes, from the results of a number of trials, that a great improvement in steam-engine economy may be effected by the use of superheated steam. The temperature should be raised sufficiently high to secure dry steam in the cylinder throughout and up to release; for this purpose, the steam should be supplied at a temperature of about 650° F. at the engine. With steam at this temperature used in a simple non-condensing engine, the same power was obtained for less than half the steam required when no superheater was used. The increased economy in steam consumption which follows the increase of temperature of the steam, is also accompanied by a proportional reduction in the extent of the heat exchange between the steam and the cylinder walls; in fact, with the highest superheats and the highest economies, the cylinder walls approach the condition of being almost perfectly non-conducting. It is stated that the superheater tubes, after

* Any of these specifications may be obtained by post by remitting 8*d*.—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

long periods of severe work, show no signs of burning, sealing, or injury of any kind, and that with proper attention as to the judicious application of lubricating oils of good quality at the working parts, no trouble is likely to arise in the lubrication of superheated steam engines. Care should be taken, by the use of suitable non-conductors, to maintain the high temperature of the steam in its passage to the engine cylinders. The steam should be superheated at least 200° F. above its normal temperature, and in some cases still higher, depending on the number of expansions, else it will part with the whole of the added heat to the cylinder walls. The steam-admission valve of an engine to work with highly superheated steam, should be practically frictionless, so as to remove the necessity for concern as to lubrication.—A. S.

PATENTS.

Dipping Ladles, An Impt. in. O. Imray, 28, Southampton Buildings, Chancery Lane, London. From The Pennsylvania Salt Manufacturing Company, Philadelphia, U.S.A. Eng. Pat. 15,502, June 29, 1897.

The apparatus comprises an overhead track, a trolley movable thereon, a hanger depending from the trolley and having an adjustable clutch consisting of two bars or parts (the one part at the side of and longitudinally adjustable along the other part), means for locking the sliding bar in any adjusted position, and a dipping ladle supported by a roller bearing within a ring connected by a swivel to the clutch.—R. A.

Conveying and Distributing Heat to the Contents of Vessels, such as Stills and the like, or to the Interior of Steam-generating Vessels, Evaporating Pans, Boiling Pans, and the like, Improved Means for and Method of. E. Martin and A. Stapley, both of Sugar House Lane, Stratford, London, E. Eng. Pat. 16,774, July 29, 1896.

The heat is conveyed to the interior central parts of the distilling or other utensils by the employment of cones, cylinders, or other hollow heat conductors, adapted to any suitable part of the utensils.—R. A.

Filtering Fluids [Water], Impts. in the Method of. J. E. Bennett, Ridgway Street, Moss Side, Manchester, and H. Beresford, Tetlow Fold Farm, Godley, Chester. Eng. Pat. 18,103, Aug. 15, 1896.

The fluid to be filtered is caused to pass upward instead of downward, through the filtering medium, the solid matter collected being allowed to gravitate gradually to the bottom of the vessel. The filtering medium is cleaned by cutting off the supply and causing the body of filtered liquid above the medium to pass downward through the same.—R. A.

Filter Presses [Sludge], Impts in. S. H. Johnson, Stratford, London, E. Eng. Pat. 9582, April 15, 1897.

Relates to improvements in the class of filter presses such as that described in the specifications of Eng. Pats. 7045 and 11,415, both of 1886, in which fluid pressure is employed for pushing the plates or frames, which form the filter cells, into juxtaposition to effect the closing of the joints. In the present invention, the filter press is worked in combination with a differential intensifier reacting on a hydraulic ram, in which the working pressure of the feed liquor before entering the filter chambers, exerts pressure externally on the movable head or follower, in excess of the internal pressure, the condition being that the internal pressure within the cells, tending to force open the joints, shall be exceeded by the external pressure tending to force them together, so as to maintain the cell-joints fluid-tight, no matter what the working pressure may be. The external and internal pressures are both derived from the feed pressure, and are interdependent. When the sludge is very gritty, or likely to injure the interior surface or the cup-leathers of the intensifier, the external pressure is obtained from an auxiliary water-forcing receiver actuated by air pressure common to both the sludge and water forcing receivers, the intensifier in this arrangement being operated by clean water instead of by the feed liquor.—R. A.

Filtering Liquids, Impts. in the Process of and Apparatus for. E. Simoneton, 15, Rue des Halles, Paris. Eng. Pat. 11,700, May 11, 1897.

Filtering apparatus is employed into which the liquid is introduced directly, or through a transparent inspection chamber, or in which the liquid may be subjected to preliminary filtration, the filtering body within the apparatus being formed by superposed discs or rings of a flexible filtering medium, such as non-metallic fabric or paper. The rings filter edgewise and radially into a central running-off chamber or passage, and can be more or less compressed together during filtration.—R. A.

Carboys, Demijohns, and the like, Impts. in. W. R. Lake, 45, Southampton Buildings, Middlesex. From R. Spaciani, Milan, Italy. Eng. Pat. 19,297, Sept. 1, 1896.

Glass receptacles, such as carboys, demijohns, &c., are provided with protecting envelopes or coverings consisting of fibrous or cellular vegetable substances (cork shavings, sawdust, bark, chopped straw, or other waste), which are caused to adhere to the outer surface of the glass by uniting them with mastic, or by surrounding them with jackets of cloth, thin metallic plates, wire gauze, or the like.—R. A.

Concentration of Liquids [Wine, Cider, Beer, Solutions, &c.], An Improved Process and Apparatus for the. H. Baudoin and E. Schribaux, both of 11, Rue Michelet, Paris. Eng. Pat. 21,694, Sept. 30, 1896.

"The apparatus" consists "of the combination, with ordinary heaters, analysers, and condensers, of an evaporator formed of sets of superposed series of tubes, discharging the vapours at both ends and then causing them to meet and combine in a common collector, the concentrated liquors being discharged below" and conveyed to a suitable receiver. The concentrated liquors and condensed vapours can be recombined, when desired, in another receiver, which is provided with a mechanical stirring or shaking device.

—R. A.

Granulation of Crystallisable Liquids, An Improved Evaporating or Vacuum Apparatus for. J. Grosse, Kiew, Russia. Eng. Pat. 27,477, Dec. 3, 1896.

This apparatus, which is fixed in an upright position, has an internal delivery pipe, within which a helical screw, worm vanes, or propeller blades rotate on a vertical shaft. The solution to be crystallised is by these means conveyed from the lower and hotter parts into the upper and cooler parts of the liquid. An evaporative and cooling action is produced, which is stated to considerably accelerate crystallisation.—J. L. B.

Steam Boilers, An Improved Composition for Preventing Incrustation in. L. Lewis, Pentywain, Newport. Eng. Pat. 14,226, June 11, 1897.

Apples, acorns, oak apples, and the "muss" or residue left after expressing the juice from cider-apples, are ground to a pulp and strained; the juice is then mixed with a lye made from oak bark and peat ashes.—L. A.

II.—FUEL, GAS, AND LIGHT.

Monazite in Idaho. W. Lindgren. Eng. and Mining J. 1897. **64**, 69.

See under XX., page 755.

Air, Contamination of the, in the Purifying and Revivifying Houses of Gasworks. H. Drehschmidt. J. für Gasbeleucht. 1897, **40**, 517.

See under XVIII. B., page 752.

PATENTS.

Gas for Lighting and Motive Power, Impts. in the Production of, by the Cold Process, and Apparatus therefor. C. Kremer, Gilly, Belgium. Eng. Pat. 19,953, Sept. 9, 1896.

The generator is divided into several compartments by horizontal partitions, and the lowest charged with the petroleum or other carburetted liquid to be gasified. Air is then pumped in under pressure and the liquid thereby forced up a number of pipes leading to the next compartment, each pipe debouching into an atomiser shaped like a double truncated cone, whereby part of the liquid is very finely sprayed, the remainder falling back on to the floor of the compartment. This goes on at each stage until the topmost chamber is reached, and here the sprayed liquid and air impinge on a metal sheet, which retains most of the liquid particles, whilst the gas, passing through lateral screens of gauze, ascends through a large double cone fitted with similar screens (whereby the intimacy of the mixture is increased), and passes thence to the gasholder, which may be superimposed or otherwise arranged. A suitable pipe is provided for the return of the liquid left unvaporised in the top compartment, to the bottom of the apparatus, and the movement of the gasholder can be employed as a means of regulating the feed supply of oil, a constant level being maintained by a float valve in the lowest compartment.
—C. S.

Carburetted Water-Gas, Impts. in the Manufacture of, and in Apparatus therefor. W. R. Addicks, Boston, U.S.A. Eng. Pat. 1398, Jan. 19, 1897.

The improvements consist in utilising the heat of the blast gas by finally passing it through the boiler generating the steam for the water-gas, and through the feed-water heater for the said boiler. Also in allowing the temperature of the carburetted gas to recede in the mixing chamber and then gradually raising it again by passage through an intermediate heated chamber on its way to the fixer, where the maximum is attained. Then, in order to prevent the injurious condensation resulting from the usual sudden cooling of the carburetted gases, they are first conducted through the aforesaid boiler and feed-water heater and thence to the condenser, the water in which is utilised for feeding the boiler, and so reducing the amount of water required for the process. The gradual cooling of the gas also obviates the inconvenience arising from heated tarry matters in the hydraulic main and tar well.—C. S.

Carburetted Water-Gas, Improved Method and Apparatus for Making. A. C. Humphreys and A. G. Glasgow, London. Eng. Pat. 11,130, May 4, 1897.

In order to more fully utilise the heat generated in blowing up the generator, the inventors provide an additional regenerative chamber on each side of the (duplicate) apparatus, which is heated by the blast gases and serves in turn to heat the air and steam on their passage to the generator fire, so that the said air and steam may be also passed through the fixing and carburetting chambers without unduly lowering their temperature. The extra chambers are not traversed by the water-gas, this latter being drawn off from the fixing chambers into a receptacle common to both sides of the apparatus. On the completion of a gas-making run with one side of the apparatus, the reversal of the steam is secured by the same movement of the lever that shuts off communication between that side of the apparatus, with the gas receiver, and simultaneously opens the gas conduit leading to the latter from the other side.
—C. S.

Carburetted Water-Gas, Improved Process and Apparatus for Making. A. C. Humphreys and A. G. Glasgow, London. Eng. Pat. 11,131, May 4, 1897.

In this invention the patentees, instead of duplicating the entire apparatus (as in the preceding abstract), provide two extra regenerative chambers to each set of generator, carburettor, and fixer, the said chambers being heated and utilised alternately.—C. S.

Hydrogen Gas, Impts. in and connected with, and Apparatus for the Manufacture or Production of. L. V. Pratis and P. Marengo, Turin, Italy. Eng. Pat. 15,509, June 29, 1897.

To overcome the difficulties in the way of generating hydrogen from sulphuric acid and iron, the inventors propose to employ the following approximate proportions, by weight:—Broken iron, 5 parts; water, 5 parts; 50° B. sulphuric acid, 5·8 parts; these being found to produce a pasty non-caking residue, easy to remove from the apparatus and to work up for the manufacture of ferrous sulphate or Nordhausen sulphuric acid.

The apparatus consists of a generating cylinder, fitted with a valve for discharging the residue. The acid and water are run in on to the charge of iron from reservoirs at a higher level, the supply valve being controlled by the bell of the gasholder, and self-closing when the bell sinks below a certain level; or, if the gas is to be collected in receivers at high pressure, the full charge of liquids may be added at once. Purifiers are arranged between the generator and gasholder, and an excessive rate of generation is prevented by gas checks, which cause an increase of pressure in the generator, whereby the acid is driven back in the supply pipe and the evolution of gas diminished.—C. S.

Smoke consuming Hydrocarbon Furnaces, Impts. in or connected with. L. E. Fraipont, Paris. Eng. Pat. 19,860, Sept. 8, 1896.

Furnaces for burning gaseous fuel are formed of two horizontal fire-brick or tile floors, one above the other, the lower perforated with small holes to allow a regulated amount of air to enter, the upper perforated with larger holes having flanged caps of suitable refractory material, while between the two, pass several parallel gas-supply pipes, each having a number of nozzles, one under each capped opening. In use, both floors become heated. The air is heated while passing through the lower floor, and the gas also is heated before reaching the nozzles. The gas and air are thoroughly mixed by the caps, and form a compound which there ignites.—R. S.

Furnaces, Impts. in Apparatus for Burning Liquid Fuel in. C. MacC. Sharp, Greenock, N.B. Eng. Pat. 21,341, Sept. 26, 1896.

A vaporiser is fixed within the furnace, being connected at its upper end to a supply of water or steam under pressure, whilst the steam generated or superheated therein is led off by a pipe passing nearly from the top and through the bottom to a jet nozzle or nozzles having a chamber connected to an oil supply tank. The vaporiser is first heated by externally applied heat, as by a small oil flame, and the steam formed, mixed with the oil, is ignited at the nozzle, the flame being directed on to the vaporiser. When the latter is sufficiently hot, live steam is passed through it instead of water.—R. S.

Combustible Compound or Fuel, An Improved. D. D. Esson, Kennington Park, London. Eng. Pat. 20,895, Sept. 21, 1896.

The combustible compound is intended for burning in a closed chamber without the addition of air, and, if desired, in contact with water. It is proposed to form it of the following materials:—Nitroglycerin, 55 parts by weight; dry guncotton, 30 parts; vaselin, 10 parts; and camphor, 5 parts. An apparatus for using this fuel in the form of sticks is described in Eng. Pat. 20,894 of 1896.—R. S.

Heating by Means of Liquid Hydrocarbons, Apparatus for. S. Pitt, Sutton, Surrey. From Compagnie Internationale des Procédés Adolphe Seigle, Paris. Eng. Pat. 14,844, June 18, 1897.

The apparatus consists of:—(1st) a closed reservoir containing water and air under pressure; (2nd) a helical tube for vaporising water; (3rd) a reservoir for liquid hydrocarbons; (4th) a sprayer; and (5th), suitable connecting pipes, cocks, a gauge, and a compressed air and steam receiver. Air under pressure is led from the closed reservoir to the receiver and thence to the sprayer, to which also the liquid

hydrocarbons are led. These are then ignited, and the flame passing through the helical tube, raises its temperature. Water is now allowed to pass from the closed reservoir to the helical tube, and the steam therefrom to the receiver and sprayer, the air supply being cut off. An extremely hot flame is said to be produced.—R. S.

Acetylene Gas, Impts. in Apparatus for the Generation and Storage of. A. F. Bowers, Paris. Eng. Pat. 13,511, June 18, 1896.

The improvements claimed comprise means for regulating the supply of water to the generator by the pressure of the gas in the latter. The pressure causes depression of the level of the feed water in one limb of a U-tube below the outlet to the generator, or moves a piston which cuts off the supply. Several forms of generating apparatus embodying such regulating devices are described. They are designed to obviate the necessity of a gasholder.—J. A. B.

Acetylene Gas Generator, An Improved. M. Duffield, Slough, Bucks. Eng. Pat. 17,646, Aug. 10, 1896.

Laterally perforated cells, packed with carbide, are admitted to a vertical cylinder through its base, which is then closed by a lid. Several of these generators are ranged round a bell gasholder, and water is admitted by a siphon device to each cell in turn until all are exhausted.—J. A. B.

Acetylene Gas for Lighting Purposes, Impts. in and relating to Apparatus for Generating. H. H. Lake, London. From O. Grenier and J. Grand, both of Lyons, France. Eng. Pat. 17,904, Aug. 12, 1896.

The invention comprises a hopper with conical valve closing on knife edges and containing calcium carbide in grains 1 to 2 mm. in diameter, which are fed into a large tank of water at the base of the apparatus. The hopper and mechanism can be withdrawn from the receiver in which they are placed merely by disjoining water seals. Spy-glasses in the receiver and purifying apparatus are described.—J. A. B.

Acetylene, Impts. in or relating to the Generation and Storage of. G. W. Gaskell and R. R. Gibbs, both of Liverpool. Eng. Pat. 20,074, Sept. 10, 1896.

The bell of a gasholder has a central tube reaching, when the bell is grounded, nearly to the bottom of the tank. A cylinder with perforated bottom contains carbide, and is supported in the tube by a tightly fitting plug. The gas escapes from this generating cylinder by a non-return valve to the gasholder, the rising of which lifts the carbide from the water.—J. A. B.

Acetylene Gas, Impts. in Apparatus for Generating and Storing. W. P. Thompson, London. From V. I. Comucci, San Giovanni di Val D'Arno, Italy. Eng. Pat. 20,694, Sept. 18, 1896.

A generator consisting of a container, bucket, and carbide basket, communicates by two tubes controlled by a single cock with the lower of two containers of a gasholder, the upper container of which has a float connected to an electric bell, and contains water which reaches the generator through one tube, while gas returns to the lower container through the other tube. A method of hermetically closing the generator is also claimed.—J. A. B.

Acetylene Gas, Impts. in and relating to Apparatus for the Generation and Storage of. A. Kay, Doune, Perth. Eng. Pat. 21,351, Sept. 26, 1896.

One or more generating vessels are placed at an inclination near a bell gasholder, and the supply of water to them from the tank of the latter is controlled by the movements of the bell.—J. A. B.

Acetylene Gas, Impts. in and relating to Generators for. A. H. Barker, London. Eng. Pat. 21,698, Sept. 30, 1896.

In a generator with overhead water tank, the carbide container fits into a receiver attached to the roof of the generator. The receiver has cocks to prevent communication between the generator and the air during refilling of the container. The gas generated, passes to an overhead bell gasholder, the inlet to which is closed by the rising of the bell; any increase in pressure in the generator then drives the water away from the carbide.—J. A. B.

Acetylene, Impts. in Apparatus for Generating and Storing. E. Chesnay, Dijon, L. Pillion, Dijon, and C. Bertolus, St. Etienne, France. Eng. Pat. 21,758, Oct. 1, 1896. (Under Internat. Convention.)

The water in a generator is in communication with an annular ring which forms the counterweight of a gasholder bell and surrounds the tank. Water flows from the generator to the ring when the bell rises, and the generation of gas is thus automatically controlled. A safety pipe, the orifice of which becomes unsealed before the bell rises unduly, is also claimed.—J. A. B.

Acetylene Gas, Improved Process and Apparatus for the Production and Treatment of. H. E. Oving, Rotterdam. Eng. Pat. 23,669, Oct. 24, 1896.

To facilitate proper combustion of acetylene in any suitable burner, the patentee forms a mixture of the gas and air by letting the gas under the pressure at which it is generated, play upon the blades of a drum, which it rotates. Another drum on the same shaft is also thus rotated, and draws in air, which mixes with the gas in the space above the drums. —J. A. B.

Acetylene or other Gas for Lighting, Heating, or other Purposes; Impts. in or relating to the Treatment of. H. E. Oving, Rotterdam. Eng. Pat. 23,670, Oct. 24, 1896.

This invention consists in so proportioning the dimensions of the drums referred to in the preceding specification (23,669) that the mixture of acetylene and air is made in the desired proportions.—J. A. B.

Acetylene Gas Generators, Impts. in. H. K. Spence, A. Beveridge, W. G. Beveridge, and S. M. Beveridge, all of Kirkcaldy. Eng. Pat. 29,554, Dec. 23, 1896.

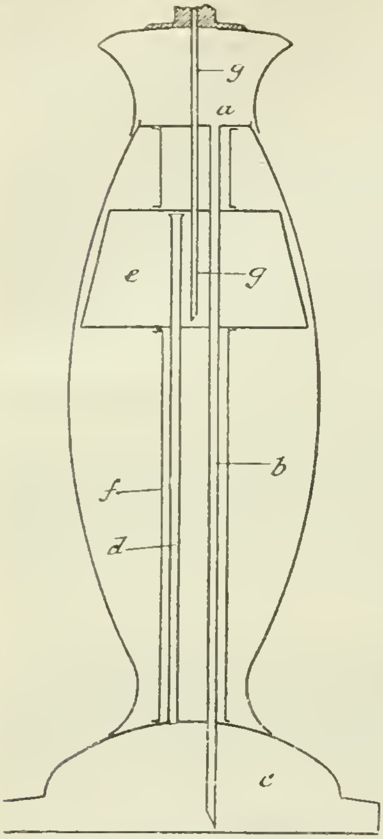
Within a tank with removable lid is a gasholder bell suitably guided, and engaging, as it falls and rises, with catches, which operate valves regulating the admission of water to carbide containers below the tank. By a special device, when one container is exhausted, the water supply is automatically diverted to the other.—J. A. B.

Acetylene Gas, Impts. in Apparatus for Producing. F. Trendel, Berlin. Eng. Pat. 14,015, June 8, 1897.

Several carbide receptacles are so arranged in a generator that their contents are successively precipitated into the water by its rise. A telescoping safety device for acetylene gasholders is also claimed.—J. A. B.

Petroleum Incandescence Burner without Wick. G. Shenton, London, and D. A. F. de Villepigue, Paris. Eng. Pat. 16,351, July 23, 1896.

The body of the lamp is shaped like a vase, the upper part, *a*, of which constitutes a closed receiver, from the bottom a tube, *b*, descending to the bottom of a second closed receiver, *c*, in the base of the lamp. From the top of *c* rises a tube, *d*, which ends within and near the top of a third closed receiver, *e*, supported a short distance below *a*. Finally, through the top of the receiver *e* passes an upright tube, *g*, the lower end of which is near the bottom of *e*, and the upper end of which terminates in the burner. The whole operates on the principle of the "fountain of Hero," oil poured into *a* rising in the tube *g* until it is rather more than 12 c.m above the oil level in *a*. From the top of the lamp body rises the burner, consisting of a metal block bored with a vertical passage communicating with *g*, and controlled by a screw cock; from the upper end of the passage rise tubular rods, which end in an annular chamber made between the adjacent faces of two cones, and at the upper (apex) part of the cones are an ejector, burner tube with air inlets, &c., surmounted by a mantle, as usual. Below the cones is fastened, between the hollow rods, a small cup,



serving to hold spirit for the preliminary heating of the burner. After heating the burner, the screw cock is opened, allowing the petroleum to rise to the annular chamber, where vaporisation occurs. The lamp as a whole, and the various combinations, are claimed.—H. B.

Incandescence Bodies for Illuminating Purposes, Impts. in the Manufacture of. W. H. Wheatley, Chancery Lane. From C. Schmid, Brussels. Eng. Pat. 17,605, Aug. 8, 1896.

Mantles are to be made as usual by impregnation in a solution of zirconium nitrate, 40 parts; calcium chloride, 15 parts; aluminium nitrate, 10 parts; ammonium nitrate, 29 parts; strontium nitrate, 5 parts; rubidium nitrate, 1 part.—H. B.

Incandescent Lamps for the Combustion of Petroleum, Impts. in. W. Clark, Chancery Lane. From A. Meyenberg, M. Wendorf, and S. Henlein, Frankfort-on-the-Main. Eng. Pat. 18,819, Aug. 25, 1896.

This invention relates to improvements on Eng. Pat. 23,863 of 1895 (this Journal, 1897, 36). It comprises the combination, with the expansible air-reservoir bulb contained in the upper part of the lamp base, of an air-pumping bulb situated in the lower part of the base, and connected with the reservoir bulb; a lever for operating the air-pumping bulb; and a lever for arresting the action of the air-pumping lever, the former being pressed upon and caused to act by the expanding air reservoir when sufficiently inflated. There is described and claimed a cock for the petroleum and benzoline, rendered tight against leakage by making the cock plug and the cock casing of oppositely conical form at their larger end, so as to produce a groove of V form in cross-section, into which there is forced, by means of a screw-cap or other device, a presser ring, also of V section, an intermediate leather washer having been first placed between the two. The improved benzoline and petroleum reservoirs are also claimed.—H. B.

Incandescence Lamp for Liquid Fuel. O. Helfft, Berlin. Eng. Pat. 8807, April 6, 1897.

Round the upper part of the lamp is arranged a ring-shaped reservoir for the liquid fuel. At a lower part of the lamp, and connected with this reservoir, is a smaller ring-shaped reservoir, from which rise a number of thin tubes which pass upwards within a draught tube placed above the lamp chimney, and which serve as vaporisers for the fuel. The tubes meet at the top of the lamp in a small dome, from which two tubes pass downwards into a large glass globe, meeting in a small chamber partly filled with asbestos wool, from which rise an ordinary incandescence Bunsen burner, mantle, and chimney. All parts are arranged centrally, so that the heat from the Bunsen flame heats the vaporising tubes, dome, &c., maintaining a supply of gas. The igniting arrangement consists of a cup fitted beneath the vaporising tubes, and connected by a supply pipe to the reservoir, and on opening a cock with a bored plug, the cup receives a measured quantity of fuel; this filling cock also forms the ordinary cock for controlling the feed pipe of the vaporising tubes. To start the lamp, the fuel in the cup is lit, when the vaporisation of the fuel commences. All the combinations of parts, and the lamp as a whole (which is adapted for suspension), are claimed.—H. B.

Rare Earths [" Kosmia "], A Process for the Separation of certain, and the Manufacture therefrom of Fabrics for use in Incandescent Gas Lighting. B. Kosman, Berlin. Eng. Pat. 18,915, Aug. 26, 1896.

The patentee claims the discovery of a new earth, which he calls " oxide of kosmium," in the cerium and didymium salts obtained from the crude liquor resulting from the solution of monazite sand. " The process of obtaining the new earthy oxide ('kosmia') from monazite sand and other cerite minerals, consists in first fractionally precipitating the hydrates of cerium, didymium, and lanthanum by adding peroxide of hydrogen and ammonia so that the solution retains an acid reaction, then treating the filtered-off solution with the same reagents, but adding so much ammonia that the solution becomes alkaline, separating the precipitate thus obtained from the liquor, dissolving the precipitate in nitric acid, treating this solution in the hot state with oxalic acid, washing the precipitate with hot acidulated water, decomposing the washed oxalate by heat, dissolving the residue in nitric acid, treating this solution with hydrogen peroxide and ammonia, so that the solution retains an acid reaction, separating the precipitate from the liquor, and adding ammonia in excess, whereby the hydrate of the new earthy oxide is obtained as a whitish precipitate." The use of the earthy oxide in incandescent lighting is also claimed.—H. B.

Mantles and the like for Incandescent Gas Burners, Impts. in and relating to. J. C. J. Steinbach, Hamburg. Eng. Pat. 14,499, June 15, 1897.

Mantles composed of 99 parts of thoria and 1 part of ceria are said to give the maximum illuminating power obtainable from these two oxides, and if the ceria be increased to, say, 4 per cent., the light is yellowish and of much less illuminating power. According to the patentee, on adding 25 per cent. of silicic acid to a mixture of 96 parts of thoria and 4 parts of ceria, " the colour of the light emitted from a mantle made of said mixture is no longer a pronounced yellow, but is pure white, and its illuminating power is even greater than that of a mantle composed of 99 parts of thorium oxide and 1 part of cerium oxide." Nevertheless, " as a rule, with the presence of silicic acid, the percentage of cerium oxide in proportion to the thorium oxide, in order to obtain the most favourable illuminating effect, must not be appreciably greater than is the case with mantles or the like which are composed solely of thorium oxide and cerium oxide." The first claim is for " a mantle composed of thorium and cerium oxide characterised by a percentage of silicic acid."—H. B.

Benzene or other Products contained in certain Gases, Triple-Action Apparatus for the Recovery of. G. C. Marks, London. From " La Compagnie pour la Fabrication des Compteurs et Matériels d'Usines à Gaz," Paris, France. Eng. Pat. 10,577, April 28, 1897.

See under III., *page* 728.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Wood, Modern Distillation of, for the Production of Acetic Acid, Wood Spirit, and Acetone in a Pure Form. M. Klar. Chem. Ind. 1897, **20**, [9] and [10], 191—198 and 219—224 (see this Journal, 1897, 667—671).

With regard to the yields of products resulting from the destructive distillation of wood on a large scale, the results published by Sartig in a comparatively recent communication (this Journal, 1894, 389) may be taken as representing approximately reliable data.

A modern plant for the carbonisation of wood is illustrated; it is worked with eight horizontal retorts, capable of charring 8,000 Rm.* of wood per annum (395·9 kilos. = 1 Rm.), no provision being, however, made for the utilisation of residual products.

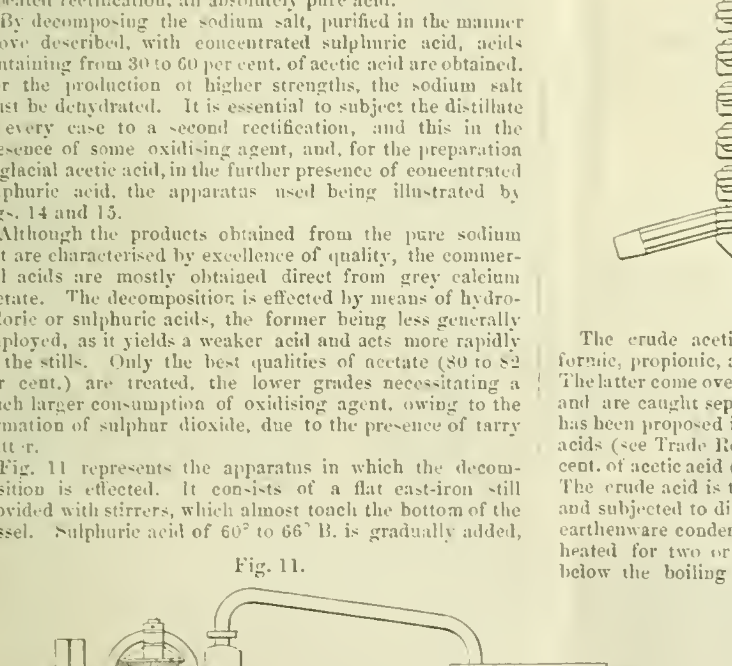
Preparation of Commercial Acetic Acid in a Pure Form.—Grey calcium acetate contains as impurities, tarry constituents, and more especially homologues of acetic acid, the removal of which is accompanied with difficulty. For the purification on a large scale, one of the following two methods is adopted:—(1.) Conversion of crude acetic acid or calcium acetate into the sodium salt, purification of the latter by fractional crystallisation, filtration through charcoal, and decomposition of the higher fatty acids still present in the product, by fusion. (2.) In the second method calcium acetate is treated with strong mineral acids, and the resulting crude acetic acid subjected to fractional distillation in column stills. Since the higher homologous acids come over in the early stages of the distillation along with the more dilute products, the middle fractions are collected separately, and from them it is possible to obtain, by repeated rectification, an absolutely pure acid.

By decomposing the sodium salt, purified in the manner above described, with concentrated sulphuric acid, acids containing from 30 to 60 per cent. of acetic acid are obtained. For the production of higher strengths, the sodium salt must be dehydrated. It is essential to subject the distillate in every case to a second rectification, and this in the presence of some oxidising agent, and, for the preparation of glacial acetic acid, in the further presence of concentrated sulphuric acid, the apparatus used being illustrated by Figs. 14 and 15.

Although the products obtained from the pure sodium salt are characterised by excellence of quality, the commercial acids are mostly obtained direct from grey calcium acetate. The decomposition is effected by means of hydrochloric or sulphuric acids, the former being less generally employed, as it yields a weaker acid and acts more rapidly on the stills. Only the best qualities of acetate (80 to 82 per cent.) are treated, the lower grades necessitating a much larger consumption of oxidising agent, owing to the formation of sulphur dioxide, due to the presence of tarry matter.

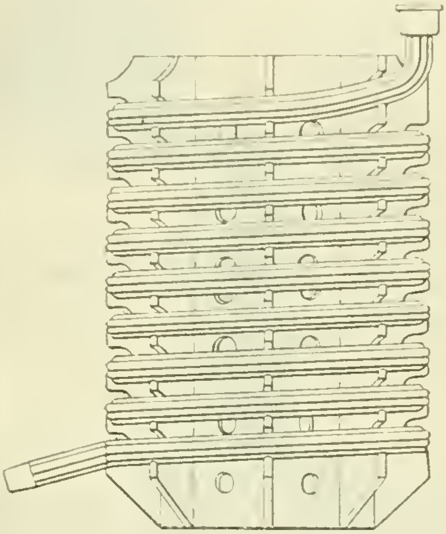
Fig. 11 represents the apparatus in which the decomposition is effected. It consists of a flat cast-iron still provided with stirrers, which almost touch the bottom of the vessel. Sulphuric acid of 60° to 66° B. is gradually added,

Fig. 11.



the heating being effected by a direct fire, which should not be hot enough to give rise to the formation of acetone. Occasionally superheated steam is used, or the still is provided with a steam jacket and the distillation performed in a partial vacuum, a washing apparatus (containing alkali) being placed between the condenser and the exhauster to absorb acid, which may be carried over mechanically. The resulting acid is then free from sulphurous anhydride, and sufficiently pure for commercial purposes without further rectification. A copper pipe, to which a mud or dust separator is attached, is fixed into the top of the still, and carries the vapours to a stoneware condenser immersed in a wooden tank. The construction of the condenser is shown in Fig. 12.

Fig. 12.



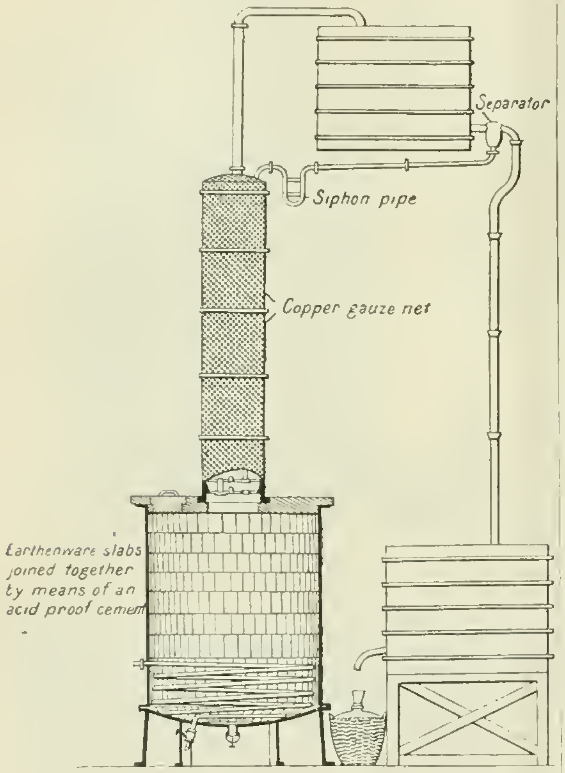
The crude acetic acid contains sulphurous anhydride, formic, propionic, and butyric acids, and empyreumatic oils. The latter come over at the commencement of the distillation, and are caught separately as an impure acid distillate. It has been proposed in France to use this liquid for denaturing acids (see Trade Rep., page 774) containing less than 10 per cent. of acetic acid (known as "acetic acid mauvais goût"). The crude acid is treated with "acetate" in small quantity, and subjected to distillation in a copper still provided with earthenware condenser, the contents being in the first place heated for two or three hours at a temperature slightly below the boiling point of acetic acid (120°). Thus the impurities are removed to a great extent, although the distilled product is not sufficiently pure to pass the permanganate test. It is transferred to a still provided with a column apparatus, and oxidised therein at a temperature of 50° by the addition of chromic acid mixture or potassium permanganate.

In working up brown calcium acetate, the decomposition takes place in earthenware vessels, in which earthenware screens are suspended. Hydrochloric acid of 20° B. is used, the acetate being added gradually. The tarry and pitchy constituents remain on the screens, whilst the solution is introduced into a cast-iron still, which is heated by a direct fire. The still is surmounted by a tower containing a series of screens, the intervening spaces being filled with lumps of manganese dioxide. The acid vapours rising in the tower carry traces of hydrochloric acid into the partitions, whereby chlorine is liberated, the result being the production of a perfectly colourless acid. The final traces of acetic acid are expelled by the introduction of steam into the still. The acid is then oxidised and fractionated in a column still similar in construction to that used for the rectification of wood spirit. The columns are constructed of copper lined with porcelain, or entirely of stoneware. The

* Rm. = Raum-metre, and refers to 1 cb. metre of wood in "Scheitern," i.e., rough pieces, each 1 m. long, piled as many as possible into a frame measuring 1 m. × 1 m. Roughly, 1 Rm. contains 0·75 cb. m. of solid wood.

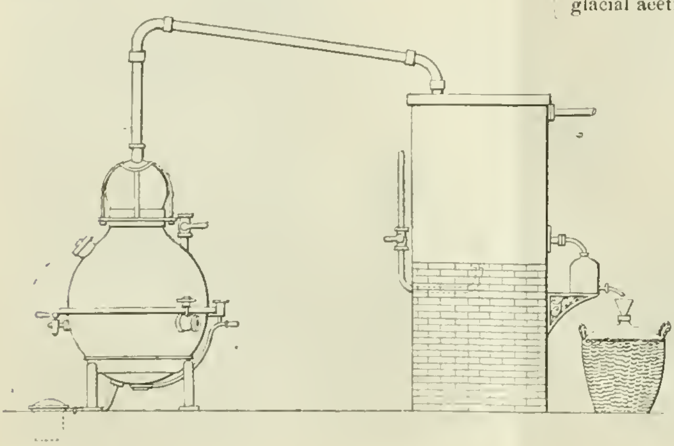
stills are iron or copper, lined with stone slabs or enamelled, and are heated with steam coils or jackets. The vapours issuing from the column pass into the condensing worm, thence into the separator, to which a siphon pipe communicating with the column is attached, and are finally condensed in the cooler, the distillate being collected in carboys. The apparatus illustrated in Fig. 13 consists of an iron still,

Fig. 13.



lined with earthenware plates and joined together with a cement capable of resisting the action of acetic acid. The column is made up of a number of screen-like, perforated earthenware compartments with overflow cylindrical tubes. In some cases the partitions are filled with earthenware "cones," which have been recently introduced for rectifying and condensing purposes. They are easily cleaned, and are said to carry out the principle of dephlegmation more completely than any other appliance. To protect the workers from the possibility of the sudden bursting of the column, it is surrounded by a copper-wire netting or provided with a metal screen placed loosely over the column, the intervening space being packed tightly with a clay material, which, upon the appearance of a rent, fixes itself firmly to the wall of the column and prevents the escape of gases or liquids. The condenser is a stoneware coil contained in a wooden tank.

Fig. 14.



On subjecting the crude acid from grey calcium acetate to rectification, the first fraction, which is a comparatively small one, consists of weak acetic acid, contaminated with other acids. It is introduced into commerce in strengths varying from 30 to 60 per cent. The next fractions, forming the main portion of the distillate, are perfectly pure, and are made up into acids containing from 80 to 100 per cent. of acetic acid. If necessary, the rectification is repeated, the strongest fractions being collected separately and utilised for the preparation of glacial acetic acid. The crude acid from brown calcium acetate yields on rectification a large proportion of weak acid, containing from 30 to 50 per cent., and only a comparatively small amount of concentrated acid. Although the strong acid (once or twice distilled) is quite pure and passes the requirements of the permanganate test, it lacks in so-called "bon goût," and is not suitable for culinary or medicinal purposes. To give it this quality, the respective fractions (80, 95, 96, to 100 per cent.) are distilled in a special apparatus, the construction of which is illustrated in Figs. 14 and 15. It consists of a copper still heated by a fire or a steam jacket. The still head, the pipe leading to the condenser, and the worm are composed of silver or of earthenware, glass, or porcelain, unacted upon by acetic acid. Although silver is the most durable material, earthenware protected with copper gauze is now largely employed. The distillation is sometimes carried on in the presence of concentrated sulphuric acid. Acids sold as 100 per cent. rarely contain more than 99 per cent., owing to the difficulty of preparing chemically pure acid in large quantities. A very pure acid may also be obtained by cooling the acid until it crystallises, removing the liquid portions, melting the crystals, and repeating this operation.

According to Sandmann (this Journal, 1883, 246), glacial acetic acid is obtained from the crude acid in one operation by subjecting it to distillation and allowing the vapour to pass into a second still, where it is separated into strong acetic acid, which is condensed, and weaker acid, which distils over.

Rohrmann proposes to concentrate and purify crude acetic acid by utilising the plate towers designed by Lunge and Rohrmann (this Journal, 1887, 584). The vapours rising within the column (maintained at a temperature of 120°) come into intimate contact with a constantly descending stream of concentrated sulphuric acid, to which eventually chromic acid is added, whereby, after one passage, the strength of the acid is said to be raised from 40 to 70 per cent. Heinzerling obtains acetic acid from acetates by heating with magnesium chloride.

Johnson-Johnson (Stratford) proposes to purify the crude acid by repeated oxidation and rectification over acetate, glacial acetic acid being obtained by distilling with concentrated sulphuric acid.

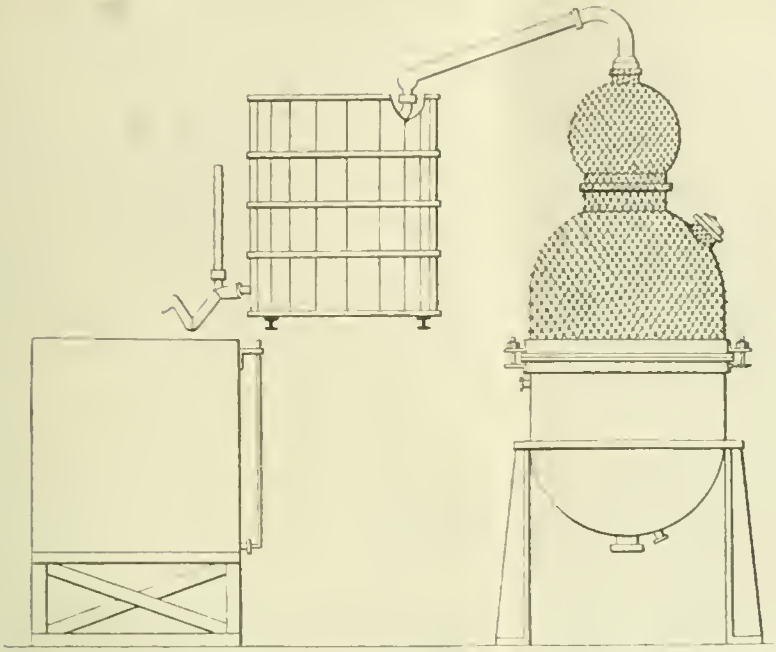
Marco Michela prepares acetic acid from calcium acetate by distilling it with sodium bisulphate in a partial vacuum.

J. E. Johnson recommends the decomposition of calcium acetate with sulphur dioxide, separating the acid from the calcium bisulphite, recovering the sulphur dioxide from the latter, and purifying the crude acid in the usual manner.

The working of an acetic acid plant capable of treating 2,000 kilos. of calcium acetate in 24 hours is illustrated in plan and elevation, but without dimensions.

Rectification of Crude Wood Spirit, 80° Tr.—The raw material forming the source of rectified spirit, methylated spirit, and pure methyl alcohol, is largely imported into Germany, mainly from America and Austro-Hungary. It is a yellowish to brownish liquid,

Fig. 15.



has an empyreumatic odour, gives a turbidity with water differing in intensity with its purity, and contains as much as 12 per cent. of acetone. The following table gives the properties of a number of samples of wood spirit found in commerce. The acetone was determined according to Messinger's method (this Journal, 1896, 299), and the capacity for absorbing bromine according to the method prescribed by the German Excise for testing denaturing spirit :—

Name.	Colour.	Miscibility with Water.	Tralles Degrees.	Acetone Per Cent.	C.c. of Spirit required to Decolorise 100 c.c. of Potassium Bromide and Bromate Solution.
1. Crude wood spirit from Silesia	Yellow.	Very turbid.	80	6·44	50·00
2. Crude wood spirit (?)	Yellow.	Turbid.	80	10·00	15·90
3. Rectified wood spirit (?)	White.	Clear.	94	5·60	79·30
4. Rectified wood spirit from the Hartz....	White.	Clear.	92—95	37·46	14·75
5. Crude wood spirit from the Hartz	Yellow.	Clear.	85	20·94	51·20
6. Crude wood spirit (American)	Yellowish-brown.	Clear.	80	11·00	65·00
7. Rectified wood spirit ("A. xylolyse") ...	Almost white.	Clear.	98—99	4·80	52·00
8. Rectified wood spirit ("B. xylolyse") ...	Yellowish.	Clear.	95	13·72	19·40
9. Rectified wood spirit (Westphalian)	White.	Clear.	93—94	4·50	70·00

The first operation consists in converting the crude spirit into white rectified spirit by subjecting it to fractional distillation in a column still (see diagrams Nos. 8 to 10, this Journal, 1897, 670) in the presence of milk of lime. The first runnings are highly charged with acetone; the middle fraction gives a clear solution with water, shows 99 to 99·5° Tr., and contains only a comparatively small amount of acetone. The last fractions give a turbidity with water and high iodine numbers, due to the presence of ketones, which react with iodoform like acetone. The miscibility with water decreases with the reduction in the percentage of alcohol, until finally wood oils come over. The following fractions were obtained by the rectification of American and Silesian wood spirit, 80° Tr. :—

1. *American Wood Spirit.*—894 kilos. gave—
   - 50 kilos. of first runnings of 96° Tr. containing 57·72 per cent. of acetone.
   - 491 kilos. of rectified white wood spirit of 99°—99·5° Tr., miscible with water (1 : 2) to a clear solution.
   - 53 kilos. of rectified white wood spirit of 97° Tr., forming a turbidity with water.
   - 47·5 kilos. of rectified white wood spirit of 96° Tr., forming a turbidity with water.
   - 35 kilos. of wood spirit of 64° Tr.
   - 50 kilos. of water of 9° Tr.
   - 48 kilos. of water of 2° Tr.
   - Wood oils.
2. *Silesian Wood Spirit.*—489 kilos. gave—
   - 49 kilos. of first runnings of 91° Tr., containing 23 per cent. of acetone.
   - 226 kilos. of white rectified wood spirit of 99—99·5 Tr. Clear solution with water.
   - 25 kilos. of white rectified wood spirit of 99° Tr., forming a turbidity with water.
   - 51·4 kilos. of white rectified wood spirit of 97° Tr., forming a turbidity with water.
   - 29 kilos. of wood spirit of 69° Tr.
   - 65·5 kilos. of water of 5° Tr.
   - 64·5 kilos. of water of 3° Tr.
   - Wood oils.

For the preparation of wood spirit for denaturing purposes, the first and last runnings containing much acetone, are redistilled, mainly with a view of obtaining a colourless product. This is then tested for specific gravity, percentage of acetone, and bromine absorption. The quantities of acetone and empyreuma are mostly below the excise standard, and are adjusted by the addition of pure acetone and last runnings. The mixture is then brought to a strength of 91° Tr., when it will be found to give the boiling points required by the German Excise. The following are the official tests framed in June 1888 and July 1896 for wood spirit used for the denaturing of alcohol :—

1. *Colour.*—The colour must not be darker than that obtained by dissolving 2 c.c. of a decinormal solution of iodine in a litre of distilled water.

2. *Boiling Point.*—100 c.c. of wood spirit are distilled in a metal flask provided at the top with a small globe, which is connected with a Liebig condenser. An officially standardised thermometer is fitted in the globe, and the flask is then moderately heated, so that the distillation proceeds drop by drop. The distillate is collected in a graduated cylinder, and when the thermometer indicates 75°, the barometric pressure being normal, at least 90 c.c. must

have come over. A correction of 1° in temperature is made for every variation of 30 mm.

3. *Miscibility with Water.*—20 c.c. of spirit mixed with 40 c.c. of water must give a clear or only slightly opalescent solution.

4. *Separation with Sodium Hydroxide.*—If 20 c.c. of the spirit be shaken with 40 c.c. of sodium hydroxide solution (sp. gr. = 1·3), at least 5 c.c. of the spirit should be separated after standing for half an hour.

5. *Percentage of Acetone.*—1 c.c. of a mixture containing 10 c.c. of spirit and 90 c.c. of water is introduced into a glass-stoppered cylinder and well mixed with 10 c.c. of sodium hydroxide solution (80 grms. of NaHO per litre); 5 c.c. of double normal iodine solution (254 grms. of iodine per litre) are then added, the mixture being again well shaken. The separated iodoform is taken up with 10 c.c. of ether (sp. gr. 0·722); 5 c.c. of the ethereal solution are then transferred with a pipette to a tared watch glass, evaporated to dryness, and weighed, after standing over sulphuric acid for two hours. The increase of weight should not amount to less than 0·07 grm.

6. *Capacity for absorbing Bromine.*—100 c.c. of a solution of potassium bromate and bromide, prepared in the manner described below, are treated with 20 c.c. of dilute sulphuric acid (1 : 3 of water). To this mixture, which represents a solution containing 0·703 grm. of bromine, wood spirit is added from a burette divided into 0·1 c.c., drop by drop, shaking all the time, until the colour of the bromine has disappeared. The addition of the spirit is regulated, so that not more than 10 c.c. are added per minute. Not more than 30 c.c. and not less than 20 c.c. should be required. The tests should be made in full daylight and the temperature should not exceed 20°. To prepare the bromine solution, pure potassium bromate and bromide are dried for 2 hours at 100° and allowed to cool in a desiccator. 2·447 grms. of the bromate and 8·719 grms. of the bromide are then dissolved in 1 litre of water.

The middle fractions obtained by the rectification of the crude naphtha are made up to certain strengths and brought into commerce as white rectified naphtha of the following qualities:—

90 per cent. (French spirit).
92—93 per cent. (English spirit).
95—96 per cent.
99—99·5 per cent. (pure spirit).

Although this product is "water-white" when first prepared, it gradually assumes a yellow colour, and gives a brownish to blackish coloration with concentrated sulphuric acid, due to the presence of empyreuma. It is purified by treatment with weak oxidising agents or by filtration through charcoal. In order to remove traces of ammonia and amines, the filtrate is treated with a small quantity of sulphuric acid and subjected to fractional distillation in a column still. In this manner a very pure product is obtained. The first and last runnings are collected separately and worked up into spirit for denaturing purposes.

For the valuation of the methyl alcohol prepared as above, it is converted into methyl iodide; this is distilled off and estimated by volume. (See Krell, Berichte, **6**, 1310. Krämer and Grotzky, Berichte, **7**, 1492, and **9**, 1928. Bardy and Bordet, Dingler's Polyt. J. **233**, 245.)

Preparation of Acetone.—This product, which is largely used in the manufacture of smokeless powder and the preparation of celluloid goods, is obtained by the destructive distillation of calcium acetate (80—82 per cent.). The operation is effected in the apparatus illustrated in Fig. 11. The construction of the mixer is of importance, as unless it is capable of thoroughly stirring the mass during the whole operation, the yield of acetone is greatly diminished. The best results are obtained by maintaining a dull red heat (500°—600°), care being taken that the pipes, which convey the gaseous products to the tar separator and condenser, are of sufficient width to prevent stoppages. They should, moreover, be constructed so that they can be easily and rapidly removed and replaced. Having heated the still to the requisite temperature, the mixer is started and finely ground calcium acetate (as dry as possible) introduced through the manhole. The distillation is then proceeded with, which with a charge of 100 kilos. should occupy from two to three hours. The first distillate is water, originating from the moisture in the acetate. It is more or less coloured, has an empyreumatic odour, and contains a few per cent. of acetone. After a time the flow of water slackens, followed by a stage when hardly anything comes over; and when the temperature has risen high enough, acetone oils distil over. The total distillate forms a dark brown, highly inflammable liquid, having an empyreumatic odour and being contaminated with tarry substances and calcium acetate which has been carried over mechanically. On standing, it separates into two layers. The top layer, comprising about one-tenth of the bulk, contains the so-called "Heavy acetone oils," whilst the bottom layer is chiefly composed of acetone and the lighter acetone oils held in solution by water. The following table gives the quantities obtained from 100 kilos. of grey calcium acetate, the consumption of fuel being 30 to 35 kilos. of coal:—

	Kilos.	Sp. Gr.	Acetone.	Acetone.
			Per Cent.	Kilos.
Acetone water	7—15	1·01	3·0	0·4
Crude acetone	32	0·90	67·5	22·0
Total distillate	42	0·93	53·5	22·5

The crude acetone thus obtained is largely contaminated with acetone oils, which are mainly composed of higher ketones originating from homologues of acetic acid present in calcium acetate; they also contain aldehydes, due to the presence of formic acid, condensation products of acetone, acids, phenols, and tarry substances. To minimise the formation of these secondary products and obtain better yields, Bauschlicher proposes to effect the decomposition in the presence of lime. Although the latter retains the tarry constituents, it does not act on the higher ketones and condensation products; and as these comprise by far the largest proportion of the secondary products, the author questions the utility of the process.

For the preparation of pure acetone, the crude product is diluted with water, treated with milk of lime, and allowed to stand for some time. The supernatant oily layer is then mixed with water and subjected to distillation in a column still. With a well-constructed apparatus the first rectification gives, as main fraction, a very pure product showing 99° to 99·5° Tr. and failing to give a turbidity with water. It contains only traces of aldehyde and empyreuma, and these are entirely removed by another distillation. The following are the commercial tests for English acetone:—(1.) The specific gravity should not exceed 0·802 at 15°, and the acetone should volatilise completely, without leaving a residue, when heated to 100°. (2.) When subjected to distillation, four-fifths of the original quantity taken should distil over at 58·8°, and, besides acetone, the residue should contain no substances other than those originating from the manufacture of acetone. (3.) 100 c.c. of acetone mixed with 1 c.c. of potassium permanganate solution (0·1 per cent.) should show no change of colour within two minutes. (4.) The acetone should not contain more than 0·005 per cent. of acid calculated as acetic acid. For testing this, 50 c.c. of acetone are diluted with 50 c.c. of water, 2 c.c. of a solution of phenolphthalein (1 : 1,000 alcohol of 50 per cent.) are then added, after which the mixture is titrated with sodium hydroxide solution (1 c.c. = 0·0006 grm. of CH_3COOH).

The first and last runnings and the oils recovered during the clarification process with milk of lime are mixed together and redistilled, when a further quantity of suitable acetone is obtained. The residual oils constitute the so-called acetone oils, introduced into commerce as light oils (boiling between 75° and 130°) and heavy oils (boiling between 130° and 250°).

The boiling point of the commercial acetone oils is one lying between wide limits, and may be generally stated as from 75° to 220° C. The following results of a laboratory fractionation of 600 grms. of an acetone oil demonstrate

this, and also the proportions distilling at the various intervals of temperature.

91·5 grms.	at	72°–85°	light yellow.
97·5	"	85 –95	"
116·5	"	95 –115	yellow.
81·0	"	115 –130	"
66·5	"	130 –145	"
34·0	"	145 –160	"
23·0	"	160 –175	dark yellow.
22·0	"	175 –190	brown.
24·0	"	190°–205	"
24·5	"	205 –220	"
19·0	"	above 220	black residue.
599·5			

—D. B.

Natural Gas and Petroleum, Genesis of. F. C. Phillips. Proc. Amer. Philos. Soc. **36**, [154], 116—121.

CRITICISING the Mendéleeff hypothesis, the author points out that Moissan has shown that few of the metals form definite carbides, and that as these are metals of low specific gravity, their presence must—if they are regarded as producers of natural gas—be assumed at depths where heavy metals alone are believed to occur. Moreover, aluminium, oxidising, as it does, more readily than iron, would, in presence of this latter metal, precede it in order of oxidation, and be thereby rendered incapable of producing hydrocarbons. A boring driven to a depth of 2,900 feet in Ontario, and extending to within a short distance of the Archæan rock, yielded gas from deep strata, underlying 1,600 feet of shale, which gas contained no hydrogen, acetylene, or carbon monoxide: and similar negative results have been given by Pennsylvania gas. Now, iron carbide according to Mendéleeff's theory, the active agent in the formation of gas, yields mainly free hydrogen; and although, if we regard the gas as a stored product, this constituent may, in the course of long ages, have escaped by diffusion, this could not occur so rapidly if the gas be a contemporaneous product, but a sufficient quantity to be estimated by chemical means would still be present in the gas issuing from the borehole.

Therefore, no theory of the origin of natural gas that assumes the production of free hydrogen. can be regarded as satisfactory until the presence of that constituent in the effluent gas can be demonstrated. The composition of the gas suggests that the formative reactions occured at low temperatures, and that it has been stored below 500° C., since above that temperature, the constituents are more or less completely dissociated. Petroleum, on the other hand, contains hydrocarbons pointing to high-temperature reactions—a circumstance that should be considered in selecting any hypothesis as to its origin.—C. S.

Petroleum (Pennsylvania), Suggestion as to the Origin of. D. T. Day. Proc. Amer. Philos. Soc. **36**, [154], 112—115.

THE theory advanced by MacGonigle ascribes the formation of the oil deposits in the Trenton and Clinton limestones to the action of the heat that, at the close of the carboniferous period, caused the uplift forming the Appalachian chain, which heat, passing through the Silurian limestones and shales, distilled the oil in the latter into the porous conglomerates of the Devonian age. The reasons advanced in support of this theory are, the absence of sulphur, the higher specific gravity, and the variations in colour in Pennsylvania oil, whereas the Silurian oil of Ohio exhibits converse peculiarities. Moreover, no oil has been tapped in Pennsylvania in the "corniferous" limestone, lying at the top of the Silurian period, though in Western Canada this formation is the chief oil rock; and it is also very doubtful (no remains having been discovered) whether any organic life existed in the Pennsylvania (Devonian) measures to form a source of petroleum. Another point adduced is that the profitable oil wells in this region all lie to the west of a line drawn from Brady's Bend to Waynesborough, whilst to the east of the line are found some of the richest gas pools, thus indicating that as the line of greatest upheaval (and therefore heat) is approached the distilled oil from the Silurian horizons remained in an uncondensed gaseous condition.

The author favours the above hypothesis, but considers the transference may have occurred without distillation, the existence of cracks in the cover over the Silurian limestones enabling the oil to leak through, and that filtration would account for the differences in colour, the oil passing through first being the lightest in shade. As to how the removal of sulphur has been accomplished, this is more difficult to explain, though the action of aluminium chloride in this direction indicates a metallic chloride as the agency.

—C. S.

Petroleum, Nature and Origin of. S. F. Peckham. Proc. Amer. Philos. Soc. **36**, [154], 103—112.

IN discussing the Engler-Hofer theory of origin, the author expresses a belief that "steam has been a very potent factor in determining, not only the formation, but the transference of bitumens."

Texan turrellite he finds to consist of a white shell rock cemented by lime dissolved from the shells by hot water and then re deposited, anterior to the introduction of the impregnating bitumen, and not composed of shells cemented together by bitumen, as generally asserted.

Esters of the pyridine bases have been discovered in the lighter fractions of Californian petroleum. Such petroleums on distillation decomposed even at 100° C. with evolution of H_2S, more or less separation of carbon, and rise of boiling point of the residue in the retort. Sulphur compounds have also been found by the author in Californian petroleum, and he considers that sulphur, as well as nitrogen, contributes to the conversion of petroleum, through "maltha," into asphaltum, the prime factors being polymerisation, decomposition of the sulphur compounds with condensation of the residual molecule, and removal of hydrogen through deoxidation of the sulphates in the accompanying water, with substitution of sulphur in the oil. The fact that Californian petroleum is found to lose up to 25 per cent. by volume in four days at between about 70° and 90°, shows that at the surface, natural evaporation may also play a considerable part in this conversion.

In the large asphaltum deposit on the eastern side of the coast range (California), enormous springs of maltha have flooded the valleys in many places with large plains of asphaltum, the maltha being invariably accompanied by water, with many indications of the springs having at one time been hot and silicated, like those believed by the author to have produced the Trinidad pitch lake.

He thinks that no formula concerning the origin of petroleum and bitumen is capable of universal application but that in some places these bodies have been produced *in situ*, from animal remains; that in other instances the deposits contain primary distillates from older geological formations; and that in some cases bitumens "have been transferred and stored by a secondary invasion of bituminous deposits, by heat, steam, and pressure."—C. S.

Petroleum and Natural Gas, Genesis and Chemical Relations of. S. P. Sadtler. Proc. Amer. Philos. Soc. **36**, [154], 93—102.

PASSING in review the different theories of origin put forward by various workers, the author refers to his own experience in the production of fluorescent "paraffine" oils, paraffin scale, and reduced petroleum oil or residuum, by redistilling the condensed vapours from linseed oil boiled under pressure, and he therefore considers that Engler's theory will have to be enlarged so as to include the vegetable seed oils as probable sources of petroleum.

—C. S.

Petroleum (Pennsylvania, Ohio, and Canada), Constituents of, between 150° and 220°. C. F. Mabery. Amer. Chem. J. **19**, [6], 419—482.

PENNSYLVANIA oil consists, between these limits, of decane (163°—164°), decane—probably normal decane (173—174°), hendecane (196°—197°), and dodecane (214—216°), along with smaller quantities of aromatic hydrocarbons (mesitylene, &c.) of similar boiling points. Ohio (Trenton limestone) oil is represented by the same hydrocarbons of the C_nH_{2n+2} series, the higher specific gravity being due to a larger percentage of the aromatic series.

Up to 173° the Canadian ("corniferous" limestone) oil is the same as the others, but the fractions 196°—214° have the formula C_nH_{2n}. Canadian oil contains a still larger proportion of aromatic hydrocarbons than the other two, and the higher fractions of all three probably contain other bodies, for the identification of which large quantities of distillate are required.

These results show a relation between the chemical composition of the hydrocarbons and the density of the crude oils, *viz.*, Pennsylvania, 0·80—0·82; Ohio, 0·82—0·85; Canadian, 0·85—0·88; whilst South American oil, sp. gr. 0·948, contains only the C_nH_{2n} series, which also forms the chief portion of Caucasian oil, sp. gr. 0·88.

The precise action of sulphuric acid on petroleum distillates is still undefined, but the author considers that one of its chief functions consists in the removal of unsaturated hydrocarbons. Oxygen compounds appear to be mainly restricted to the higher fractions of American oil, though all the unpurified distillates examined, gave, in presence of sodium, flocculent precipitates of decomposition products, due to nitrogen or (more probably) oxygen compounds in small amount.—C. S.

PATENTS.

Petroleum, Heavy Oils, Tar Oils, and Hydrocarbons generally; Improved Apparatus for Vaporising. L. E. Fraipont, Paris. Eng. Pat. 19,861, Sept. 8, 1896.

In this apparatus the oil is fed into a retort, where it is distilled, the vapours then passing into a second similar retort, heated independently, wherein the lightest vapours are gasified, and thence to a gas producer—consisting of three tubes connected by ports—where the conversion into gas is completed. A portion of the gas may be utilised for heating the apparatus, and arrangements are made for discharging the residue from the retorts by the pressure of the internal gases, thus preventing the formation of coke. —C. S.

Benzene or other Products contained in certain Gases, Triple-Action Apparatus for the Recovery of. G. C. Marks, London. From "La Compagnie pour la Fabrication des Compteurs et Matériels d'Usines à Gaz," Paris, France. Eng. Pat. 10,577, April 28, 1897.

This invention relates to apparatus for the recovery of benzene from furnace gases or coke ovens, and it is also applicable for the extraction of any products contained in gas capable of solution in suitable liquids. It consists of an arrangement of chambers in communication with one another, containing revolving drums, the chambers being partly filled with a suitable absorbent or solvent liquid. The gas is caused to be dispersed throughout the absorbent so as to present a large surface for attack, whilst it pursues a tortuous course through each drum and chamber alternately. A triple action of the solvent liquid or the gas under treatment is secured by the passage of the gas through the revolving drum and buckets, the gas ascending in the shape of bubbles, producing, in the first place, the appearance of a boiling or convection of a very intense mechanical nature, which is augmented by a liquid "cascade or rain," produced by a revolving system of buckets. Thus, there is presented in the second place, a large surface to the upward current of gas. Thirdly, the oil flows like a cascade, through the action of a perforated sheet-iron spiral, and is thus enabled to act with increased advantage upon the upward current of the gas, so as to cause further absorption of benzene from it.—D. B.

IV.—COLOURING MATTERS AND DYES.

a-Monochlorcamphor, An Oxidising Action of. H. Vittenet. Bull. Soc. Chim. 1897, **17**, 705—712.

On heating aromatic amines (at 200° for four hours) with α-monochlorcamphor, the author has observed the formation, in very small quantities, of certain colouring matters generally obtained by oxidation of the amines. For example, with *o*-toluidine, or a mixture of *m*-xylidine and aniline, magenta was formed, and with diphenylamine, there was produced Diphenylamine Blue.—A. C. W.

The Sacchareïns: New Colouring Matters derived from Saccharine. P. Monnet and J. Kœtschet. Bull. Soc. Chim. 1897, **17**, 690—702.

Benzoic sulphimide under certain conditions reacts with phenols and amidophenols to give compounds analogous to the phthaleïns—

$$C_6H_4\left\langle\begin{matrix}CO\\SO_2\end{matrix}\right\rangle NH + 2C_6H_5.OH =$$

$$H_2O + C_6H_4\left\langle\begin{matrix}C{=}(C_6H_4.OH)_2\\SO_2\end{matrix}\right\rangle NH$$

These substances, the sacchareïns, have, in general, properties similar to those of the phthaleïns. Thus, phenol sacchareïn dissolves in alkalis to a red solution decolorised by acids; resorcinol sacchareïn gives a yellow solution with a green fluorescence similar to that of fluoresceïn. Whilst fluoresceïn can take up two acid groups, resorcinol sacchareïn combines with three molecules of acetic acid, to give a triacetylated compound.

The sacchareïns of the substituted *m*-amidophenols are quite analogous to the rhodamines in colouring and general properties, but since the hydrogen in the group $SO_2.NH$ may be replaced by acid or alcohol radicals, a large number of derivatives may be obtained. These sacchareïns, however, differ from the rhodamines in that their coloured salts are instantly decolorised by alkalis, even by dilute ammonia in the cold. But if the sacchareïn be transformed into the corresponding sulphureïn, or if an acid radical be introduced in the imido group, the colouring matters obtained are among the most stable, towards alkalis.

Resorcinol Sacchareïn. — Heat 18 grms. of saccharine for seven hours at 200°—220° with 22 grms. of resorcinol and 2·2 grms. of aluminium chloride. When cold, powder, extract with dilute sodium carbonate solution, precipitate the colouring matter with acid, take up with alcohol, and precipitate by adding water. A brown resin is obtained with a green metallic reflection, soluble in alkalis to a yellow solution, insoluble in water but very soluble in alcohol. It could not be crystallised.

The acetyl derivative is formed by boiling with acetic anhydride under a reflux condenser, cooling, and pouring into alcohol. The crystalline precipitate is purified by solution in glacial acetic acid and precipitation by alcohol; it melts at 286°, and is insoluble in water. By boiling this acetyl derivative with alcoholic soda, diluting, filtering, and precipitating with water, the sacchareïn is obtained in crystals, melting at 265°—267°.

Tetra-ethyl-m-amidophenol Sacchareïn.—Heat 100 grms. of the amidophenol for 36 hours with 200 grms. of saccharine at 165°, powder the product, suspend in sodium carbonate solution, boil until carbon dioxide is no longer evolved, cool, and filter. The excess of saccharine may be obtained from the solution. The insoluble product is digested for several hours with caustic soda, filtered, washed, dissolved in dilute hydrochloric acid, and filtered. On cooling, the hydrochloride crystallises in small green crystals with a metallic lustre. These are very little soluble in water, but impart to it an intense reddish-violet colour with a yellow fluorescence.—A. C. W.

α-Naphthol, Sulphonic Acids of. P. Friedländer and R. Taussig. Ber. **30**, 1456—1463.

The authors characterise and give methods of preparation of a number of the sulphonic acids of α-naphthol, and find that in several cases the zinc salt can be employed as a means of purification. The action of sulphuric acid on α-naphthol yields sulphonic acids in which the entering group has the position 2, 4, or 2′, whilst 1.3′-naphthol sulphonic acid is only formed in small quantity. The same is also the case with 1.2-naphthol carboxylic acid, of which the 4.3′ and 4.2′-sulphonic acids easily separate carbonic acid, and naphthol carbonate also yields chiefly 4 and 4.2′ derivatives, with but little 4.3′. For the preparation of other sulphonic acids, indirect methods have to be resorted to, such as the substitution of the amido group by hydroxyl, by decomposition of the diazo compound or the replace-

ment of a sulphonic acid group in a polysulphonic acid by fusion with caustic soda. Moreover, sulphonic acid groups in the α position can be eliminated from α-naphthol polysulphonic acids by treatment with sodium amalgam.

1.2-*Naphthol Sulphonic Acid.*—Equal weights of sulphuric acid and of finely powdered α-naphthol are heated for a short time to 60—70° C. until dissolved. The melt, after cooling, is dissolved in water and extracted with benzene; when about 16 per cent. of the naphthol is recovered. A concentrated solution of potassium chloride precipitates the potassium salt of the acid as a sandy powder easily soluble in hot water, the yield (76—80 grms. from 100 grms. of α-naphthol) being about 50 per cent. of the theoretical amount. This appears to be the only sulphonic acid of α-naphthol which is sparingly soluble in dilute mineral acids.

1.3-*Naphthol Sulphonic Acid* can be prepared by diazotising and boiling 1.3-naphthylamine sulphonic acid (Ber. **28**, 1951), or by eliminating the amido group from γ-amidonaphthol sulphonic acid (Ger. Pat. 57,910). It is purified by means of its zinc salt, which contains 8·5 H_2O. As Gattermann has pointed out (Ber. **30**, 51; this Journal, 1897, 232), the acid gives *o*-azo dyestuffs with diazo compounds.

1.4-*Naphthol Sulphonic Acid.*—The crude product was obtained by heating equal weights of sodium naphthionate and 50 per cent. caustic soda lye under pressure to 240°—260° C. (Ger. Pat. 46,307), and purified by adding a small quantity of zinc chloride to a hot concentrated solution of the sodium salt, filtering, and precipitating with an excess of syrupy zinc chloride solution. The same acid can also be obtained by careful sulphonation of α-naphthol with about an equal weight of sulphuric acid and 0·5 part of glacial acetic acid.

1.4′-*Naphthol Sulphonic Acid* is obtained by boiling diazotised 1.4′-naphthylamine sulphonic acid with dilute sulphuric acid (1:4), and is purified by means of the zinc salt. The acid yields 2.1.4′-nitrosonaphthol sulphonic acid with nitrite, and gives with iron salts the characteristic green reaction for *o*-nitrosonaphthols.

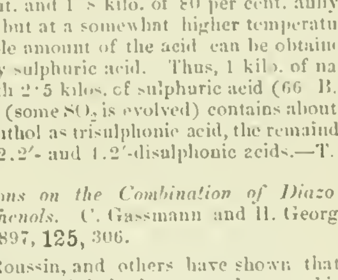
1.2′-*Naphthol Sulphonic Acid* is best prepared from 1.2.4.2′-hydroxy-carboxy-naphthalene disulphonic acid (Ber. **29**, 37). When heated with acids to a high temperature, the carboxyl group 2 and the sulphonic acid group 4 are eliminated. This operation may also be performed in two stages. By prolonged boiling with 3 per cent. hydrochloric acid, the carboxyl group is removed, 1.4.2′-naphthol disulphonic acid being formed: whilst by heating for a short time to about 140° C. with tolerably dilute sulphuric acid, the sulphonic acid group 4 is split off, yielding 1.2.2′-hydroxy-carboxy-naphthalene sulphonic acid. A better method is to dissolve hydroxy-naphthoic-disulphonic acid in hot sulphuric acid boiling at 160° C., and heat for a short time to 140—145° C. The hydroxy-naphthoic-sulphonic acid then separates out, and is purified by dissolving in water and precipitating with concentrated hydrochloric acid. The acid (or its sodium salt) has the characteristic property of separating as a gelatinous mass from a hot aqueous solution. The conversion into 1.2′-naphthol sulphonic acid is best effected by heating under pressure with a little water to 120° C. This acid gives, with diazo compounds, azo dyestuffs unstable to acids. With *p*-nitrodiazobenzene in acetic acid solution, a yellowish-red dyestuff is obtained which turns violet-blue with alkalis, whilst in an alkaline solution a disazo compound is produced which is brownish-red with acids and bluish-violet with alkalis. It is noteworthy that the fastness to alkalis of the sulphonated naphthol azo dyestuffs depends not only on the *o*-position of the azo group, but also on the nature of the second component. Thus, almost all the azo derivatives of *p*-nitraniline (including those of the β-naphthol sulphonic acids and of 1.4′-naphthol sulphonic acid) are red when acid, and violet when alkaline.

1.3.3′-*Naphthol Disulphonic Acid*—This acid was obtained by Gürke and Rudolph (Ger. Pat. 38,281) by fusing 1.3.3′-naphthalene trisulphonic acid with caustic soda under pressure at 170°—180° C., but its constitution has not hitherto been determined. The same acid is formed by heating α-naphthylamine disulphonic acid with three times its weight of water to 180° C. Both acids give with diazo compounds, dyestuffs fast to acids, and on reduction give a solution which, when made alkaline, turns blue on exposure to the air (test for 2.1-amidonapthol sulphonic acids); and the complete identity of the acids formed by the two methods was proved by heating them with ammonia and sal ammoniac to 160—180° C., when 1.3.3′-naphthylene diamine sulphonic acid was obtained.

1.2.4.2′-*Naphthol Trisulphonic Acid*, which is employed technically for the manufacture of naphthol yellow S, possesses a characteristic sparingly soluble barium salt, which separates out on evaporating its aqueous solution, and is then no longer readily soluble. The salt is readily soluble in dilute acids and even in acetic acid. Sodium amalgam removes the sulphonic acid group 4, whilst sodium nitrite added to a solution of the barium salt faintly acid with hydrochloric acid gives an immediate precipitate of barium sulphate. The filtrate contains 4.1.2.2′-nitroso-naphthol disulphonic acid, which does not react with iron salts. According to Ger. Pat. 10,785, the trisulphonic acid is obtained by heating 1 kilo of α-naphthol with 2 kilos. of 25 per cent. and 1·8 kilo. of 80 per cent. anhydride to 40—50° C.; but at a somewhat higher temperature, a not inconsiderable amount of the acid can be obtained by means of ordinary sulphuric acid. Thus, 1 kilo. of naphthol, after heating with 2·5 kilos. of sulphuric acid (66° B.) for 4 hours to 125° C. (some SO_2 is evolved) contains about one-seventh of the naphthol as trisulphonic acid, the remainder consisting chiefly of 2.2′- and 1.2′-disulphonic acids.—T. A. L.

Observations on the Combination of Diazo Compounds with Phenols. C. Gassmann and H. George. Comptes Rend. 1897, **125**, 306.

Griess, Roussin, and others have shown that phenols in general and naphthols in particular combine with the alkaline salts of diazo compounds. This reaction is subject to certain conditions. Phenol itself will not combine with normal diazo compounds in presence of sodium acetate, while resorcinol and α-naphthol sulphonic acid ($OH:SO_3H = 1:4$) readily combine with diazo compounds, even in very acid solutions. From their experiments, the authors conclude that all the phenols and naphthols which, on combination with normal diazo compounds, form ortho-oxyazo substances, react either in neutral or acid solution. β-naphthol combines differently according to the solvent. Thus, in the reaction with mono-diazotised phenosafranine, if the β-naphthol is dissolved in acetic acid, a very soluble colour (Diazine Blue, Naphthindone) is formed—



while in alkaline solution a less soluble colour is obtained. 15 per cent. solutions of β-naphthol in triacetyl glycerin can also be used, and they possess the advantage of being very permanent.

Nitrated diazo compounds react with phenols much more readily than do the non-nitrated diazo substances. For example, if a mixture of the chlorides of diazobenzene and *p*-nitrodiazobenzene reacts with β-naphthol in either neutral, acid, or alkaline solution, *p*-nitrobenzene-azo-β-naphthol is almost exclusively formed. This fact is interesting in connection with the manufacture of Anthracene Red (Ger. Pat. 72,867, Fr. Pat. 223,176), which probably has the constitution—

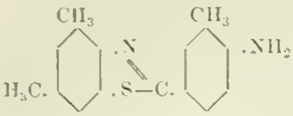
$$O_2N.C_6H_3.N = N.C_6H_3(OH)CO_2H$$
$$C_6H_4.N = N.C_{10}H_5(SO_3H)OH.$$

Ortho mono-nitro-benzidine is diazotised and combined, first with salicylic acid, and then with α-naphthol sulphonic

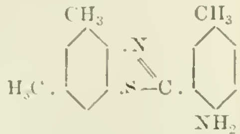
acid ($OH : SO_3H$ = 1 : 4) : if the order of combination be reversed, a colour of no commercial importance is obtained.
—C. M.

Dyestuffs [*Synthetic*]. *Manufacture of. II.* L. Paul.
Zeits. f. angew. Chem. 1896, 679—692.

1. *Dyestuffs from Dehydrothio Bases.*—When *m*-xylidine is heated with sulphur, two thio compounds are produced, having the following formulæ:—



Dehydrothioxylidine.

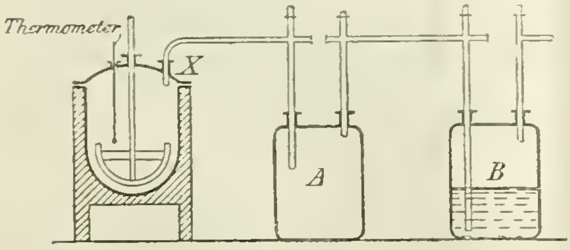


Isothioxylidine.

The former is employed for the manufacture of Erica and Salmon Red, whilst the latter, when combined with β-naphthol sulphonic acid (Schaeffer), yields Emin Red. The two isomers can be separated by means of hydrochloric acid (20° B.), in which isothioxylidine is perfectly insoluble. The dehydrothioxylidine is employed as sulphate in the form of a 28—30 per cent. paste. 40 kilos. of the paste are mixed with 40 kilos. of hydrochloric acid (20° B.), allowed to stand overnight, cooled with 50 kilos. of ice, and diazotised with 10·9 kilos. of a 25 per cent. nitrite solution diluted with 11 litres of cold water. The whole is then diluted to 250 litres and filtered if necessary. For the preparation of Salmon red, the solution so obtained is run into 120 kilos. of a 10 per cent. Amido-R-salt solution, 2.3.3′.$C_{10}H_5.NH_2(SO_3H)_2$ (obtained by the action of ammonia on R-salt), containing 50 kilos. of sodium carbonate, with constant agitation. After stirring three hours, the dyestuff is filtered, washed, pressed, and dried. The yield is 17 kilos., which is diluted for the market with 15 per cent. of sodium sulphate. The product dyes unmordanted cotton salmon shades. The experiments carried out by the author on the manufacture of Erica (diazotised dehydrothioxylidine and α-naphthol-ϵ-disulphonic acid) did not give satisfactory yields, probably due to the presence of the large quantity of acid necessary for the diazotisation. He considers it probable that in order to increase the yield, it is necessary to precipitate the diazo compound by means of salt, sodium sulphate, nitrate, acetate, or the like. The dyestuff gives bluish red shades on unmordanted cotton.

Emin Red.—80 kilos. of dry isothioxylidine chloride are stirred into 160 kilos. of sulphuric acid (66° B.) in a jacketed, lead-lined vessel. When completely dissolved, which requires a temperature of about 50° C., the whole is run into 700 litres of water and 200 kilos. of ice. The vat is then filled up with water, the sulphate allowed to settle, part of the clear liquor run off, leaving sufficient sulphuric acid for the diazotisation. At a temperature of about 5°—10° C., 20 kilos. of nitrite as a 10 per cent. solution are then run in until the diazotisation is complete. After filtering the diazo solution, it is run into 1,200 litres of water containing 52 kilos. of 100 per cent. Schaeffer salt and 70 kilos. of sodium carbonate. The dyestuff is finally salted out with 30—50 kilos. of salt, yielding, after drying, about 130 kilos. of dyestuff. Emin Red is very sparingly soluble in water, and dyes chromed wool red, absolutely fast to milling. The combinations with 1.4-naphthol sulphonic acid and with R-salt give bluer dyestuffs, but they are not so fast to milling.

Fig. 1.



Dehydrothiotoluidine.—6 kilos. of *p*-toluidine and 1·5 kilo. of sulphur are heated in a cast-iron pot provided with an agitator to 190°—195° C. (Fig. 1). Water and *p*-toluidine collect in A, and B contains 6 kilos. of soda-lye (40° B.) in order to absorb the sulphuretted hydrogen. After heating for five hours to 190°—210° C., the receiver is changed and the *p*-toluidine distilled off in about 2½ hours by heating to 300° C. (Fig. 2). Altogether about 2·7 kilos. of *p*-toluidine distil over, and the residue, weighing 3 kilos., is dissolved in a boiling mixture of 9·6 kilos. of sulphuric acid (66° B.) and 14·5 litres of water. The solution is run into 200 litres of water and the sulphate filtered off. The paste weighs 34·5 kilos., and, on titration with 10 per cent. nitrite, contained 2·8 kilos. of 100 per cent. thio-base sulphate, equal to 75·7 per cent. of the theoretical yield. According to Cassella and Co. (Ger. Pat. 53,938), an increased yield is obtained by using naphthalene as a diluent for the melt, the proportions being 2 kilos. of *p*-toluidine, 1·6 kilo. of sulphur, and 2 kilos. of naphthalene. The melt is heated in a Papin's digestor enamelled inside, for six hours to 190°—210° C. (Fig. 3), when it is poured into a hot mixture of sulphuric acid (66° B.), 8 kilos., and water, 12 litres. On boiling, the naphthalene separates, is allowed to cool, and filtered off through jute, the filtrate being precipitated by pouring into 150 litres of water, and titrated with nitrite as above. The yield obtained was 50 per cent. of the theoretical. 20 kilos. of a 5·4 per cent. thio-base paste (= 1·1 kilo. dry) are stirred up with 80 litres of water and diazotised with 1·29 kilo. of a 25 per cent. nitrite solution and 3·7 kilos. of hydrochloric acid (12° B.) (= 4 mols.). This is then run into 59 kilos. of a 3 per cent. α-naphthol δ disulphonic acid containing 2·5 kilos. of sodium carbonate. After boiling up and salting out, 2·4 kilos. of a dyestuff are obtained which give yellower and stronger shades on unmordanted cotton than Geranin 2B. Similar but still yellower dyestuffs are obtained from α-naphthol-ϵ-disulphonic acid or from 1.4-naphthol sulphonic acid, but none of these dyestuffs approach in brightness and blueness, those derived from thioxylidine.

Fig. 2.

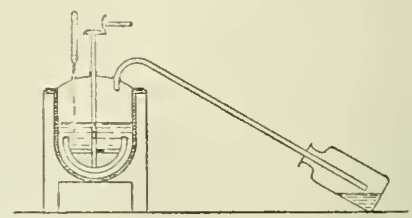
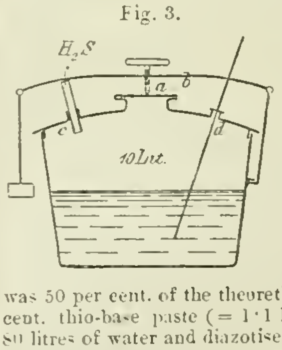


Fig. 3.

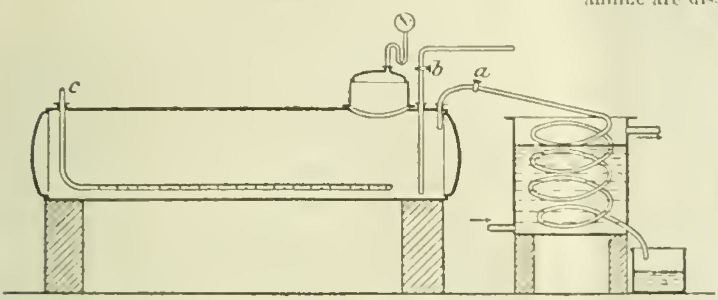


2. *Salicylic Acid Dyestuffs.*—These products, like the alizarins, are lake-forming dyestuffs, and hence are applicable to chrome-mordanted wool, possessing to a high degree fastness to light and milling. Representatives of this class of dyestuffs are Flavazol and Alizarin Yellow R. The former is obtained by combining *p*-toluidine with salicylic acid, giving a product which, after dilution with 5 per cent. of sodium sulphate, forms Flavazol Extra. Flavazol is obtained by diluting Flavazol Extra with 60 per cent. of sodium sulphate. Alizarin Yellow R is formed from diazotised *p*-nitraniline and salicylic acid, and gives on chromed wool somewhat redder shades than Flavazol.

3. *Tropæoline, Metanil Yellow and Orange.*—Tropæolin 00 from diazotised sulphanilic acid and diphenylamine is prepared as follows:—9 kilos. of aniline are stirred into 10 kilos. of sulphuric acid (66° B.) and heated in an oil-bath for 7—8 hours to 215° C. The product is dissolved in water with 5·5 kilos. of sodium carbonate, boiled to remove unaltered aniline, and, after filtering, precipitated with hydro-

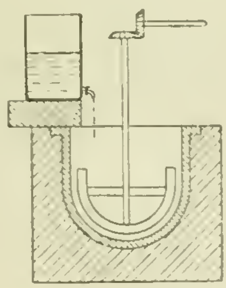
chloric acid. It is then mixed with the calculated quantity of nitrite in 100 litres of water, and 10 kilos. of dilute sulphuric acid (60° B.) are run in within about half an hour, the temperature being kept at 10°—15° C. Meanwhile 18 kilos. of diphenylamine, 63 kilos. of dilute sulphuric acid and water are dissolved in a leaden vessel and allowed to run in a thin stream into the diazo solution obtained as above. The colour acid so formed is filtered, washed, and finally dissolved in dilute potash solution, when the whole is transferred to a boiler (Fig. 4), in which it is heated by

Fig. 4.



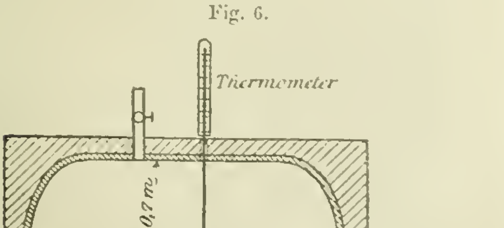
means of the wet steam pipe *c*, any excess of diphenylamine distilling off through *a*. When the distillate is free from diphenylamine, *a* is closed, and the solution is forced by means of *b* through a filter-press into a crystallising vat, where, on cooling, the dyestuff separates out. The sulphanilic acid can also be prepared by means of the baking process, for which purpose 24 kilos. of aniline are run in a thin stream into 27 kilos. of sulphuric acid (66° B.) contained in a cast-iron vessel furnished with an agitator (Fig. 5).

Fig. 5.



The yield of acid sulphate is 49·6—49·7 kilos. 32 kilos. of the salt are heated in an iron chamber, of which a section is given in the accompanying Fig. 6.

Fig. 6.



The temperature is maintained at 230°—235° C. until sulphurous acid is no longer given off. The yield is about 29·5—29·6 kilos., which is close to the theoretical. Traces of aniline are removed as above by boiling the alkaline solution. Naphthionic acid can be prepared in a similar way. Melted naphthylamine is poured into the quantity of concentrated sulphuric acid (66° B.) necessary for the acid sulphate, the temperature rising to 180°—200° C. In order to keep the mass porous, oxalic acid is added to the melt before baking it. In place of the method given above for the preparation of Tropæoline, it can also be made in an alcoholic solution. 10 kilos. of sulphanilic acid free from aniline are dissolved in 36—40 litres of boiling water with soda lye and diazotised with 15 kilos. of hydrochloric acid and 4·2 kilos. of nitrite in a concentrated solution at about 7°—8° C. The diazo compound, after filtering, is mixed with a small quantity of ice water, and run into 7 kilos. of diphenylamine dissolved in 200 kilos. of alcohol (84°—90° Tr.) and 50 kilos. of hydrochloric acid. After standing some time the temperature is allowed to rise to 15° C., when the dyestuff is filter-pressed, and 15—18 kilos. of the product are boiled up with 100—150 litres of water, 4—5 kilos. of ammonia added, and the whole allowed to cool to 30° C., when crystals of the ammonium salt separate out. The yield is 12—14 kilos. of Tropæoline, which is made up to 16 kilos. with sodium sulphate. The spirit is recovered by liming and distillation, and the loss on a monthly consumption of 12—16,000 kilos. is 7—9 per cent., or in summer about 10—12 per cent.

Metanil Yellow.—Metanilic acid is prepared by heating together, at a temperature not exceeding 110° C., 35 kilos. of fuming sulphuric acid and 15 kilos. of nitrobenzene. When the latter has disappeared, the melt is blown into about 350 litres of water, and reduced by adding 27 kilos. of zinc dust. The reduced solution is then allowed to settle, filtered, cooled with ice, diazotised at 5°—10° C. with 5·7 kilos. of nitrite, and 10 kilos. of dilute sulphuric acid are finally added. The combination with 20 kilos. of diphenylamine is performed as above. In place of zinc dust, the following process may be employed:—75 kilos. of nitrobenzene are run into 150 kilos. of fuming sulphuric acid at 100°—105° C. The melt is then heated for two hours to 110°—115° C., then blown into 1,000 litres of water, and reduced with 75 kilos. of cast-iron borings in the course of 1½ to 2 hours. After boiling up, liming, and filtering, the calcium salt solution is concentrated to 22°—24° B. and filtered from calcium sulphate. The yield is 300—320 kilos. of a 32—36 per cent. strength, corresponding to 100—110 kilos. of metanilic acid. The combination with diphenylamine can take place in an alcoholic solution, and 20—25 kilos. of the colour acid so obtained are boiled up with 200 litres of water and 4—5 kilos. of sodium carbonate, filtered, mixed with 25—30 kilos. of salt, and allowed to cool to 30° C., when 18—20 kilos. of Metanil Yellow (M G F) crystallise out, which, after grinding with salt, give about 55 kilos. of Metanil Yellow (M G F O O). The colour acid can also be converted into a sulphonic acid by treatment with five-sixths its weight of fuming sulphuric acid (40 per cent. SO_3) at about 60° C. The solution of the calcium salt is then evaporated nearly to crystallisation, converted into the sodium salt, and dried down.

Curcumein.—About 19—21 kilos. of pure Tropæoline are stirred up with water and treated with 4—5 kilos. of nitrite as a 25 per cent. solution, 18 kilos. of nitric acid, and 21 kilos. of sulphuric acid (66° B.). The whole is then boiled for three hours, when quantities of nitrous acid vapours escape. After filtering, the dyestuff is salted out and pressed.

4. *Orange II.* is produced from diazotised sulphanilic acid and β-naphthol. The former is obtained as above described, but no details are given as to the strength of the naphthol solution or as to yield.

Orange R. — 60 kilos. of α-naphthol are added to 96 kilos. of sulphuric acid heated to 70° C., and raised to 90° C. for three hours. The melt is dissolved in

water, and this solution is poured into 500 litres of brine containing 100—150 kilos. of salt. After filtering off the precipitated acid sodium salt of the α-naphthol sulphonic acid, the filtrate is treated with 10 kilos. of sodium carbonate, and a solution of diazoxylene (from 12 kilos. of xylidine, 30 kilos. of hydrochloric acid, and 1,000 litres of water) is run in. This yields, after salting out with 300 kilos. of salt, about 80 kilos. of Orange R. The acid sodium salt of the α-naphthol sulphonic acid is dissolved in 1,250 litres of water, boiled up with 70 kilos. of sodium carbonate, and filtered. The solution is halved and combined with diazoxylene as above. The filtrate still contains some naphthol sulphonic acid, which is combined with the diazonaphthalene sulphonic acid from 8 kilos. of naphthionate, 150 litres of water, 12 kilos. of hydrochloric acid, and 2·5 kilos. of nitrite. The yield of Ponceau is 15 kilos In Ger. Pat. 28,820, the author describes a process for the production of brown and red dyestuffs, and in the present paper, details of the manufacture are given. The products obtained, however, have been replaced by others which show a greater evenness in dyeing and are faster to milling. The process consists in combining diazotised amido-azobenzene with naphthionic acid, and converting the dyestuff so obtained into the corresponding naphthol derivative by diazotising and boiling with water. The following method is given for the preparation of amido-azobenzene. A vat holding 1,000 litres, provided with a cover, agitator, and draught pipe, contains 464 kilos. of aniline. Whilst the agitator is running, about 640 kilos. of hydrochloric acid (20 B.) are run in, until Congo paper is turned blue or magenta paper white. The temperature rises to about 75° C. 27·6 kilos. of the mixture are mixed in a half petroleum cask with ice, bringing the temperature to about 8—10° C. 13·8 kilos. of nitrite solution are run in, keeping the temperature by means of ice at about 10° C. The diazo-amidobenzene formed is placed in a second vat and agitated, when the transformation into amido-azobenzene takes place. It is complete when a sample dissolved in alcohol and a few drops of ammonia does not turn red on the addition of soda lye. The amido-azobenzene hydrochloride is filtered off on flannel, and washed with water and hydrochloric acid about six times to obtain it free from aniline. The yield is 173 kilos. of a 44 per cent. paste = 76 kilos. of dry amido-azobenzene hydrochloride from 116 kilos. of aniline. Adding to this the amount of aniline recovered from the wash liquors, the yield is 75 per cent. of the theoretical. For the diazotisation, the above amido-azobenzene hydrochloride is stirred up with a small quantity of water, and brought up with 63 kilos. of hydrochloric acid and 150 kilos. of ice to about 400 litres. 25 kilos. of nitrite in 50—75 litres of water are then run in, so that nitrous acid and ice are always present. The volume increases to about 1,000 litres. The diazo compound is filtered off and washed four times. The filtrate, together with the first wash water is precipitated with Chili salpetre or nitric acid, the total yield being 376 kilos. of diazo-azobenzene paste, which, on titration with Schaeffer salt, corresponded to 18 kilos. of aniline. On combination with sodium naphthionate, the author obtains Cotton Red, which, on diazotisation and boiling with water, yields Fast Red. Neither of these colours has, however, any technical importance. When amido-azobenzene is treated with fuming sulphuric acid, it is converted into a mono- and a disulphonic acid. The former, when diazotised and combined with 2.1-naphthol sulphonic acid, yields Croceine 3 B, whilst the disulphonic acid gives with β-naphthol, Biebrich Scarlet. For the sulphonation, the hydrochloride is heated with $2\frac{1}{2}$ times its weight of alcohol and ammonia, filtered, and the filtrate precipitated with sulphuric acid. One kilo. of the dried, ground, and sieved sulphate is added to 1·5 kilo. of fuming sulphuric acid (25 per cent. SO_3), the temperature being kept below 35° C. A further quantity of 2·5 kilos. of the fuming acid is then added, and the melt is maintained at 35° C. until a sample dissolves in dilute ammonia. The principal products then are mono- and disulphonic acid, whereas at 70° C., di- and trisulphonic acids are chiefly formed. In the former case the melt is poured into brine and the precipitate extracted with boiling water, when the monosulphonic acid remains behind as a brownish-yellow paste. The disulphonic acid is precipitated by salt from the filtrate in bluish-red plates or needles. The diazo compound of the monosulphonic acid is almost insoluble in water, whilst that of the disulphonic acid is only precipitated on adding salt. By combining this latter with sodium naphthionate, a brown dyestuff for wool is obtained. When this is diazotised and boiled with water, it forms a red dyestuff which dyes wool from an acid bath and cotton from an alum bath bluish-red, but lacks advantages rendering it of technical importance.—T. A. L.

Dyestuffs [*Synthetic*], *and Raw Products therefor; Manufacture of. III.* L. Paul. Zeits. angew. Chem. 1897, 20—25, and 47—52.

The diazotisation of diamidotriphenylmethane and its homologues, and subsequent conjugation with R salt, gives a series of red dyestuffs for wool, which cannot, however, compete with the various Ponceaus, both on account of price and from the fact that they do not exhaust the dye-bath. They are, however, employed for the production of lakes and for printing, one of these being Vermilion Scarlet. Diamidotriphenylmethane is obtained by heating together under a reflux condenser for 3—4 hours, 53 grms. of benzaldehyde, 93 grms. of aniline, and 130 grms. of hydrochloric acid (20° B.) About 41 c.c. distil over, containing 4·5 c.c. of benzaldehyde. The melt is dissolved in 750 c.c. of water, steamed to remove unaltered benzaldehyde, made alkaline, and again steamed to remove aniline, and the diamidotriphenylmethane is filtered off. To purify it further, it is again dissolved in hydrochloric acid and carefully neutralised with sodium carbonate solution. The yield is 250 grms. of paste = 88 grms. of 100 per cent. base, or 65 per cent. of the theoretical yield. For the diazotisation, 50 grms. of the paste are dissolved in 250 c.c. of water, together with 75 grms. of hydrochloric acid (12° B.). A 10 per cent. solution containing 8·8 grms. of sodium nitrite is then slowly added, and the clear diazo solution, measuring about 750 c.c., is run into 600—700 c.c. of water containing 65 grms. of R salt and 40 grms. of sodium carbonate, so that the final volume amounts to about 2 litres. After standing some time, the whole is warmed to 50°—60° C., and separated by means of 500—600 grms. of salt, when the dyestuff is filter-pressed and dried. The yield of damp paste amounts to 130 grms. = 87·5 grms. of dry dyestuff containing 10·5 grms. of salt. From 200 grms. of base, 342 grms. of dyestuff were obtained free from salt. For producing a lake, four solutions were made up, containing (1) 71 grms. of sodium carbonate; (2) 216 grms. of barium chloride; (3) 130 grms. of magnesium chloride; (4) 43 grms. of caustic soda, each in 1 litre of water. 2 grms. of the colour were dissolved and mixed successively with 50 c.c. of (1), 50 c.c. of (2), 25 c.c. of (3), and 25 c.c. of (4). The lake is then filtered off, smeared on paper, and dried. Compared with Vermilion Scarlet, the colour is 10 per cent. stronger, but also bluer. On substituting G for R salt, only an incomplete combination takes place with the diazotised diamidotriphenylmethane. By condensing *m*-xylidine with benzaldehyde as above, diazotising the base, and combining it with R salt, an orange lake is obtained. From *p*-toluidine (107 grms.), 53·5 grms. of benzaldehyde, and 130 grms. of hydrochloric acid, a yield of only 17 grms. of the diamido base was obtained. When *p*-xylidine 1,210 grms., benzaldehyde 530 grms., and 1,300 grms. of hydrochloric acid are heated together under a condenser for 45—60 minutes at 110° C., a separation of the diamido hydrochloride takes place. After filtering off and washing with 1—1·5 litres of hydrochloric acid, a yield of 1·1 kilo. is obtained, corresponding to 50 per cent. of the theoretical. A further amount can be obtained by prolonged heating of the mother-liquor. A nitro derivative of this base is formed by heating together 7·2 grms. of *p*-nitrobenzaldehyde, 11 grms. of *p*-xylidine, and 11·5 grms. of hydrochloric acid. The melt, after dissolving in water and filtering, yields the base on adding sodium carbonate. By applying the condensation to dimethyl-*p*-xylidine, a tetramethylated base is obtained, which, on oxidation with lead peroxide, gives a green soluble in spirit. Dimethyl-*p*-xylidine is prepared by heating 20 grms. of *p*-xylidine, 70 grms. of methyl iodide, 47 grms. of sodium carbonate, and 500 c.c. of water under an inverted condenser until the methyl iodide has disappeared. After extracting with ether

and distilling off the latter, a light yellow oil remains, boiling at 200°—202° C. 6·2 grms. of this oil, 2·2 grms. of benzaldehyde, and 5·7 grms. of hydrochloric acid were heated together as already described. Benzaldehyde distils over and the viscid melt is steamed to remove dimethyl-*p*-xylidine. The residue is filtered off and washed. An attempt to methylate *p*-xylidine by the ordinary method as below gave no satisfactory result. For the preparation of dimethylaniline the following quantities are employed. An autoclave is charged with 100 kilos. of aniline, 96·5 kilos. of methyl alcohol, 18—19 kilos. of sulphuric acid (64°·5 B.), and heated for 7 hours to 200°—210° C. After adding 40 kilos. of soda lye (40° B.), the dimethylaniline is driven off with steam. The yield is 125 kilos., whilst 12—14 kilos. of methyl alcohol are recovered.

Indazine.—By condensing nitrosodimethylaniline with diphenyl-*m*-phenylene diamine, a valuable blue dyestuff is formed, and a homologue of this is obtained by oxidising dimethyl-*p*-phenylene diamine together with ditolyl-*m*-phenylene diamine. This latter substance is produced by heating together 1·1 kilo. of resorcinol, 4·3 kilos. of *p*-toluidine, and 1·4 kilo. of *p*-toluidine hydrochloride, in a cast-iron vessel, provided with an agitator, to 170°—180 C., when a mixture of water and *p*-toluidine distils over. The next day the temperature is raised to 270° C., and after about 30 hours' heating, the melt is boiled up in a 40 litre earthenware pot containing 1·5 kilo. of hydrochloric acid (20° B.). The residue is then extracted with soda lye and the insoluble portion is recrystallised, from one and a half to twice its weight of benzene, forming slightly coloured needles. The yield is 2·6 kilos. For the preparation of the amidodimethylaniline, 37·3 grms. of nitrosodimethylaniline suspended in 750 grms. of alcohol (90° Tr.) were treated with 55 grms. of zinc dust. The reaction is completed by warming to 40°—50° C., and when the solution is decolorised, it is heated to boiling, and filtered from the zinc dust. 29 grms. of ditolyl-*m*-phenylene diamine are then dissolved in the filtrate, and to the clear, hot solution is added in small quantities, 300 c.c. of 50 per cent. alcohol, containing 60 grms. of sodium bichromate ($Na_2Cr_2O_7 2H_2O$). The oxidation is completed by heating for 2 hours on the water-bath, when the alcohol is driven off with steam. The hot aqueous solution measuring about 2 litres, gives, on the addition of 240 c.c. of hydrochloric acid (11° B.), a precipitate of the dyestuff, which, when dried, weighs 51 grms. It contains 4 per cent. of ash and is 30 per cent. stronger than ordinary Indazine. The mother-liquor, on adding zinc chloride, yields a further 5—6 grms. of the dyestuff.

Naphthohydroquinone.—500 grms. of β-Naphthol Orange are dissolved in 2 litres of boiling water, to which 500 grms. of hydrochloric acid are added. At the same time 750 grms. of tin salt are dissolved in 725 grms. of hydrochloric acid, and added tolerably quickly to the first solution, with constant agitation. A mixture of sulphanilic acid and 1.2-amidonaphthol hydrochloride separates out, and, after filtering off, is dissolved in 8—10 litres of hot water. A small quantity of pure sulphanilic acid crystallises out, which is filtered off. The filtrate is precipitated with a concentrated sodium acetate solution, in the proportion of 300 grms. of the salt to 1 litre of the filtrate. After drying at air temperature, 160—170 grms. were obtained, corresponding to 70—72 per cent. of the theory. On evaporating the filtrate, about 80 grms. of sulphanilic acid are recovered. For oxidising the base, 160 grms. of it are stirred up with water to a thin paste, dissolved cold with 400 grms. of dilute sulphuric acid (1:1), and oxidised with 200 grms. of sodium bichromate in 300 c.c. of water. After 10 minutes the formation of the β-naphthoquinone is complete, giving 140—142 grms., corresponding to a yield of 90 per cent. of the theoretical. In order to reduce it, 140 grms. of the β-naphthoquinone as a 15 per cent. paste mixed with ice, are stirred into 8 litres of an aqueous solution of sulphurous acid. The temperature is maintained at 8°—9° C. by means of ice, and the β-naphthoquinone gradually dissolves. After standing 12 hours, naphthohydroquinone separates out, the filtrate being extracted with ether. The crude product so obtained was dissolved in 4 litres of water, together with 50 grms. of sulphurous acid, at about 60° C. The hydroquinone which separates out on cooling is again recrystallised from 3 litres of water and 50 grms. of sulphurous acid. Any dinaphthylhydroquinone is removed by filtration, and the β-naphthohydroquinone separates out on cooling. The yield is 80 grms., being about 50 per cent. of the theoretical. It is, however, much cheaper to prepare 1.2-dihydroxynaphthalene from 1.2-nitrosonaphthol, which is easily obtained from β-naphthol by the following method:—20 kilos. of β-naphthol, 50 kilos. of denatured alcohol, 8 kilos. of zinc chloride in 2½ litres of water, and 5 kilos. of nitrite in 4—5 litres of water, are mixed in a stoneware three-necked Woulff's bottle which can be heated externally with steam. One tubulure is closed, another serves for the insertion of a thermometer, and the third is connected to an inverted condenser. The reaction commences at 60°—70° C., gradually rising to 80°—85° C., and is complete in about half an hour. The whole is then allowed to cool, the contents removed from the vessel by means of a vacuum, the zinc salt then filtered off, and twice washed each time with 10 kilos. of alcohol. After centrifugating, 22·5 kilos. of damp 1.2-nitrosonaphthol zinc salt are obtained. For the reduction to 1.2-amidonaphthol, it is necessary to first obtain the acid, and for this purpose the zinc salt obtained is stirred to a thick paste with water, cooled with ice to 0°—5° C., and decomposed with 20—25 kilos. of hydrochloric acid (20° B.). The sludge so obtained is added to a solution of 30 kilos. of tin crystals (stannous chloride) in 60 kilos. of hydrochloric acid, keeping the temperature at about 5° C. The solution solidifies to a mass of greyish-white crystals, which, after filtering off, are dissolved in about 100 litres of water with the addition of about 10 kilos. of hydrochloric acid. Scrap zinc is then added so long as tin is precipitated, and the solution is filtered into an aqueous solution of sulphurous acid. After standing several days, 1.2-amidonaphthol hydrochloride separates out, or is precipitated by adding hydrochloric acid. The yield is 70—80 per cent. of the theoretical, calculated on the naphthol. The oxidation to β-naphthoquinone is effected by means of ferric chloride, the preparation of which from the iron sludge obtained in manufacturing aniline is described subsequently. 220 grms. of 1.2-amidonaphthol hydrochloride together with 100—110 grms. of sulphurous acid are dissolved in 2—2½ litres of boiling water. After filtering from a bulky greenish precipitate, the solution (which, with the washings, amounts to 5 litres) is, after cooling, mixed with 135 grms. of hydrochloric acid (20° B.) and subsequently with a solution of 440 grms. of ferric chloride in 1 litre at a temperature of about 10° C. The light yellow crystalline precipitate of β-naphthoquinone is filtered off and washed with 2 litres of water, and after thoroughly draining weighed 270 grms. For the conversion into the hydroquinone, the precipitate is stirred up with water and added to 10—15 litres of water containing 1·5 litres of sulphurous acid. After standing some time the solution is extracted with 3 litres of ether, giving 82 grms. of the product, which corresponds to about 50 per cent. of the theoretical, assuming that 195 grms. of sodium 1.2-nitrosonaphtholate correspond to 220 grms. of amidonaphthol hydrochloride. By combining diazotised sulphanilic acid with naphthohydroquinone, an orange-yellow dyestuff is obtained which gives bluish-violet shades on chrome wool very fast to milling. Similar dyestuffs can be obtained from 2.1-naphthylamine sulphonic acid or from naphthionic acid, but since only low yields were obtained, their manufacture was not proceeded with.

For the preparation of the ferric chloride above alluded to, 33 kilos. of iron sludge from the manufacture of aniline were dissolved in 66 kilos. of hydrochloric acid (20° B.) by boiling with direct steam, the final weight being 117 kilos. This solution was then oxidised with a mixture of 14 kilos. of nitric acid (29° B.) and 28 kilos. of hydrochloric acid (20° B.). The whole is again boiled, and gives finally 225 kilos. of a 26·5 per cent. ferric chloride solution (20° B.), corresponding to 60 kilos. of solid salt, at a cost of about 1·25*d.* per kilo.

A sulphonic acid of the above dihydroxynaphthalene has also been patented, and is capable of yielding dyestuffs. The starting point for this acid is Eikonogen, which is the sodium salt of 1.2.3′-amidonaphthol sulphonic acid. It is obtained by reducing 1.2.3′-nitrosonaphthol sulphonic

acid with tin salt and crystallising it from a concentrated sodium bisulphite solution. For use as a photographic developer it is ground with sodium metabisulphite, to prevent oxidation. The following method is given for converting it into the dihydroxynaphthalene sulphonic acid. 500 grms. of Eikonogen dissolved in 2 litres of water are cooled with ice to 5° C., precipitated with 291 grms. of sulphuric acid, and oxidised at a low temperature with 1,460 grms. of a 31 per cent. lead peroxide paste. The yellowish-orange solution is decanted from the lead sulphate and added to 10 litres of an aqueous solution of sulphurous acid. On evaporation, the ammonium salt of β-naphthohydroquinone crystallises out, and weighed, when dry, 450 grms. When combined, in presence of calcium acetate, with various diazotised aromatic amidosulphonic acids, it gives dyestuffs producing bluish shades on chrome-mordanted wool: but since they dye unevenly, and give, with the exception of that from β-naphthylamine-β-sulphonic acid (Brönner), duller shades than those from the non-sulphonated dihydroxynaphthalene, they have not been produced on a manufacturing scale.—T. A. L.

Quinones, General Reaction for Aromatic. S. Blumenfeld and P. Friedländer. Ber. **30**, 1464.

See under XXIII., *page* 764.

Nitrobenzene, Electrolytic Reduction of. W. Löb. Zeits. f. Elektrochem. **3**, 1897, 471.

See under XI. A., *page* 744.

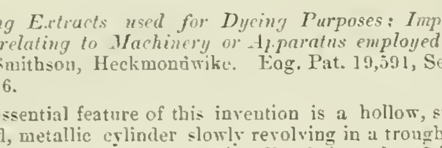
PATENTS.

Drying Extracts used for Dyeing Purposes; Impts. in or relating to Machinery or Apparatus employed for. S. Smithson, Heckmondwike. Eng. Pat. 19,591, Sept. 4, 1896.

The essential feature of this invention is a hollow, steam-heated, metallic cylinder slowly revolving in a trough containing the extract; the adhering film being thus dried, is removed from the revolving cylinder by scrapers.—I. S.

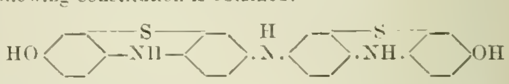
Dyes [Blues, Blacks], An Improved Manufacture of. H. R. Vidal, Paris, France. Eng. Pat. 13,093, June 3, 1896.

The patentee describes the stages in which he considers the reaction of sulphur on substances like hydroquinone and *p*-amidophenol takes place. Sulphur reacting on *p*-amidophenol yields *p*-dihydroxythiazine, together with one molecule of ammonia, which, in presence of the sulphur, effects a further condensation, and generates *p*-dihydroxytetraphenetrithiazine—



The same compound can be obtained from hydroquinone by acting on it with ammonia and sulphur.

Analogous compounds, in which the hydroxyl is totally or partially replaced by the amido group, are obtained by the action of sulphur on *p*-diamidotriazine or on *p*-hydroxyamidothiazine. Thus, *p*-diamidotetraphenetrithiazine, a black dyestuff, soluble with difficulty in alkaline sulphides, can be obtained by heating in an autoclave to 210°—240° C., 24 kilos. of *p*-amidophenol, 6·4 kilos. of sulphur, and 1·5 kilo. of phospham (PN_2H). The amido-hydroxy derivative is obtained by heating together at the same temperature, 12 kilos. of *p*-amidophenol, 12 kilos. of *p*-phenylene diamine, and 6·2 kilos. of sulphur. Furthermore, a class of dyestuffs is described, intermediate between the thiazines and tetraphenethiazines, which are the triphenethiazines. A mixture of hydroquinone 11 kilos., *p*-dihydroxythiazine 23 kilos., phospham 1·5 kilo., and sulphur 3·2 kilos., is heated to 210°—240° C. These substances form blue to black dyestuffs for vegetable or animal fibres, from baths reduced by sulphides or alkaline sulphites or bisulphites. They may also be obtained by the action of sulphur on *p*-diamidodiphenylamine or *p*-hydroxyamidodiphenylamine, or by the action of sulphur and ammonia or phospham on *p*-dihydroxydiphenylamine, or upon *p*-hydroxyamidodiphenylamine. If phospham (1·5 kilo.) alone be allowed to react at 210°—240° C. on two molecular proportions (23 kilos.) of *p*-dihydroxythiazine, a substance having the following constitution is obtained:—



and, by varying the components, other analogously constituted compounds are formed. (See also Eng. Pats. 3612 of 1895, 16,449 and 18,489 of 1896; this Journal, 1896, 30, and 1897, 608 and 437.)—T. A. L.

Black Colouring Matter, Production of. S. Pitt, Sutton, Surrey. From L. Cassella and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 19,831, Sept. 8, 1896.

The dyestuff is obtained from *m*-dinitrophenol ($OH : (NO_2)_2 = 1:2:4$) by heating it with alkaline sulphides and sulphur, the following quantities being taken:—25 kilos. of *m*-dinitrophenol, 125 kilos. of sodium sulphide, and 50 kilos. of sulphur are heated within an hour to 100° C. The temperature is then gradually raised to 160° C., and maintained thereat until the melt solidifies, which takes place in 2—3 hours. The melt, after breaking up, may then be used directly, or it may be carefully neutralised with hydrochloric acid, filtered off, and left as a paste, or dried. In this latter case the dyestuff must be dissolved in soda lye or sodium sulphide before use. It dyes black directly on unmordanted cotton. If less sulphur be used, a brown colour is obtained. —T. A. L.

Compounds of the Series of the Indoxylic Acids and of Dyestuffs of the Indigo Series, A Process for the Manufacture of. R. Blank, Berlin. Eng. Pat. 19,946, Sept. 9, 1896.

The process consists in converting chloro- or bromo-malonic acid or their esters by heating with aromatic amines into substituted amido-malonic acids or their esters, which, when heated, are converted into indoxylic acids and esters. These compounds, on exposure to air in an alkaline solution, are converted into the corresponding indigos. The following example gives the quantities employed, starting from *p*-toluidine. An alcoholic solution containing 9 kilos. of *p*-toluidine is mixed with 10 kilos. of bromo-malonic acid ester and allowed to stand 2 days, after which the melt is heated on the water-bath for 2 hours. About 50 litres of water and some hydrochloric acid are then added, and the insoluble oil is separated and cooled, when it crystallises. After purification, the *p*-toluido-malonic acid ester melts at 55° C., and is easily soluble in the ordinary organic solvents. The acid can be obtained from this by saponification with alkalis or alkaline earths, or it may be produced directly as follows:—A solution of 6·5 kilos. of chloro-malonic acid in alcohol is mixed with an alcoholic solution of 10 kilos. of *p*-toluidine, heated for an hour on the water-bath, and after distilling off the alcohol, the residue is extracted with dilute hydrochloric acid. The residue is re-crystallised from dilute alcohol and forms colourless crystals which melt with decomposition at 117° C. When the ester is heated to about 250°—255° C., alcohol is split off and the *p*-tolyl-indoxylic acid ester is crystallised from alcohol, forming colourless crystals melting at 156° C. It dissolves in alkalis, and can be precipitated by carbonic acid from the solution. On boiling it with alkalis or alkaline earths, salts of *p*-tolylindoxylic acid are obtained, whilst on heating it with concentrated sulphuric acid on a water-bath, it yields sulphonic acids of *p*-tolyl indigo. Paratolylindoxylic acid is obtained by heating sodium *p*-toluido-malonate with four times its weight of sodium hydrate to 300°—350° C. for about five minutes with continual agitation. The melt is then cooled and stirred into cold, very dilute sulphuric acid, when the new acid separates out as a bluish-white powder melting at 117° C. Its alkaline solution is readily oxidised in the air, depositing *p*-tolyl indigo. This product is blue,

in the form of powder, but has a reddish colour in larger lumps. It gives blue solutions in alcohol, aniline, and sulphuric acid, and its paraffin solution is reddish-violet when hot, and bluish-violet when cold. The formation of the indigo may of course take place upon the fibre.
—T. A. L.

Basic Disazo Colouring Matters, Manufacture of. O. Imray, London. From "The Farbwerke vorm. Meister, Lucius, and Brüning," Hochst a Main, Germany. Eng. Pat. 19,976, Sept. 9, 1896.

The base employed for the production of these dyestuffs is *p*-amidobenzylamine or one of its derivatives alkylated in the side chain. The product, after diazotisation, is allowed to act on an azo dyestuff capable of combining, or else the amido-azo dyestuffs produced from the amidobenzylamine derivatives and primary amines are diazotised and combined with phenols or amines. 100 kilos. of the zinc chloride double salt of *p*-amidobenzyldiethylamine are dissolved in 60 kilos. of concentrated hydrochloric acid diluted with water. A solution of 18 kilos. of sodium nitrite is then added and the whole is combined with 37 kilos. of α-naphthylamine, 150 kilos. of sodium acetate, and 70 kilos. of hydrochloric acid. After standing some hours the solution is again diazotised by adding 200 kilos. of hydrochloric acid and the necessary amount of the nitrite, and is then combined with 37 kilos. of β-naphthol in an alkaline solution. The solution is finally boiled to complete the reaction, when the dyestuff is filtered off. By adding hydrochloric acid, it is converted into a salt soluble in water. No reference is made to the shades obtainable, but the dyestuffs are said to be suitable for dyeing mixed goods from an acid bath.—T. A. L.

Para-Amido-Alpha-Naphthol and of Colouring Matters derived from them, Manufacture of Derivatives of. O. N. Witt, Berlin, Germany. Eng. Pat. 20,676, Sept. 18, 1896.

The subject matter of this specification has already been abstracted (Ber. **29**, 2945; this Journal, 1897, 134). In addition, the patentee states that naphthacetol (*p*-acetamido-α-naphthol) may also be obtained from diacetyl-*p*-amidonaphthol by stirring this product to a thin paste with strong caustic soda or potash and heating on the water-bath until a clear solution is formed. The naphthacetol is then precipitated on adding an acid.—T. A. L.

Colouring Matters [Saccharo-fluoresceïn], Impts. in and relating to the Production of. G. B. Ellis, London. From "La Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier," Lyons, France. Eng. Pat. 21,196, Sept. 24, 1896.

These dyestuffs are allied to fluoresceïn, and are obtained by condensing resorcinol with saccharine (anhydro-o-sulphamine benzoic acid). 22 kilos. of resorcinol are heated with 18 kilos. of pure saccharine for seven hours to 200°—220° C. After cooling, the melt is extracted with cold dilute sodium carbonate, and the dyestuff is precipitated with acid. It is purified by dissolving in alcohol and precipitating with water, when, after washing and drying, it forms a brown resinous product. The saccharo-fluoresceïn thus produced may be obtained in a pure state by converting it into its acetyl derivative, which is subsequently hydrolysed. The product, obtained as above, is boiled with three times its weight of acetic anhydride under a reflux condenser. The acetyl derivative is precipitated with alcohol, and separates from glacial acetic acid on adding alcohol, as a crystalline powder, melting at 286° C. When boiled with alcoholic caustic soda it dissolves on hydrolysis, and the solution, after dilution, is acidified with acetic acid. Saccharo-fluoresceïn separates out, and may be crystallised from alcohol. The pure product melts at 267° C. Like ordinary fluoresceïn, it may be converted into halogen derivatives, of which the following is an example:—10 kilos. of saccharo-fluoresceïn are dissolved in 50 kilos. of alcohol, and 11 kilos. of bromine are gradually added at about 40° C.: 3 kilos. of sodium chlorate in 3 litres of water are then run in, and the bromine compound begins to separate in small reddish-brown crystals. The crystalline powder thus obtained is insoluble in water and alcohol, but soluble in alkalis with a red colour. It has similar properties to eosine, but dyes a bluer shade. The iodo-saccharo-fluoresceïn is soluble in alkalis to a violet-red solution.—T. A. L.

New Black Mordant-dyeing Colouring Matter, Manufacture of. C. D. Abel, London. From "The Actien Gesellschaft für Anilin Fabrikation," Berlin, Germany. Eng. Pat. 21,437, Sept. 28, 1896.

The following process is employed:—22·1 kilos. of sodium picramate ($1.2.1.6.C_6H_2.OH.(NO_2)_2.NH_2$) are diazotised, and the solution is run into a solution of 22·3 kilos. of 1.3′-naphthylamine sulphonic acid acidified with acetic acid. The azo compound thus formed is rediazotised by means of hydrochloric acid and sodium nitrite, and the diazo compound which separates, is stirred into a caustic alkaline solution of 14·4 kilos. of β-naphthol. The dyestuff separates as a bluish-green precipitate, and is filter-pressed and dried. It dissolves in water to a bluish-violet solution and in sulphuric acid with a blackish-violet colour. It dyes wool from an acid-bath blackish-violet shades, which, on treatment with chromium salts, are converted into dark black, perfectly fast to washing and light. The nature of the dyestuff is not altered if 1.2′-naphthylamine sulphonic acid, or a mixture of this with the 1.3′ acid, be employed.—T. A. L.

New Violet Mono-azo Dyestuffs for Wool, Dyeing a Fast Brown by Oxidation with Chrome; Manufacture of. O. Imray, London. From "The Farbwerke vorm. Meister, Lucius, and Brüning, Höchst a Main, Germany. Eng. Pat. 23,175, Oct. 19, 1896.

Diazo compounds of 1.1′-naphthol sulphonic acids when combined with α-naphthylamine, its alkyl and alphyl derivatives in an acid solution, yield violet dyestuffs, which, on treatment with chromates or chromic acid, are converted into browns very fast to milling and light. For instance, 2·39 kilos. of 1.1′. 4-amidonaphthol sulphonic acid, together with 0·42 kilo. of caustic soda, are dissolved in about 90 litres of water. A strong solution containing 0·69 kilo. of sodium nitrite is then added, and the whole is allowed to run into an ice-cold solution containing 0·92 kilo. of hydrochloric acid. The yellowish-brown precipitate of the diazo compound separates out, and is added to 1·43 kilo. of α-naphthylamine dissolved in 30 litres of water, together with the calculated quantity of hydrochloric acid at a temperature of about 25° C., 3 kilos. of crystallised sodium acetate are added, and finally, in order to complete the reaction, the whole is heated to 80° C.; 2·2 kilos. of sodium carbonate are then added, when the dyestuff is converted into the sodium salt. It forms, when dry, a greenish-black powder, sparingly soluble in cold, more easily in hot water, and having dyeing properties as above mentioned. Other amidonaphthol acids which may be employed are amidonaphthol disulphonic acid H, amidonaphthol sulphonic acid S, 1.1′.4′-amidonaphthol sulphonic acid, 1.1′.3-amidonaphthol sulphonic acid, 1.1′.3′-amidonaphthol sulphonic acid, or 1.1′.2′-amidonaphthol sulphonic acid. The combination with α-naphthylamine, or its derivatives may also take place in an alcoholic solution.—T. A. L.

New Colour Base, and of Colouring Matters therefrom [Red]: Production of a. I. Levinstein and Levinstein, Ltd., Manchester. Eng. Pat. 25,725, Nov. 16, 1896.

The base referred to is a dichlorobenzidine, which is obtained by the action of bleaching powder on diacetylbenzidine dissolved in sulphuric acid, and subsequent hydrolysis of the product by boiling it with hydrochloric acid. The following quantities are employed:—26·8 kilos. of diacetylbenzidine are dissolved in 3 kilos. of concentrated sulphuric acid, and the solution is poured into ice-cold water. The mixture, being kept at a low temperature, has added to it, with constant agitation, sufficient of a 10 per cent. bleaching powder solution to form a dichlorobenzidine. The temperature is then raised to 40° C., and after standing 12 hours, the new dichlorodiacetylbenzidine is filtered off as a yellowish precipitate. In order to hydrolyse it, it is boiled with 3—4 times its weight of 20 per cent. hydrochloric acid; and the solution, on cooling, deposits dichloro-

benzidine hydrochloride, which is converted into the base in the well-known manner. It may also be chlorinated by suspending the same quantity of the diacetyl derivative in 200 litres of brine, introducing some iron wire to act as a carrier, and passing chlorine through so long as it is absorbed, the liquor meanwhile being cooled externally. For the preparation of a dyestuff, 26 kilos. of the base are dissolved in 300 litres of water containing 70 kilos. of hydrochloric acid (18° B.), and diazotised cold with an aqueous solution of 14 kilos. of sodium nitrite. The tetrazo compound produced is run into an aqueous solution of 50 kilos. of sodium naphthionate (100 per cent.), containing sufficient sodium acetate to neutralise the mineral acid. The dyestuff separates as a red precipitate, and after stirring 12 hours it is made alkaline, heated to 80° C., salted out, filter-pressed, and dried. It dyes unmordanted cotton bluish-red, comparatively fast to acids. By employing β-naphthylamine sulphonic acid (Brönner) in place of the naphthionic acid, a bright bluish-red is obtained, which is fast to organic acids. In general, the dyestuffs which are obtained from this base, are much bluer and faster to acids than the corresponding derivatives from tetrazodiphenyl or tetrazoditolyl.—T. A. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

Soupling of Half-Silk Goods. Textile Colorist (U.S.), 19, 140—141.

The soapling process is not applied to piece-goods consisting entirely of silk, since the strength and ductility of the fibre are affected thereby. But in the case of half-silk goods, soupling in the piece offers several advantages. In such goods the silk is generally used in the form of "grège" or untwisted threads, and only shows on the surface of the material, and, when soupled, the silk swells and entirely covers the surface; it also becomes easier to dye, while the strength of the fibre is very slightly impaired. The cotton or wool at the same time acquires a "scroopy" silk-like handle.

For wool-silk goods, soupling is effected as follows:—The material is worked for 1—2 hours in a weak, lukewarm soda solution, and rinsed. After crabbing, it is then treated in a bath containing 8 per cent. of sulphuric acid (6° B.) and 3 per cent. of tartar, raising to the boil in an hour and maintaining at that point for 1—2 hours. For white and pale shades the goods are stoved; for dark colours this is omitted, and dyeing with acid dyestuffs at once follows. Drying on the tentering-frame and lustreing, complete the operation.

Cotton-silk goods are scoured in a weak soap-bath, washed, soupled with 2 per cent. of bisulphite of soda, 3 per cent. of tartaric acid, and 3 per cent. of tartar, washed, dyed, and dried on a cylinder machine. Hot pressing, &c. may follow according to the finish required.—R. B. B.

Acid Fulling. [*Woollens and Felt.*] Textile Colorist (U.S.), 19, 143—144.

The process of fulling with acids has not made great headway, and it is now declared by H. Richard and others that it should be entirely abandoned. The defects in the process leading to this conclusion are, the necessity of cleansing the goods before fulling, far more carefully than when soap is used, the tendency of white goods to assume a dirty yellow cast, and the injurious effect on the health of the workmen. It is also stated that in material fulled with acid, the felt is spongy and coarse, and the goods possess a raw, translucent, and unfinished appearance. The process is at the best only suited for blankets and goods of inferior quality.—R. B. B.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Wool and Cotton, The Absorption of Magnesium and Aluminium Chlorides by. F. Breinl and C. Hanofsky. Chem. Zeit. 21, 563—564.

In a previous paper on the "carbonisation" of wool (this Journal, 1893, 36—37), the authors have shown that the hydrolising action of aluminium and magnesium chlorides on the vegetable impurities accompanying wool is due to the free acid liberated from these salts during the process of drying. They have now made the further observation that during the carbonising process, the salts absorbed by cotton or other vegetable bodies only are decomposed, whilst pure wool that has been impregnated with magnesium chloride does not liberate any hydrochloric acid when heated to 140°—150° C., and with neutral aluminium chloride, traces only, if any.

This conclusion has been called in question by Knecht, Rawson, and Loewenthal in their "Handbook on Dyeing," who suggest as an explanation that the absorbent power of wool for these salts in cold solutions is probably very slight, and hence only small quantities of acid are liberated during the subsequent drying process.

The authors have now tested this theory by a series of experiments on the relative absorbent powers of wool and cotton for these salts. Their results show that under like conditions wool and cotton absorb, even from cold solutions, almost identical quantities of aluminium and magnesium chlorides.

They have found, however, that an excess of hydrochloric acid is always present in the wool over that necessary to form aluminium chloride, and consequently over the alumina also absorbed, which leads the observers to the conclusion that wool does not absorb equivalent quantities of base and acid from solutions of aluminium salts.—I. S.

Albumin, A New Preparation of. Compagnie Parisienne de Couleurs d'Aniline. Fr. Pat. 256,981, 1896.

Four kilos. of the white of eggs are mixed with 25 grms. of 40 per cent. formaldehyde, and after allowing the mixture to rest for several days, water is added, and the formaldehyde is expelled by boiling. The mass is then filtered, and the water evaporated in a vacuum pan at a low temperature to dryness. The new product is precipitated by acids and alcohol, is redissolved by water, but is not precipitated by alkalis. The product is said to possess all other prominent characteristics of albumin.—I. S.

Insoluble Azo Dyes on Wool. M. Goldovsky. Färber Zeit. 8, 184.

Paranitraniline Red and the other insoluble azo dyes have hitherto found no application in the dyeing of wool, since the alkaline solutions used, act injuriously upon the fibre. The author declares, however, that these colours can be applied, without injury to wool, by either of the two following methods:—

(*a.*) The wool is dyed yellow in a hydrochloric acid solution of paranitraniline, and diazotised in the usual manner; it thus acquires a dull brownish-yellow tint. The diazotised wool is then passed into a solution of β-naphthol in caustic soda, after which it is washed in acidulated water and finally in ordinary water.

(*b.*) The wool is worked in a solution of diazotised paranitraniline, washed, developed in β-naphthol, and twice washed, as in the former process.

The author states that useful fast colours are thus obtained with paranitraniline, metanitraniline, dianisidine, &c.—R. B. B.

Commercial Egg Albumin, Estimation of. P. Carles. J. Pharm. Chim. 1897, 6, [3], 102.

See under XXIII., *page* 767.

PATENTS.

Mixed or Parti-coloured Fabrics, Impts. in the Manufacture of. H. Giesler, Molsheim, Germany. Eng. Pat. 28,376, June 3, 1896.

"The manufacture of fabrics having the effects which have hitherto been produced in textile fabrics by weaving with coloured, parti-coloured, or (*vigoureux*) 'mixed,' or fancy threads; the main feature of the said manufacture consisting in impressing or printing a design on the fabric, and then distorting the fibres by displacing, turning, or twisting them so that the sharp lines of the pattern are

destroyed, and a fabric having the appearance of a mixed woven fabric, or one formed from fibres dyed with various colours before weaving, is produced."—I. S.

Hair or Fur, Impts. in Dyeing. M. L. Kellogg, New York. Eng. Pat. 11,831, June 18, 1897.

Human hair, or the fur of animals, is dyed by alternately treating the same with "an aqueous solution of an ammonio-salt of metal," preferably "ammonio-nitrate of nickel," and a suitable mordant, such as pyrogallic acid (pyrogallol).—I. S.

Vessels [Steam Jacketed] for Dyeing Purposes, Impts. in and connected with. B. Lee, Leeds. Eng. Pat. 20,846, Sept. 21, 1896.

The improved vessel is made of iron, and is provided with a steam jacket in which the heat of the exhaust steam from an engine is to be utilised. The vessel may be lined with lead "or other suitable material."—I. S.

Printing Multicoloured Designs upon Fabrics and Warp Chains, Process and Apparatus for. C. Dratz, Brussels. Eng. Pat. 17,692, Aug. 10, 1896.

The essential feature of this invention is a printing roller, "constituted preferably by a cylinder of wood or metal upon which slides a sleeve carrying the design to be printed, made up of a great number of small blocks of solid colouring matter, conveniently arranged, in order to realise a composition in a manner analogous to type-setting," driven at a convenient speed, and pressing upon the wetted warp threads in the act of weaving.—I. S.

Dyeing or Colouring and Sizing of Yarn, and in Apparatus therefor; Impts. in or connected with the. J. Tattersall, Enschede, Netherlands. Eng. Pat. 21,765, Oct. 1, 1896.

When yarn has to be both sized and dyed, the colouring matter is generally added to the size. This method has the disadvantage that when a different colour is required, the machine has to be carefully cleaned, and the remainder of the coloured size has to be thrown away, inasmuch as it cannot be preserved for future use. To remedy these defects the inventor inserts a separate colour trough between the size or sow box and the drying cylinder, provided with a pair of squeezing rollers, the lower of which is partially immersed in the dye-bath. When yarn has only to be sized and not coloured, the top roller of the dye-box is raised, so that the yarn can pass freely between the top and bottom rollers without receiving any colouring matter.—I. S.

Mordanting Agent and Process for Vegetable Fibres, An Improved. [Antimony Lactate.] C. H. Boehringer Sohn, Niederingelheim on the Rhine, Germany. Eng. Pat. 22,522, Oct. 10, 1896.

The inventor has observed that antimony lactate, produced by the direct combination of antimony oxide and lactic acid, remains stable only in the presence of a large excess of acid. This lactate is unsuitable for the mordanting of cloth prepared with tannin, because from the strongly acid solution only small quantities of antimony oxide are precipitated on the fibre. By the following method a double lactate of antimony, suitable for mordanting without the addition of acid, may be prepared, which yields the whole of its antimony to the tannic acid on the fibre:—

One molecule of antimony oxide is digested with the aid of heat with two molecules of "acid lactate of calcium," which produces "a clear solution of antimonic oxide." This solution is used in the usual way on the tannin-prepared cloth.—I. S.

Dyeing of Combing Ribbons or Slivers or the like, Impts. relating to the Method and Machinery for Partial or Complete. F. Desurmont, Tourcoing, France. Eng. Pat. 13,712, June 3, 1897.

The sliver is wound on to a reel of special construction, provided with transverse rods and plates at stated distances. Plates are placed on the top of the wound-up sliver opposite to the plates underneath it, and secured to the latter by means of bolts. The reel is then introduced into the dye liquor and revolved until the wool is dyed of the required shade. The parts compressed between the plates will then remain undyed.—I. S.

Myrabolams, Obtaining Extracts therefrom; Impts. in or relating to Machinery or Apparatus for Treating. S. Smithson, Heckmondwike. Eng. Pat. 12,399, May 19, 1897.

A revolving tank fitted with agitating plates and steam pipes, for the purpose of extracting myrabolams and like materials of a hard gummy nature.—C. O. W.

VII.—ACIDS, ALKALIS, AND SALTS.

Caustic Potash and Soda, Commercial, Purified by Alcohol. [Analysis.] E. Murmann. Zeits. Anal. Chem. **36**, 379–380.

Commercial potassium or sodium hydrate purified by alcohol still usually contains small quantities of copper, lead, and iron, as well as alumina and silica.

Hydrogen sulphide removes them easily. To the greater portion of the warm solution sulphuretted hydrogen water is added so long as the colour darkens, but avoiding excess. A portion is drawn from time to time through a hardened filter until the filtrate is no longer coloured brown by either hydrogen sulphide or lead solution. Any slight excess of sulphide is removed by adding the remainder of the alkali solution, or rendered inert by hydrogen peroxide. The remaining impurities consist of a little silica and alumina.
—L. J. de W.

Disulphuric Acid, Colour Reaction for. E. Barral. J. Pharm. Chim. 1897, **6**, [3], 104.

See under XXIII., *page* 762.

PATENTS.

Condensing Nitrous Acid, or other Fumes or Vapour; Impts. in Means or Apparatus for. J. L. Dobell, Harlesden, Middlesex. Eng. Pat. 18,137, Aug. 15, 1896.

A vertical tubular standard or chamber, enlarged into a well below, into which a depending partition dips, acting as a seal,—has apertures on the side, one immediately above the well, and the other near the top of the chamber, from which large pipes pass, of which the lower one slopes upwards to enter the bottom of a covered-feed chamber, while the upper pipe enters the side of the same chamber, so that vapours entering a slightly inclined injector traversing the standard and projecting into the lower tube, are carried through the feed chamber and then through the upper pipe, into the upper part of the standard, which is provided at the top with an exit pipe for carrying off any uncondensed vapours. The two long pipes are charged from the feed chamber with broken pumice stone, and as the upper tube is slightly inclined, and the lower tube much more so, the liquid of condensation flows into the well. If nitrous acid fumes are to be recovered as nitric acid, air and steam are injected with the fumes. Several of the condensing apparatus are shown connected to form a series.—E. S.

Bleaching Powder, Impts. in the Treatment of [by Compression], to preserve it from Loss of Active Chlorine. J. Y. Johnson, London. From Chemische Fabrik Elektron "Actien Gesellschaft," Frankfort-on-Main, Germany. Eng. Pat. 19,222, Aug. 31, 1896.

Bleaching powder is compressed sufficiently to occupy a bulk approaching to, but not less than that of an equal weight of water. Bleaching powder thus treated may be packed in drums made of thin sheet metal, is not appreciably liable to change, and admits of ready breaking up into a powder for use. Higher compression than that stated is objectionable. Reference is made to Eng. Pat. 5673, 1886 (this Journal, 1887, 367).—E. S.

Utilising the Residual Products [Caustic Alkali Solution of Zinc] obtained in the working of certain kinds of Voltaic Batteries, Impts. in. W. Walker, jr., and F. R. Wilkins, Birmingham, Jabez Jones and Joseph Jones, Smethwick. Eng. Pat. 19,308, Sept. 1, 1896.

See under XI. A., *page* 745.

Cyanides, Improved Means for Manufacturing and Recovering [from Coal-Gas], and Apparatus therefor. G. P. Lewis, Catford, Kent, and R. A. Cripps, Hayward's Heath, Sussex. Eng. Pat. 20,883, Sept. 21, 1896.

The gas to be treated enters a rectangular casing, containing several superposed compartments, at the bottom, and is led by a described arrangement through a series of inverted troughs, having serrated edges, so as to bubble up through a washing liquor supplied to the top compartment, and overflowing successively into each compartment below, until it is led into a receptacle from the bottom. The gas traverses the troughs in each compartment, and is led away from the top. The washing liquid in each compartment is kept agitated by a revolving screw propeller. When it is desired to recover the cyanogen of the gas as a sulphocyanide, solution of an alkali or alkali-earth hydrate is sulphuretted, either by passing through it hydrogen sulphide, or by addition of the liquid condensed from the "waste gases" in the manufacture of ammonium salts, and is used as the washing liquor, after addition of flowers of sulphur and exposure to the air. A small proportion of oxygen in the gas to be thus treated, is desirable. To obtain ferrocyanides, iron sulphide is suspended in the alkaline washing liquor. Prussian blue is obtained by using crude gas liquor with iron sulphide in suspension; the ammonium ferrocyanide solution obtained, is run through ammonia stills and treated with lime, and the effluent containing calcium ferrocyanide is treated for the recovery of Prussian blue by the method described in Eng. Pat. 5184, 1896 (this Journal, 1896, 239).—E. S.

Baking Powders, Impts. in. F. Dietrich, Riegel, Germany. Eng. Pat. 20,891, Sept. 21, 1896.

Acid ammonium phosphate ($NH_4H_2PO_4$) is substituted, wholly or in part, for acid potassium tartrate in baking powders; and one or more "retarding substances," such as calcium carbonate, magnesium carbonate, or dicalcium hydrogen phosphate, are introduced, to prevent premature or too rapid evolution of carbonic acid. In one of the examples given, stated proportions are used of acid ammonium phosphate, sodium bicarbonate, calcium carbonate, or magnesium carbonate, and of a filling substance, such as corn starch. Generally stated, the baking powder consists, "in the main, of pure crystallised acid ammonium phosphate and sodium bicarbonate, to which distinctly characterised acid or basic substances producing carbonic acid are still added, or both substances together."—E. S.

Copper Sulphate, Impts. in the Production of. T. Savage, Birmingham. Eng. Pat. 21,953, Oct. 3, 1896.

Copper is treated in a tank heated by steam pipes, with sulphuric acid of sp. gr. 1·20, to which latter there is added about half its weight of sodium nitrate. The solution of the copper is said to be effected much more rapidly than when dilute sulphuric acid alone is employed, as in the ordinary process. When the solution is saturated with copper, it is taken to a lead-lined vat having lead hangers, for crystallisation.—E. S.

Double Chloride of Aluminium and Soda, A New Process of Manufacture of. The Compagnie Générale L'Alumine Exploitation des Brevets F. Raynaud, Société Anonyme, Brussels, Belgium. Eng. Pat. 12,266, May 18, 1897. (Under Internat. Convention.)

A mixture of ferruginous bauxite, sodium chloride, and carbon is moulded into porous blocks, which are heated to redness in suitable retorts, through which hydrogen sulphide is passed. When the sulphuration is complete, a current of air is substituted for that of the hydrogen sulphide, when sodium-aluminium chloride distils over and is collected. The processes are repeated in the same order as often as may be necessary to exhaust the ore. The iron is sulphuretted in the first, and oxidised in the second process. The sodium sulphate formed undergoes slight reduction.—E. S.

Ammonium Bichromate, A New Process for the Production of. W. Majert, Grünau, Germany. Eng. Pat. 13,551, June 1, 1897.

Molecular proportions of ammonium chloride and of sodium bichromate or calcium bichromate are dissolved in three parts of hot water; ammonium bichromate separates on cooling, and may be recovered by centrifugal action. According to a second process, solutions of calcium bichromate and of ammonium sulphate, each in 5 parts of hot water, and in equivalents, are mixed, and the mixture is boiled until calcium sulphate separates in a crystalline form. Ammonium bichromate is then crystallised out of the concentrated filtered solution; or, for a less pure salt, the latter may be dried.—E. S.

VIII.—GLASS, POTTERY, ENAMELS.

PATENT.

Gas Furnaces and Muffles, Combined, Self-Producing, for Use in Enamelling Metallic Articles; Impts. in. T. and A. Preece, Wolverhampton. Eng. Pat. 19,800, Sept. 8, 1896.

The gas from an ordinary producer-furnace is passed over a brick arch into a flue running under the muffle (which is one designed for use in enamelling metallic articles), and opening at one side into a number of lateral passages leading, *viâ* a series of upright flues, into an arched flue over the muffle. This arched space is constricted on the opposite side to prevent the gases from escaping too freely through the descending flues there situated, and leading to the chimney. To secure equalisation of heat in the bottom flue, the lateral openings decrease in size towards the furnace end of the muffle. A series of air-flues keeps the firebrick arch and the bricks at the bottom and sides of the gas flues cool, the heated air passing into the arched flue and there producing intense combustion.—C. S.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Tiles and Brickwork, Efflorescence on. Dingler's Polyt. J. 1897, **305**, 8.

An investigation has recently been carried out by Hans Günther (Baumaterialienkunde, **24** and **25**, 385) into the cause and prevention of the dry efflorescence sometimes seen upon tiles and brickwork. This is usually ascribed to the formation of sulphates in the bricks or tiles, owing to the presence of sulphuric acid in the kiln gases. Sulphur dioxide, produced by the combustion of the pyrites in the coal, oxidises to sulphuric acid, and this attacks the bases present in the clay, more especially if lime be present in considerable quantity. Whether, as some suppose, this can take place only in the presence of water vapour, and therefore only during the smoky and incomplete stage of combustion, or whether it can also take place at a red heat, has been a disputed point. One thing, however, is certain, namely, that sulphuric acid cannot be formed in the presence of reducing gases; and this probably explains the superiority of the old intermittent kiln, in which there was frequently a deficiency of air, over the modern continuous ring-kiln, in which the air is always in excess.

The following experiments were made:—Samples of felspar, kaolin, and clay marl were heated to redness in air containing sulphur dioxide. The felspar was not affected at all, but both the kaolin and clay were attacked, with formation of calcium, sodium, and other sulphates. Further experiments, made with mixtures of clay and chalk, showed that the greater the proportion of lime, the larger was the quantity of sulphates formed. The author considers this proves that moisture is not essential to the formation of sulphates by the kiln gases, and that therefore their formation is not restricted to the smoky part of the kiln. Magnesia, when present in the clay, is also converted into sulphate, but, whereas calcium sulphate first reacts upon the alkaline salts in the mortar, magnesium sulphate effloresces unchanged on the surface of the bricks.

The author, however, believes the efflorescence to be due more to the pyrites in the clay itself than to that present in the fuel. Of 21 samples of brick and tile clays of different descriptions which were examined, every one

proved to contain pyrites. Further, quite considerable quantities of sulphates are formed when certain clays are burnt in air absolutely free from sulphur dioxide. Cubes made from clay containing 0·11 per cent. of SO_3 were found, after heating to redness for seven hours in an atmosphere free from sulphur dioxide, to contain as much as 0·69 per cent.

In order to ascertain the behaviour of certain salts when ignited with clay, the following experiments were undertaken:—Six portions of clay were taken, and each mixed, in uniform proportions, with one of the following salts, *viz.*, (1) Na_2SO_4, (2) K_2SO_4, (3) NaCl, (4) KCl, (5) $BaCl_2$, and (6) Na_2CO_3. The several portions were shaped into tiles and burnt, the SO_3 being determined both before and after burning. In (1) and (2) the SO_3 diminished from 4·65 and 3·75 per cent. to 0·83 and 0·88 per cent. respectively; in (3) and (5) the chlorine disappeared altogether; and in (6) 0·16 per cent. of SO_3 was found after burning.

In conclusion, the author recommends when the continuous method of burning is employed, that barium chloride and carbonate should be mixed with the clay used, to guard against soluble sulphates being formed.—H. H. B. S.

PATENTS.

Non-burnt Sand Brick. A. Easterbrook, St. Austell. From T. T. Wood, St. Joseph City, Michigan, U.S. Eng. Pat. 17,635, Aug. 10, 1896.

The inventor claims an unburnt brick made from sand, cement, soluble silica, hydrochloric acid, and water in specified proportions.—V. C.

Artificial Stone, Impts. in. A. McLean, London. Eng. Pat. 19,419, Sept. 2, 1896.

In the preparation of paving stones from natural flagstone (which has a laminar structure), a large quantity of "waste" is produced. The inferior qualities of the stone are also practically waste. The inventor grinds these "waste" materials finely, and mixes with Portland cement, with the addition of water in the usual way. The concrete thus formed is pressed in moulds, making an artificial paving or "flagstone."—V. C.

Cements for Resisting the Action of Acids, Acid Vapours, and Heat; Impts. in. J. C. Rombach and E. S. Restieaux, both of Melincrethan, Neath, Glamorganshire. Eng. Pat. 12,024, May 15, 1897.

A mixture of silicate of soda and asbestos, to form a cement by dilution with water, hydrochloric acid or other acid solution being used as a finishing wash or fixing agent. —V. C.

Enamels or Cements, Process of Manufacture of Agglutinant. B. J. B. Mills, London. From La Granitine Soc. Anonyme, Brussels. Eng. Pat. 15,336, Sept. 2, 1897.

A solution of silicate of soda is prepared having a density of 1° to 2° B. In this, chloride of magnesium is dissolved until the density reaches 31° to 32° B., then solution of sulphate of alumina is added to bring it up to 32° or 33° B. Finally, 55 per cent. of this liquid is mixed with 45 per cent. of calcined magnesite, forming a cement which "not only constitutes on the surface of ceramic products a most brilliant enamel, but also a powerful element of cohesion of foreign substances with which it is mixed."—V. C.

X.—METALLURGY.

Pyritic [Copper] Smelting, The Present Mode of. H. Lang. Eng. and Mining J. 1897, **64**, 37—38.

The author comes to the following conclusions from the experience of the past two or three years.

Charges containing no more than one-third of their weight of sulphides can be smelted with as little as $2\frac{1}{2}$ per cent. of coke and with a very high concentration. The slags made in pyritic smelting are as free from copper as those from ordinary matting, and those which have iron as almost the only base, are as free from copper, gold, and silver as those containing lime or other base. The proportion of silica in the charge undergoing fusion, influences the concentration of pyritous ores to a great extent; the charge should contain as much silica as the furnace will stand for the heat necessary to fuse the material, and to induce the separation of the iron.—A. S.

Gold Field, The Witwatersrand. W. Y. Campbell. Eng. and Mining J. 1897, **64**, 36.

The country rock on the Witwatersrand is sandstone, quartzite and shale, and the seams, which dip south at angles of from 15°—85° from the horizon, vary from knife-edge selvages of black ferruginous sand, rich in gold, up to solid conglomerate seams of 10 and 15 ft. The strike of the seams is mainly east and west, and the average thickness is 3 in. on the east sections and 2—4 ft. on the Central Rand. The gold is practically all in the cement coating binding the pebbles, and is very seldom indeed found in the pebbles themselves or in the country rock. It nearly always occurs in very fine crystals and in mechanical combination with the cement or pyrites. Greenish diamonds are occasionally found in the mortar boxes. The line of oxidation is erratic, oxidised ore being sometimes found below unoxidised at some depth; the average depth is about 200 ft.—A. S.

Cyanide Process [Gold], Prof. Christy on the: A Review. W. Bettel. South African Min. J., May 8, 1897.

This is a criticism of Christy's paper on the Solution and Precipitation of the Cyanide of Gold (this Journal, 1897, 332). The Elsner equation is deemed insufficient, as an intermediary reaction producing H_2O_2 or an equivalent substance, has been observed. Bodlaender, of Clausthal, has represented the two stages thus:—

$$\text{I. } Au_2 + 4KCy + 2H_2O + O_2 = 2KAuCy_2 + 2KHO + H_2O_2.$$

$$\text{II. } 4KCy + H_2O_2 + Au_2 = 2KAuCy_2 + 2KHO.$$

Bettel suggests the following:—

$$\text{I. } 6KCy + Au_2 + O_2 + 2H_2O = KAuCy_2 + KAuCy_4 + 4KHO.$$

$$\text{II. } KAuCy_4 + Au_2 + 2KCy = 3KAuCy_2.$$

MacArthur asserted that gold will dissolve in cyanide solutions in the absence of free oxygen, and this is possible in the presence of basic ferric sulphate, which forms potassium ferricyanide with the cyanide. Free cyanogen in the presence of cyanide solution is almost without action on metallic gold. The addition of the oxidising agents covered by Julian's patents has been proved of no value to the industry; and it is not true to assert that potassium chlorate, nitrate, and bichromate are of assistance in promoting the solution of gold, as they are too stable in alkaline solution to produce any such action. The author's experiments do not corroborate the statement that nearly all the oxidising agents are found to be effective. In regard to the solubility of gold sulphide in potassium cyanide, it is pointed out that the presence of silver in the gold would greatly impede or even prevent the action by the formation of a film of insoluble silver sulphide. The author has also shown previously that the presence of soluble sulphides in the fully aërated cyanide solution prevents the solution of metallic gold in it, unless oxidants or some substance such as lead hydroxide or carbonate be added. The regeneration of the cyanide solution proposed by Christy has already been patented by Adair in the Transvaal, but although successful in the laboratory, the process was found by Adair to be unsatisfactory on the large scale. The author considers that de Wilde's cuprous precipitation method has not the elements of a successful commercial process. In regard to the recovery of cyanide of potassium from strong solutions by zinc sulphate, Haut's method was anticipated by the author in 1893, but his patent has not been worked, owing to the distinct solubility of zinc cyanide in water, especially in the presence of hydrocyanic acid. The principle now adopted by Bettel consists in filtering the zinc-treated solution, after slight acidification, through a filter-bed of bright iron turnings or spongy iron, followed by filtration through magnesian limestone. The spongy iron removes all traces of gold from the solution. The statement that

gold cannot be precipitated by zinc shavings unless 0·1 to 0·2 per cent. of free cyanide be present, is disproved by daily practice on the Rand, where cyanide solutions containing only 0·04 per cent. of free KCy are reduced from 3 or 4 dwt. to as many grains per ton. Finally, the author ridicules as thoroughly unpractical and prohibitive as to cost, the proposal to acidify solutions containing calcium hydroxide.—W. G. M.

Slimes, Zinc-Gold: Reduction of. E. H. Johnson. Proc. Chem. and Metall. Soc. of S. Africa, for June 19, 1897, 12—15.

At the Princess Works, two 6-ft. clean-up vats have to be used in default of a filter press. One of these is fitted with a false bottom, having holes 1 in. square, covered with fine, closely-woven canvas. Below the partition, a 1¼-in. pipe is passed through the side of the vat and connected to a small suction pump, which delivers the filtrate into a launder leading to the top of the precipitation boxes. The zinc-gold slimes are bucketed into the filter vat, and are there filtered and washed with the aid of the pump. The filtration is slow, but must not be hurried for fear of bursting the canvas. When free from cyanide, the moist slimes are weighed in buckets as they are transferred to a large sheet-iron tray placed alongside the acid tank. The second vat is used for acid treatment, and is fitted with a detachable wooden stirring apparatus and a cover provided with a 3-in. pipe to lead fumes to the outside of the building. There is also a feed hopper, with sheet-iron valve below and cover above, to enable the charges of slime to be introduced without escape of fume. It is found best to use 1 lb. of strong sulphuric acid per pound of moist slime (equivalent to 1⅓ lb. of acid per pound of slime, dry). Sufficient water to form a 10 per cent. solution of the acid is first placed in the vat, the sulphuric acid is then mixed in, and finally the slimes are introduced with constant stirring, and the hopper and buckets are rinsed with a jet of water. The agitation is maintained for half an hour after action has apparently ceased. The vat is filled up with water, and the contents are allowed to settle. Working with dilute acid and without heating, perfect settlement results within an hour. The use of a steam jet renders the settlement more difficult. The deposit is then washed until neutral, by (usually four or five) successive washings, the water being well stirred each time by a paddle, and afterwards siphoned off. An average running sample of the washings assayed 13 grs. per ton. The slime is dried in large enamelled cast-iron dishes, the cakes are next broken up and heated to a higher temperature in thin layers on small sheet-iron trays; and, when cool, are ground, fluxed, and transferred to a crucible. The substance fuses quietly and with little fume, yielding 50—60 per cent. of the weight of slime as bullion. The average fineness of last year's bullion was about 820, and the slag, after panning out a few prills, contained 23 oz. per ton. 1 ton of such slag accompanied the production of 11,627 oz. of gold in the space of two years, which works out to 0·2 per cent. of the gold being locked up in the slags. From the following cost of a typical "clean-up," it is seen that the cost of reduction is 6·7*d.* per fine oz :—

Dry weight of zinc-gold slimes	504 lb.
„ after acid treatment	100 „
Yield of fine gold	620 oz.

	£	s.	d.
Cost of 672 lb. of acid @ 4½*d.*	12	12	10
„ 66 lb. of borax @ 37*s.* 6*d.* per cwt.	1	2	1
„ 9 lb. of sodium carbonate @ 2½*d.* per lb.	0	1	10
„ 9 lb. of fluorspar @ 4*d.* per lb.	0	3	0
„ 5 bags of coke @ 8*s.* 6*d.* per bag	2	2	6
„ 1 No. 60 crucible	1	7	6
	17	9	9

The cost of the acid may render this process slightly more expensive than the calcination method, but the immunity from unaccountable losses and the small amount of gold left in the slags more than compensates for this.
—W. G. M.

Nickel, The Fluidity of Melted. J. Garnier. Comptes Rend. 1897, **124**, 1447—1448.

At a meeting of the *Iron and Steel Institute* in 1891, the author propounded the view that the strengthening effect on steel, of small quantities of nickel is due to the extreme fluidity of the melted nickel, which enables it to penetrate into and fill the intermolecular interstices of the less fluid steel. He has obtained confirmation of this view from the accident of a piece of wood-charcoal becoming fixed near the lip of a crucible which was used for the reduction of nickel in the works of the Canadian Copper Co., Cleveland, Ohio. The vessels of the wood were found to be penetrated by the fluid nickel, which was obtained, after breaking up the charcoal, in the form of hair-like threads of extreme tenuity, very flexible and malleable.—J. T. D.

Nickel Refining, Modern American. T. Ulke. Zeits. für Elektrochem. 1897, [23], 521—524.

Two distinct methods are in use for the treatment of the matte produced by smelting Canadian nickel-copper ores in water jackets, and averaging 20—25 per cent. of copper, 18—23 per cent. of nickel, 25—35 per cent. of iron, and 20—30 per cent. of sulphur, according as it is desired (1) to obtain an alloy of copper and nickel, and (2) to recover the pure metals separately.

(1.) The matte is concentrated in a Bessemer plant, and the high-grade matte thus produced is roasted and reduced to the copper-nickel alloy. At the Sudbury Works, Ontario, of the Canadian Copper Company, 25 tons of cupola matte are treated and 15 tons of Bessemer matte produced in 24 hours. In the converters, the iron is almost entirely removed, the sulphur reduced to from 12—15 per cent., and the copper and nickel increased to about 43 per cent. and 40 per cent. respectively. The converter slags average 2 per cent. of copper and 3½ per cent. of nickel, and are returned to the cupolas for remelting. The exact point at which the blast should be increased, after the iron is removed and before the nickel is reduced in quantity to slag, can only be fixed by long experience.

The average composition of the Bessemer matte is 43·36 per cent. of copper, 39·96 per cent. of nickel, 0·3 per cent. of iron, 13·76 per cent. of sulphur, 7 oz. per ton of silver, 0·1—0·2 oz. per ton of gold, and 0·5 oz. per ton of platinum and palladium. The alloy produced contains about 51 per cent. of copper and 48 per cent. of nickel, and is used in the manufacture of German silver. The cost of Bessemerising the ordinary matte is stated to be 12—15 dols. per ton.

The author proposed in a previous article (Eng. and Mining J., Jan. 30, 1897) to refine Bessemer matte by dissolving it in dilute sulphuric acid, removing the copper by an electric current of low voltage, and then increasing the voltage so as to deposit the nickel free from copper, the precious metals being recovered from the slimes.

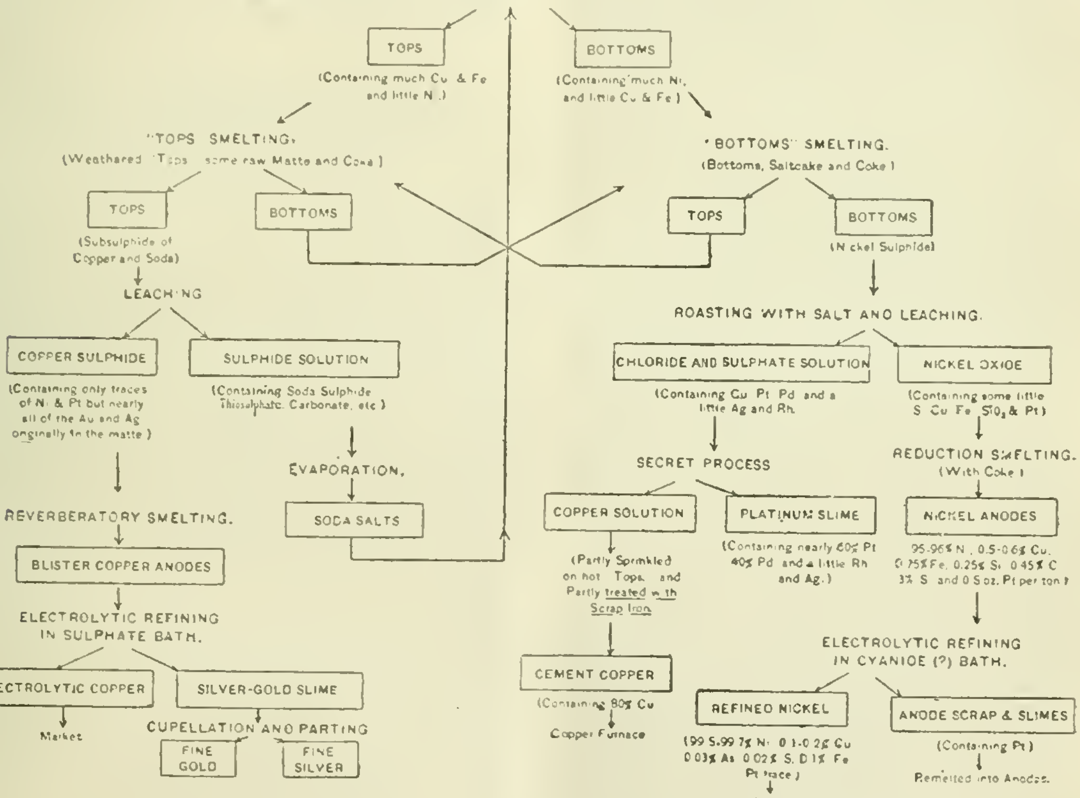
(2.) When it is desired to obtain nickel oxide or metallic nickel, the ordinary matte is smelted in a cupola with saltcake and coke. In this way the saltcake is reduced to sodium sulphide, which unites with the greater part of the iron and copper to form so-called "tops," and is tapped, together with the more nickeliferous "bottoms" into pots and allowed to cool. The tops are then separated by hand from the heavier bottoms. The weathered tops from the above process are subjected to what is termed top smelting, *i.e.*, they are smelted, together with a little matte and sufficient coke, in a second cupola, so as to again produce tops and bottoms. In this process, the soda in the tops takes up some sulphur from the nickel in the matte and forms a sulphide, which unites with part of the iron and copper.

In bottom smelting, the bottoms from the first and second cupolas are mixed with saltcake and coke in a third cupola, in which tops and final bottoms—composed of highly nickeliferous sulphide—are produced. The latter is roasted with salt in a reverberatory furnace and heated to a temperature at which as much nickel oxide as possible is formed, together with the chlorides and sulphates of copper and the precious metals. The roasted mass is then leached to separate the insoluble nickel oxide from the chloride and sulphate solution. The latter contains nearly all of the copper, silver, and palladium in the roasted bottoms.

Formerly, a cement copper was produced, containing 80 per cent. of copper with 60 oz. of platinum and 40 oz. of palladium per ton, by placing iron plates directly in the solution; but now the precious metals are precipitated out by a new method, and the residual solution is then sprinkled on hot tops or treated with scrap iron to produce cement copper. The leached nickel oxide is reduced with coke (at the Orford Works, near Constable's Hook, N.J.) to crude nickel, which is cast into anodes containing 95—96 per cent. of nickel, 0·2—0·6 per cent. of copper, 0·75 per cent. of iron, 0·25 per cent. of silicon, 0·45 per cent. of carbon, 3 per cent. of sulphur, and 0·5 oz. per ton of platinum. These anodes are electrolytically refined, a cyanide bath being probably employed, and refined nickel is finally obtained containing 99·5—99·7 per cent. of nickel, 0·1—0·2 per cent. of copper, 0·03 per cent. of arsenic, 0·02 per cent. of sulphur, 0·1 per cent. of iron, and traces of platinum. The anode scrap is remelted into nickel anodes, and the slime treated to recover platinum.

The final tops, consisting chiefly of sub-sulphide of copper and soda, are leached, and the copper sulphide thus obtained as residue contains only traces of nickel and platinum, but nearly all of the gold and silver in the original matte. The solution carries down sodium sulphide, thiosulphate, and carbonate, &c., and the sodium salts obtained on evaporation are added to the smelting charge. The copper sulphide is reduced in reverberatory furnaces to metal and cast into anodes, and the latter are refined electrolytically by the ordinary multiple process in a sulphate bath. The gold-silver slime obtained is added to a lead charge and cupelled down to *doré* bars. The *doré* is parted by sulphuric acid in the ordinary way, and cast into bars of fine silver and fine gold.

Matte, Saltcake and Coke Tops and Bottoms Smelting.



DIAGRAMMATIC SCHEME OF THE ORFORD METHOD OF TREATING NICKEL COPPER MATTE.

—A. S.

Nickel Steel: Physical Properties. Engineer, Aug. 20, 1897, 187.

STEEL alloyed with nickel possesses a property of high importance, in that it does not develop cracks. It would appear that extensive employment of nickel steel will before long be made by boiler makers, for in high-pressure steam boilers a large saving in weight will ensue. For railway purposes, too, this alloy must have a bright future, as it should be specially adapted for locomotive details.

Iron Ores, Notes on the Determination of Insoluble Phosphorus in. C. T. Mixer and H. W. Du Bois. J. Franklin Inst. 1897, **144**, 137.

See under XXIII., *page* 763.

PATENTS.

Sulphide Ores, Treatment of, for the Separation of Zinc from the Lead and other Metals. B. Mohr, Hampstead. Eng. Pat. 13,169, June 15, 1896.

THE pulverised ore is fused with acid sodium or potassium sulphate, and the fused mass is dissolved in hot water, a small quantity of sulphuric acid being added when necessary. From the solution, zinc is deposited by electrolysis, and the liquid is then concentrated by evaporation until it is strong enough to be used for the fusion of a fresh batch of ore. The lead, silver, &c. of the insoluble residue is treated by known methods.—J. H. C.

Sulphide Ores, An Improved Regenerative Process for the Treatment of. E. T. Turner, Adelaide, Sth. Australia. Eng. Pat. 15,749, July 16, 1896.

SULPHIDE ores of zinc and lead containing small proportions of gold, silver, and other metals are placed, without being pulverised, in vats or digesters provided with gas-tight lids and perforated bottoms, arranged in series, and standing

over crucibles which can be heated. The said ores are sprayed from above with aqueous hydrochloric acid from a tank, whilst at the same time gaseous hydrochloric acid is supplied below the perforated bottom from a generator. The ore is gradually disintegrated, and a strong solution of metallic chlorides passes down into the crucibles together with the undissolved portions of ore and gangue. This solution is kept at the boiling point, raised by suitable means, and made to pass through a new portion of ore, until a neutral saturated solution of chlorides is obtained.

The disintegration of the ore is accompanied by the production of sulphuretted hydrogen, which is also made to circulate through the ore under treatment until it finally passes, practically free from hydrochloric acid, into a purifying chamber, and thence to a gasholder, from which it is drawn off to be used as fuel.

As each crucible becomes filled with particles of undecomposed ore and gangue, and with the deposited chlorides of silver, lead, zinc, &c., its vat or digester is disconnected, and it is withdrawn from the series. The temperature of the mass in the crucible is now raised to about 300° C., when a fused mass of chlorides, &c. is produced, which is subsequently lixiviated, and the metals precipitated from the solution.—J. H. C.

Metals and Alloys, An Improved Method for the Production of. C. Vautin, London. Eng. Pat. 16,685, July 28, 1896.

The compounds to be reduced, are finely pulverised and mixed with finely powdered aluminium, or a mixture of aluminium and magnesium; a small portion of a mixture similar to the first, but coming more easily into reaction, is placed on the top, and this is heated so as to start its reaction, which then spreads to the main mass, being maintained by the heat developed by the initial reaction.

For example, to obtain the metal chromium, equivalent parts of oxide of chromium and aluminium are placed in a crucible which has been previously lined with magnesia. Upon the mixture is placed a little cartridge composed of peroxide of barium and aluminium intimately mixed and compressed. To this cartridge a thin piece of magnesium is attached. When this is ignited, it ignites the cartridge and reduces the peroxide of barium, and by this reaction enough heat is produced to ignite the whole mass, similarly reducing the oxide of chromium to metal.—J. H. C.

Solutions of Copper, An Improved Process for the Preparation and Purification of. J. C. Graham, London. Eng. Pat. 17,127, Aug. 1, 1896.

Crude cuprous chloride solutions, containing iron and other impurities, such as the liquors obtained by "lixiviating ores," are treated with lime or an alkali, in such proportion as to precipitate ferric hydroxide, leaving the copper salt undecomposed. The copper is then thrown down from the cleared liquor as hydrated oxide by a further addition of the lime or alkali. The precipitate, after collection, is churned with water through a centrifugal pump, and after settling, the process is repeated to complete the washing. The cuprous hydroxide may then be dissolved by sulphuric acid, and copper electro-deposited from the solution. "If the original impure solution was a sulphate and not a chloride, then the second addition of a base may be dispensed with."—E. S.

Gold and Silver, Impts. in the Recovery of, from their Solutions. W. H. Göpner and H. L. Diehl, Hamburg. Eng. Pat. 17,493, Aug. 7, 1896.

Gold and silver are precipitated from their cyanide solutions by adding cuprous cyanide or silver cyanide or their mixture, and then adding an acid to effect precipitation. The precipitate is dissolved by a fresh quantity of the cyanide solution obtained by leaching, and acid again added so as to effect successive precipitations from the said solution.—J. H. C.

Zinc Ores or Ore Residues; Impts. in and relating to the Treatment of, in the Production of Zinc, Chlorine, Sodium Sulphate, and other Products. H. Brewer. Eng. Pat. 17,839, Aug. 12, 1896.

Sulphide ores are roasted with sodium chloride and then leached, so as to dissolve out the sulphates thus formed. Calcium chloride is then added so as to convert them into chlorides and to regenerate the sodium chloride. The gypsum thus formed is separated, as also any iron present, preferably by means of calcium carbonate, as ferric hydrate. The liquid is then evaporated, and the excess of sodium chloride (and of sodium sulphate) crystallised out. Finally, the solution is electrolysed so as to yield zinc and chlorine.—J. H. C.

Tin Oxide, Treatment of, in the Form of Powder, Sludge, or Paste, for the Smelting thereof. E. E. von der Linde, Crefeld. Eng. Pat. 11,602, May 10, 1897.

The tin oxide, &c. is mixed with a small proportion of caustic alkali or alkaline carbonates, tin slag is added, and then lime or carbonate of lime. Water is added, and the mixture is ground fine so as to produce a tough, pasty mass, which is moulded into briquettes and dried. The following proportions are recommended:—100 parts of tin oxide, 10 parts of slag, 8 parts of lime or carbonate of lime, and 2·5 parts of caustic alkalis or alkaline carbonates.

—J. H. C.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(A.)—ELECTRO-CHEMISTRY.

The Copper Voltameter: Its Advantages and Disadvantages. F. Foerster. Zeits. f. Elektrochem. 1897, 3, 479—482 and 493—497.

The copper voltameter is largely used by chemists, mainly because it is so very handy. The results obtained from it, especially with small currents, are known to be rather uncertain. Investigations made by the author (with O. Seidel) on the electrolysis of copper sulphate solutions show how and why the copper voltameter can be used for accurate work. They show that in the electrolysis of copper sulphate solutions between copper electrodes, the simple transference of copper from the anode to the cathode is not always the only process which goes on. Copper forms two sets of salts, and, during the reactions which go on, cuprous salts may be formed. Hitherto the existence of a cuprous sulphate has not been admitted. The authors have not succeeded in obtaining it in the solid form, but they have no doubt that it can exist in solutions of cupric sulphate, even if only in a dilute state. It is formed at the cathode, especially when the current-density is less than 0·01 ampère per square decimetre. The tendency of cupric ions to become transformed into cuprous ions increases with temperature, so that at 100°, even with a current-density of 0·03 ampère per square decimetre, in concentrated copper sulphate solution the current forms almost exclusively, cuprous ions at the cathode.

As was long ago shown by Jacobi, these ions can even be formed without any current, by the action of copper on cupric ions (*e.g.*, in the case of cupric chloride); thus:—

$$\overset{++}{Cu} + Cu = 2\overset{+}{Cu}.$$

In neutral solutions the cuprous sulphate formed, as soon as its concentration reaches a certain value, suffers hydrolysis; thus:—

$$2\overset{+}{Cu} + \overset{--}{SO_4} + H_2O = Cu_2O + 2\overset{+}{H} + \overset{--}{SO_4}.$$

Cuprous oxide is deposited on the cathode (often in brilliant crystals), and the solution becomes acid. If the solution is acid enough, the hydrolysis does not take place, and more cuprous ions remain than in neutral solution. But as soon as the proportion of these to cupric ions reaches a certain value, they interchange, with deposition of metallic copper—

$$2\overset{+}{Cu} = \overset{++}{Cu} + Cu.$$

This should be regarded as "secondary" copper. It forms separate crystals, not like the usual smooth deposit from acid solutions.

One of the most important points in the use of the copper voltameter is the composition of the electrolyte.

This is generally concentrated, and rightly so: a weak solution is apt to give a pulverulent deposit of copper on the cathode. With regard to the presence and effects of cuprous ions, it must be pointed out that the voltameter is used in presence of air, and on exposure to air, the cuprous ions are oxidised to cupric ions. If the solution is acid, copper sulphate is formed—

$$2\overset{+}{Cu} + 2\overset{+}{H} + 2\overset{--}{SO_4} + O = 2\overset{++}{Cu} + 2SO_4 + H_2O.$$

If it is neutral, basic copper sulphate is formed—

$$2\overset{+}{Cu} + 2\overset{+}{H} + 2\overset{-}{OH} + O + \overset{--}{SO_4} = H_2O + Cu(OH)_2,CuSO_4.$$

In his second paper the author discusses the errors of the copper voltameter, and how they can best be avoided. It has one unavoidable defect, *viz.*, the low electro-chemical equivalent of copper. But when the composition of the electrolyte and the density of the current are suitably chosen, the instrument is a more accurate one than is usually supposed. It is handier and simpler than the silver or mercury voltameter, and will remain the favourite instrument for technical work. For general use an electrolyte containing 125 grms. of crystallised copper sulphate and 50 grms. of pure sulphuric acid (sp. gr. 1·84) per litre is recommended. When small currents and low current-densities have to be used, it is well to add alcohol, as recommended by Oettel, who uses an electrolyte made up of a litre of water, 150 grms. of copper sulphate, 50 grms. of sulphuric acid, and 50 grms. of alcohol. By proper adjustment of the current-density, currents from 0·01 ampère down to 0·005 ampère can be measured with sufficient accuracy. The cathodes are made of copper wires 1 mm. thick, flattened out at the ends. But for very small currents the author prefers to use a closed voltameter in which the electrolyte and electrodes are kept from contact with the air. In this case no alcohol is added to the electrolyte, but hydrogen is allowed to bubble through it before and during the electrolysis. In order to prevent undue variations of current-density, a number of cathodes of varying size should be kept ready, the smallest being made by flattening out the ends of platinum wires 0·5 mm thick. When large currents are measured, it is well not to allow the current-density to exceed 2 ampères per square decimetre. Above this the copper is apt to be deposited on the cathode in a loose powdery form.

—D. E. J.

Electrolysis of Mixtures. A. Schrader. Zeits. f. Elektrochem. 1897, **3**, 498—505.

When a mixture of aqueous solutions of copper sulphate and sulphuric acid is electrolysed, hydrogen and copper are generally set free at the cathode, but sometimes copper alone. According to Hittorf's views as to the nature of electrolytic processes, the current traverses the two dissolved electrolytes in proportion to their respective conducting powers in the mixture. Primarily, hydrogen and copper are set free at the cathode, but by a secondary reaction, a part of the primary hydrogen may deposit more copper. The author has endeavoured to determine this part under various conditions. The investigation naturally falls under two heads:—(A) The determination of the ratio x, in which the current divides between the two electrolytes. Upon this can be based (B) the actual experiments upon the question above stated. (A) has an interest apart from (B), and from the purely theoretical point of view is perhaps the more important question. The most important experimental results are the following:—(1.) By translation experiments the author has determined the ratio in which the current divides between the components of two mixtures, *viz.*, (*a*) aqueous solutions of potassium chloride and potassium iodide, (*b*) aqueous solutions of sulphuric acid and copper sulphate. The results obtained are used to calculate the change of the degree of dissociation of the components produced by mixing. (2.) The translation-number of acetic acid approximates with increasing dilution to the theoretical value $N = 0·119$; but even with a dilution of $V = 4$ this value is not quite attained. (V is the number of litres which contain a gramme-molecule of $CH_3.COOH$. The "translation-numbers" are those of the anions of the components in accordance with Hittorf's definition; so that if these be N and N_2 the translation-numbers of the cations are $1 - N_1$ and $1 - N_2$.) (3.) From the results of (1) the author determines what fraction of the primary hydrogen goes to deposit copper (in the case of mixed solutions of $CuSO_4$ and H_2SO_4). Within the limits of experimental error the fraction σ can be expressed by the formula—

$$\sigma = \frac{A}{C + B}$$

when C is the current-density and A and B are constants. For sufficiently dilute solutions, B appears to be equal to A, so that the formula takes the simpler form—

$$\sigma = \frac{A}{C + A}$$

—D. E. J.

Luckow's Method for Electrolytic Production of Insoluble Oxides and Salts from Metallic Anodes. Zeits. f. Elektrochem. 1897, **3**, 482—485.

With regard to the Luckow patents, it has long been known that lead peroxide is formed on lead anodes in certain solutions, and white lead, chrome yellow, &c., in other solutions. But although these problems have apparently been solved, everyone who has tried to apply them technically knows how very difficult it is to carry out the methods in practice. Hitherto there has been no electrolytic method which would produce upon the anodes products of sufficiently approximate purity for commercial purposes. Even lead peroxide thus prepared contains 20 per cent. or more of impurity. Tibbits' process apparently resembles Luckow's, but owing to the high concentration of the electrolyte or some other cause, the white-lead produced by it is neither pure nor cheap enough. In fact, we have not, until now, had any technically useful process for the direct electrolytic production of lead peroxide, white lead, chrome yellow, zinc white, or similar products.

The essential point of Luckow's discovery is in the establishment of the following fact:—When the electrolyte is diluted so far that its electrolytic dissociation reaches as high a degree as will still admit of good conductivity (what may be called its practical maximum), the desired products are obtained in a state of purity hitherto unattained. In addition to the actual electrolyte, there is generally present a second salt. The ionisation of the first is of primary importance; it brings the anode-metal into solution. The *rôle* of the second is to convert the dissolved metal into the desired compound. Both should be highly diluted. For the preparation of insoluble salts, a solution is used in which the primarily active electrolyte (which dissolves the anode) is to the secondary electrolyte (which causes the precipitation) in the proportion of 80 to 20. The end-product does not adhere firmly to the anode; indeed, it is formed at a slight distance from it. It is found at the bottom of the bath in the form of a finely divided but dense powder of great covering power. When oxides are required, a strongly diluted solution is used of a salt, the oxide of which does not form with the anode any soluble salt; to this is added quite a small quantity (0·5 per cent. of the solid constituents) of a salt the anion of which forms with the anode-metal a soluble compound. It is convenient to use as cathodes, plates of the same metal which serves as the anode. The solution is generally kept neutral; in many cases this is best attained by the addition of a salt, which easily suffers hydrolytic dissociation.

Preparation of Lead Peroxide.—The electrolyte is a 1·5 per cent. aqueous solution of a mixture of 99·5 parts of sodium sulphate with 0·5 part of sodium chlorate by weight. It is very slightly acidified with sulphuric acid. E.M.F., 2·8 volts; current-density, 0·2 ampère per sq. dcm.

Preparation of White Lead.—The electrolyte is a 1·5 per cent. aqueous solution of a mixture of 80 parts of sodium chlorate with 20 parts of sodium carbonate, and is kept slightly alkaline. E.M.F., 2 volts; current-density, 0·5 ampère per sq. dcm.

Preparation of Neutral Lead Chromate.—The electrolyte is a 1·5 per cent. aqueous solution of a mixture of 80 parts of sodium chlorate with 20 parts of sodium chromate, and is

kept neutral. E.M.F., 1·8 volts; current-density, 0·5 ampère per sq. dcm.

Preparation of Acid Lead Chromate.—The electrolyte has the same composition as the preceding, but is kept acid with chromic acid. E.M.F., 1·5 volts; current-density, 0·5 ampère per sq. dcm. In all the above, the anode is of soft lead and the cathode of hard lead.

Preparation of Copper Oxide.—The electrolyte is a 2 per cent. solution of a mixture of 95 parts of sodium borate and 5 of sodium chloride, and is slightly alkaline. The electrodes (here and in the next two) consist of copper. E.M.F., 2 volts; current-density, 0·5 ampère per sq. dcm. (Same conditions for the next two.)

Preparation of Basic Copper Carbonate.—The electrolyte is a 1·2 per cent. solution of a mixture of 80 parts of sodium chloride and 20 of sodium carbonate, and is slightly alkaline.

Preparation of Basic Copper Phosphate.—The electrolyte is a 1·5 per cent. solution of a mixture of 80 parts of sodium chlorate and 20 of sodium phosphate, and is slightly alkaline.

There is no doubt that the oxide, zinc carbonate, Prussian blue, and other substances which are insoluble or nearly so, can be prepared by the same process. The manufacture of white lead has already been tested in all the technical details, and capitalists in Cologne and the neighbourhood have formed a company to exploit the process.—D. E. J.

Potassium Bromide and Fluoride, Electrolysis of. H. Pauli. Zeits. f. Elektrochem. 1897, **3**, 474—478.

After the electrolysis the amount of hypobromite was determined by means of potassium arsenite in alkaline solution ($2KBrO + As_2O_3 = As_2O_5 + 2KBr$), and the total bromine in both salts (hypobromite and bromate) by means of potassium iodide and dilute hydrochloric acid. The iodine was titrated with sodium thiosulphate. From the two results the bromine in the form of potassium bromate can be calculated. Before commencing the electrolytic work, experiments were made on the formation of hypobromite and bromate of potassium chemically. To equal quantities of 10 per cent. potash solution, bromine was added in quantities proportional to 1:2:4:8:32:48. The results of the titration are as follows:—

	Bromine in	
	Hypobromite.	Bromate.
1	0·0140	0·0032
2	0·2168	0·0060
3	0·2752	0·0120
4	0·5528	0·0232
5	0·6755	0·2717
6	Traces	0·8235

Thus, when potash is in excess, the chief product is hypobromite. As more bromine is added, it acts upon the hypobromite first produced and forms bromate; and when a large excess of bromine is present the hypobromite almost disappears. By heating the solution, hypobromite is partially converted into bromate; the change takes place more rapidly in dilute than in concentrated solutions.

The results obtained in electrolysing solutions of potassium bromide are in general similar to those obtained by Oettel in his investigation on the formation of hypochlorite and chlorate by electrolysing solutions of potassium chloride. In two respects they differ. It is impossible to obtain equally good yields, because the bromate formed (unlike the chlorate) is reducible in alkaline solution. Now it is possible to conduct the electrolysis under conditions such as to produce (as in the electrolysis of potassium chloride in alkaline solutions) only bromate (with a negligible amount of hypobromite). Details are given of electrolyses of 10 and 20 per cent. solutions of potassium bromide, neutral and alkaline, hot and cold.

Attempts were also made to produce hypofluorides and fluorates from fluorides. Neutral 20 per cent. potassium fluoride solution was electrolysed under various conditions of temperature and current-density; a strong smell of ozone was noticeable, but no other result was obtained. Caustic potash was then added in gradually increasing quantities to the fluoride; but again the experiments gave negative results.—D. E. J.

Nitrobenzene, Electrolytic Reduction of. W. Löb. Zeits. f. Elektrochem. 1897, **3**, 471—474.

Gattermann succeeded in reducing nitro-compounds electrolytically in sulphuric acid to amidophenols, phenylhydroxylamine being produced as an intermediate product. Probably the author's electro-syntheses of chlorinated and brominated aniline by reduction of nitrobenzene in hydrochloric (or hydrobromic) acid solution took place in analogous fashion. The whole course of the reaction indicates that phenylchloramine (or phenylbromamine) was present as an intermediate product, although it could not be isolated. The author has now endeavoured to carry out electrolytically a general reaction of the following nature:—

$$C_6H_5NO_2 + 2H_2 + HX = C_6H_5NHX + 2H_2O,$$
$$C_6H_5NHX = C_6H_4XNH_2$$

Details of three series of experiments are given, in which the nitrobenzene in various solvents was electrolysed in a beaker with a porous cell. The cathode was of platinum or lead. The anode was of platinum or carbon, and the liquid in its neighbourhood was dilute sulphuric acid. It is not advisable to use lead as the positive electrode, because it dissolves in the solutions. I. *Reduction in Formic Acid Solution.*—The reaction expected was analogous to that which occurs when halogen acids are used:—

$$\text{(A.) } C_6H_5NO_2 + 2H_2 + HCl = C_6H_5NHCl + 2H_2O$$
$$C_6H_5NHCl = C_6H_4ClNH_2$$

$$\text{(B.) } C_6H_5NO_2 + 2H_2 + HCO_2H = C_6H_5NHCO_2H + 2H_2O$$
$$C_6H_5NHCO_2H = C_6H_4.COOH.NH_2.$$

Thus amido-benzoic acid would be obtained direct from nitrobenzene. The expectation was not fulfilled. The chief product of the reaction is hydrazobenzene, which, in the presence of the acid, is transformed into benzidine formate. II. *Reduction of Solutions of Nitrobenzene in Acetic and Oxalic Acid.*—Here again the chief product of the reaction is benzidine (acetate or oxalate). III. *Reduction in Ammoniacal Solution.*—Nitrobenzene dissolved in alcoholic ammonia, or a mixture of aqueous ammonia and alcohol, has an exceedingly low conductivity. This, however, can be increased (without affecting the reaction) by adding ammonium chloride. Here again, hydrazobenzene was formed and precipitated; on passing steam through the filtered liquid, azobenzene was obtained.—D. E. J.

Nitric Acid, Quantitative Determination of, by Electrolysis. K. Ulsch. Zeits. für Elektrochem. 1897, **3**, 546.

See under XXIII., *page* 762.

PATENTS.

Electrical Accumulators [*Electrodes, "Forming," &c.*], *Impts. in.* X. M. Roux, Paris. Eng. Pat. 12,696, June 9, 1896.

"(1) The combination with the positive plates of a coating of unoxidisable metal or non-metallic substance, such as carbon, silica, or the like, by means of electrolysis . . . . (2) A plate consisting of a support of glass, porcelain, or other inert material, in combination with windings of lead wire, strip, or tube, perforated or not . . . . (3) Pellets or fillings of minium and litharge, combined with asbestos or other absorbent and inert material . . . . (4) An electrolyte, consisting of oxalic acid, with or without sulphuric acid, when used as the 'formation' electrolyte, or with sulphate of manganese . . . (5) . . . . sheaths of non-conducting absorbent asbestos for the purpose of insulating the plates from each other and to enable the plates to be packed closely together. . . ."—J. C. R.

Electric Battery [*Carbon with Fused Metallic Oxide*], *An Improved.* H. F. Kirkpatrick-Picard, London. Eng. Pat. 15,223, July 9, 1896.

(1) The production of electricity by the consumption of carbon in an electrolyte composed of a molten metallic

oxide which is decomposed more readily by the carbon than by the metal vessel ("litharge melted in a copper crucible") in which it is held and which forms the other element . . . (2) By the consumption of carbon in an electrolyte composed of a molten metallic oxide . . . . (3) By the consumption of carbon in an electrolyte such as litharge or other suitable oxide contained in a copper vessel, whereby the metallic lead is deposited on the vessel and the oxygen of the oxide converted into carbonic acid . . . (4) In the production of electricity by the consumption of carbon in an electrolyte such as litharge, the regeneration of litharge or the oxide by an air or oxygen blast. (5) The employment, as the electrolyte in a battery such as described, of litharge or other oxide diluted with oxide of sodium, or with caustic soda or potash, or with soda and silica, forming a double silicate, for the purpose described, "to dilute the litharge," to reduce "the strong local action (of a purely chemical nature) and the consequent production of heat," and "to reduce the melting point of the bath." (6) "The employment, as the electrolyte in a battery such as described, of litharge or other oxide diluted with oxide of sodium, or with caustic soda or potash, forming, in the case of lead, alkaline plumbates, or with other metallic oxides forming similar alkaline and metallic compounds; or with soda and silica forming double alkaline silicates."—J. C. R.

Phosphorus, An Improved Process of Manufacturing. [*Electric Furnace.*] L. L. Billaudot, Paris, France. Eng. Pat. 15,977, July 18, 1896.

The process consists in heating metallic phosphates with carbon in an electric furnace so as to obtain the corresponding metallic phosphides, and subsequently heating these phosphides, or phosphides obtained by any other means, with carbon in an electric furnace, with the production of phosphorus and the carbide of the metal. The two stages may be combined in one operation, as in the case of the production of calcium carbide and phosphorus from tricalcium phosphate.—G. H. R.

Primary Batteries [*Leclanché, Air-tight*], *Impts. in and relating to.* R. M. J. Heurtey, Paris, and P. Germain, Auxerre, France. Eng. Pat. 16,597, July 27, 1896.

Relates to the construction of an air-tight battery of Leclanché type, with double chloride of ammonium and magnesium for electrolyte, and cellulose (cocoa-nut, rush) between or around electrodes.—J. C. R.

Electrical Accumulators [*Filling-Grids*], *Impts. in.* A. Conder, London, and P. Michélot, Paris. Eng. Pat. 18,951, Aug. 27, 1896.

A grid, formed by burning or riveting together two separate grids for holding active material, which, as a paste, is made up of sulphuric acid, water, glycerin, and magnesium sulphate, in combination with oxides of lead. The acid (1 in 10) is first added to the glycerin (3 parts by vol.), and allowed to cool.—J. C. R.

Accumulators, Zinc [*Amalgamated*], *Negative Electrode for.* S. Pitt, Sutton, Surrey. From L. Bomel and Société Bisson, Berges and Co., Paris. Eng. Pat. 15,013, June 21, 1897.

"A negative electrode for accumulators constituted by a combination of a supporting conducting strip [copper, &c.], and a trough or basin [ebonite, porcelain, celluloid] containing mercury, in which the said strip rests [vertically]; this arrangement allowing, whilst charging, of continuous amalgamation by the mercury, which creeps along the strip of the electrolysed zinc deposited upon it, and, whilst charging, of the recuperation and preservation of the mercury, which may thus be used again and again indefinitely."—J. C. R.

Utilising the Residual Products [*Caustic Alkali Solution of Zinc*] *obtained in the working of certain kinds of Voltaic Batteries, Impts. in.* W. Walker, jr., and F. R. Wilkins, Birmingham, Jabez Lones and Joseph Lones, Smethwick. Eng. Pat. 19,308, Sept. 1, 1896.

Zinc is precipitated as sulphide from spent caustic alkali battery solutions, either by boiling with flour sulphur, or by addition of an alkali sulphide, or by hydrogen sulphide. The washed and dried zinc sulphide may be fitted for use as a white pigment by heating with 3 per cent. of sulphur to dull redness for half an hour, and then throwing into water; the latter is then removed and the product dried. To regenerate the alkali after removal of zinc, the thiosulphate that has been formed in the initial process, is oxidised, either by hydrogen peroxide, or barium peroxide, or by electrical action; the liquid is then diluted to a density of 1·05, and is boiled with lime, with addition, at the end, of barium hydroxide solution. After clearing, the solution is concentrated for re-use.—E. S.

Rubies, Impts. in the Manufacture of Artificial. [*Electric Furnace.*] W. P. Thompson, London. From the Firm of Gin and Leleux, Paris, France. Eng. Pat. 19,962, Sept. 9, 1896.

A mixture of 95 per cent. of alumina and 3 per cent. of sesquioxide of chromium are melted and volatilised in an electric furnace, heated by an alternating current, and the gaseous products are led through a tube to a condensing chamber heated to 1,500° C. To facilitate the formation of large crystals, a mixture of moist air and hydrochloric acid is forced under pressure into the tube; or, whilst moist air is passed through the tube, small quantities of fluoride of aluminium or cryolite are cast on to the hearth of the furnace from time to time, thus causing the formation of hydrofluoric acid. The fine crystalline deposit formed in the condensing chamber and tube are removed periodically, and can be utilised in place of emery.—G. H. R.

Electrolyser or Electrolytic Apparatus, An Improved. [*Mercury Cathode.*] J. G. A. Rhodin, Eccles. Eng. Pat. 21,509, Sept. 29, 1896.

The electrolyser consists of two concentric cylindrical vessels, the inner one of which contains the electrolyte, and is capable of rotation, and in its bottom is formed a series of hollow tubes or projections containing the carbon or other suitable anodes. The bottom of the outer vessel is provided with radial ribs or projections to retain the layer of mercury which covers it, and above the mercury is water or other liquid which will absorb the cations from the amalgam formed. The electrolyte is preferably a metallic salt, the cation of which is soluble in mercury, and when the inner vessel is rotated, the amalgam formed in the tubes is brought into contact with the body of the mercury and with the supernatant liquid. If it be desired to apply heat during the operation of electrolysis, the outer vessel is preferably formed of iron, and the inner vessel is constructed as a still and hermetically sealed.—G. H. R.

Galvanic Elements, Primary and Secondary [*Deposited Zinc Electrodes*]; *Impts. in and connected with.* H. Leitner, Berlin. Eng. Pat. 24,473, Nov. 2, 1896.

"An electrode for primary or secondary galvanic element consisting in (1) electrolytically depositing a layer of zinc upon a suitable base or core by the aid of a lead peroxide [anode] plate; (2) amalgamating the said layer; and (3) depositing electrolytically an outer layer of zinc . . . . upon the said amalgamated layer, the said outer layer being adapted to serve as the active material of a primary or secondary battery . . . ." The zinc is deposited from a bath made up of zinc sulphate, sulphuric acid, and alum (preferably ammonia alum).—J. C. R.

Secondary or Storage Batteries or Accumulators [*Forming, Filling, Terra-Cotta Supports*], *Impts. in, and in the Manufacture of the same.* J. Y. Johnson, London. From J. P. Clare and G. E. Hatch, Quincy, Mass., U.S.A., and J. H. Taylor, Chelsea, Mass., U.S.A. Eng. Pat. 15,510, June 29, 1897.

1. "The process of preparing a secondary battery element . . . . consisting in autogenously forming active material from the metal of an electrode by electrolytic action, then loosely confining against the coated face of the plate thus produced, granular or pulverulent ['dry powder' preferred] active material, or material to become active, and continuing the electrolytic action so as to effect the union of this mass with the autogenously produced material on the plate . . . .

Other claims relate to the mechanical construction of receptacles and electrodes, in which plates of porous earthenware (terra-cotta) are requisitioned for supports.—J. C. R.

Peat Charcoal, Process for Producing, by Means of an Electric Current. C. A. Jensen, London. From P. Jebsen, Bruvik, Norway. Eng. Pat. 15,513, June 29, 1897.

The process consists in compressing the peat into thin layers, and inserting it as a resistance in an electrical circuit so as to carbonise it.—G. H. R.

(B.)—ELECTRO-METALLURGY.

Bronze and Brass, Analysis of, by Electro-chemical Methods. A. Holland. Comptes Rend. 1897, **124**, [25], 1451.

See under XXIII., *page* 763.

Metals, Electrolytic Methods of Analysis of, or Quantitative Electrolysis of the Heavy Metals. L. Wolman. Zeits. für Elektrochem. 1897, **3**, 537.

See under XXIII., *page* 763.

PATENTS.

Tubes, Impts. in Apparatus for Manufacturing, by Electrolytic Deposition. [*Reciprocating Mandrel.*] A. S. Elmore, Leeds. From J. O. S. Elmore, Kapurthala, India. Eng. Pat. 7222, April 2, 1896.

The depositing vat is of U-section, and divided along its length into compartments, which contain alternately anode shells of the crude metal to be deposited, which surround, or nearly surround a mandrel, and fixed rubber making electrical contact with this mandrel, which is mounted in the tank and rotated, and is also given a reciprocating motion between burnishers, which are also supported in the same compartments. The mandrel is lengthened at each end by pieces of non-conducting material, passing through stuffing boxes at the ends of the tank. Two or more tanks and mandrels may be arranged in line, the latter being connected by non-conducting material, and the anode shells of each tank being electrically connected to the rubbers on the cathode mandrel of the next tank. The electrolyte is circulated through the tank in large volume by means of a suitable pump.—G. H. R.

Tin or Antimony Ores, Impts. in or relating to the Treatment of, and the Separation of the Gold and Silver contained therein. C. F. Claus, Chiswick. Eng. Pat. 13,641, June 20, 1896.

The crude metal obtained by smelting, is immersed in an electrolyte (such as sulphide of sodium of sp. gr. 1·065, or in some cases sodium and potassium hydrate, and heated to 90° C.) and employed as the anode for an electric current of about 10 ampères per square foot of cathode; any suitable material may be employed for the cathode. The crude metal is gradually dissolved and pure tin is deposited upon the cathode. The gold, silver, lead, copper, zinc, iron, antimony, &c. remain insoluble in the anode-mud, some as sulphides and others in the metallic form.

For the separation of antimony, the anode-mud or the ore is smelted with sodium sulphate, carbon and sulphur being added when required. The slag from this smelting contains the antimony in a soluble state; it is dissolved in water and the antimony is deposited by means of an electric current or by known methods, as sulphides.

In some cases, instead of smelting as above, the ore may be fused with proper proportions of sulphur and soda, or sodium sulphate and carbon; or boiled with a solution of polysulphide of sodium, adding an ammonium salt, so as to obtain a proper solution for electrolysis.—J. H. C.

Zinc or other Metals, Impts. in Means or Apparatus for Effecting the Electro-Deposition of, upon Tubes or other Circular or Analogous Objects. [*Internal Anodes.*] The Cowper-Coles Galvanising Syndicate, Limited, and S. O. Cowper-Coles, London. Eng. Pat. 17,608, Aug. 8, 1896.

The apparatus for the electro-deposition of zinc or other metals on tubes or other circular or hollow objects consists of a vat approximating in shape to the article to be coated, in which are suitable bearings carrying supports capable of rotation, and which hold the tube at each end. These supports or holders consist of three portions, and have V-shaped converging projections with their edges outward to bear on the object to be coated. A hollow perforated anode passes through, and is attached to the central portion of one holder, and on the other end of it slides a holder formed of a vulcanite outer portion and an inner metallic portion consisting of a ring carrying V-shaped projections, and means are provided for rotating the object to be plated. The interior of the containing vessel is lined with the metal to be deposited, and forms an external anode, and the electrolyte is circulated through the internal anode and the tank under pressure.—G. H. R.

Electrolytic Apparatus, Impts. in. [*Tapered Cathodes.*] W. L. Wise, London. From The Aluminium Industrie Aktien Gesellschaft, Neuhausen, Switzerland. Eng. Pat. 21,027, Sept. 22, 1896.

In the reduction of sodium or of other light metals by electrolysis, the oxidation of the reduced metal is obviated by employing tapered or rounded cathodes, which dip only a portion of their surface into the electrolyte, while the anodes have a protective casing fixed by asbestos, which allows of the escape of the oxygen. The electrodes are arranged alternately, and suspended over the vessel containing the fused electrolyte from two beams, the anodes being carried by one, and the cathodes by the other, so that they may be raised or lowered independently as required. The deposited metal is removed by scraping from time to time, and placed in a collecting vessel, cylindrical in its upper part and conical in the lower, which is provided with a central hole, closed with a pin, so that the electrolyte can be allowed to escape while retaining the metal. This vessel is supported in the bath by its handle.—G. H. R.

XII.—FATS, OILS, AND SOAP.

"Saponification" of Unsaturated Alcohols and Neutral Bodies. R. Henriques. Zeits. angew. Chem. 1897, 398.

The author has already stated that on treatment with alcoholic alkali, either hot or cold, at ordinary or increased pressures, wool-fat suffers greater change in constitution than corresponds to the mere decomposition of its esters; and he has suggested that the phenomenon is due to the action of the alkali on the unsaturated alcohols. This view Herbig objects to, on the ground of the impossibility of such reaction. Henriques has therefore endeavoured to solve the problem by direct experiment; but as of the unsaturated alcohols and neutral bodies in wool fat only cholesterin has yet been isolated, he has studied the properties of some alcohols derived from other sources. The results of the investigation show that all such compounds are more or less attacked by alkalis, that among the products of the action some acid is to be found, and that they consequently possess a "saponification number."

By heating for two hours at 100° C. in a sealed tube with 10 per cent. alcoholic potash, linalool gave figures corresponding to the presence of 1·16 to 1·86 per cent. of ester, or to the saponification number 4·2 to 6·8. In the cold its saponification number in 24 hours was 4·2; in seven days, 3·9. Similarly, with geraniol, the values were 2·8 and 3·92. In both cases the acids were extracted, and found to be yellow oily bodies insoluble in water. Cinnamic alcohol yielded saponification numbers of 9·35 and 12·04 after being mixed with normal alkali for 48 hours in the cold, while the residual alcohol, on fresh treatment, gave 3·36; boiled for 1½ hour with the same reagent, its number was 14·44. Safrol, anethol, and eugenol were also examined, but the strong red colour produced by the last prevented the titration from being carried out. The figures in the former cases were:—

——	1 Hour Warm.	24 Hours Cold.
Safrol	9·7—4·8	5·52
Anethol	6·05	4·48—5·68

The exact mechanism of this decomposition is the subject of a research which is not yet concluded; but in the meantime it has been proved that cinnamic alcohol is totally changed in structure by cold normal alkali in 24 hours. When the product is distilled from the acids, the thermometer rises constantly to 360° without remaining at any one point, and the residue is thick and oily, and contains benzoic acid. The distillate smells strongly of bitter almonds, and, on treatment with bisulphite and sulphuric acid, yields benzaldehyde. Another ketone of the empirical formula $C_9H_{10}O_2$, giving a hydrazone $C_{15}H_{16}N_2$ (m.p. 120°) is also present, and probably consists of $C_6H_5.CO.CH_2.CH_2OH$. Under similar conditions, anisaldehyde and anisic acid were obtained from anethol. Lanolin also yields an aldehyde, but it has not yet been produced in sufficient quantities for identification.—F. H. L.

Spontaneous Combustion of Oils, Causes of. W. Lippert. Zeits. angew. Chem. 1897, 434.

See under XIII. B., *page* 747.

Lard, Detection of Beef Fat in. M. Ballo. Zeits. Nahrungsm. Hyg. Waar. **11**, 193; through Chem. Centr. **68**, 392.

See under XXIII., *page* 764.

PATENTS.

Lubricants, Impts. in. F. W. Klever, Cologne, Germany. Eng. Pat. 17,118, Aug. 1, 1896.

Heavy mineral oils are mixed with oleic acid and alkali or alkalis, preferably ammonia. This mixture readily forms an emulsion with water.—C. A. M.

Soap, Impts. in the Manufacture of. E. S. Wilson, Strood, Kent, and E. Stewart, London. Eng. Pat. 21,048, Sept. 23, 1896.

Ground linseed or other oil seeds are incorporated with the soap, with the object of neutralising any excess of alkali and of providing an excess of fatty matter not liable to become rancid.—C. A. M.

Soaps. A New Manufacture of, and Process therefor. A. Jolles, Vienna. Eng. Pat. 15,156, June 24, 1897.

Asbestos or infusorial earth, or both, with silicates such as steatite, all suspended in a finely-divided state in an aqueous alkaline solution, are mixed with the soap-body during saponification.—C. A. M.

XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

(*B.*)—RESINS, VARNISHES.

Spontaneous Combustion of Oils, Causes of. W. Lippert. Zeits. angew. Chem. 1897, 434.

On exposure to air, old linseed oils absorb oxygen corresponding to an increase in weight of 15 or 16 per cent.; freshly expressed samples 17 to 18, or occasionally 19 per cent., but never as much as 20 per cent. When the oil is raw, or after it has been boiled without driers (*e.g.*, lithographic varnish) the absorption only reaches a maximum after several days: but in presence of lead or manganese, the action may be complete in 20 hours. The danger of "spontaneous combustion" is therefore much greater in the latter case, and it is liable to occur whenever fibrous or porous materials, saturated with a drying varnish are exposed to the atmosphere. The actual "combustion" depends on the spontaneous ignition of the finely divided and pyrophoric carbon, which is produced as a result of the decomposition of the oil caused by the sudden rise in temperature; and the mass will only break into flame in presence of sufficient air. The same phenomena may be observed with turpentine varnishes containing resins as well as linseed oil; and a thermometer placed in the centre of a mass of cotton wool of about 0·5 metre in diameter, and saturated with an oil varnish, will soon reach 60° C., and in the present case the temperature rose to 138° in 15 minutes, and to 275 in three quarters of an hour. Later on still, vapours of unpleasant odour were emitted, and then the temperature suddenly rose to over 300 C., and the thermometer had to be removed.

The author's conclusions agree closely with those arrived at by Kissling (this Journal, 1895, 479); and he urges that whenever varnishing is done with a rubber in place of a brush, the old pieces of rag, cotton wool, &c. should be burnt immediately, and that fabrics coated with linseed oil should never be packed in heaps until the oxidation of the oil is fully complete.—F. H. L.

Asphaltites of Utah, The Uinta and the Uncompahgre. Eng. and Mining J., July 3, 1897, 10.

The greater portion of the asphaltite is uintaite or gilsonite, but there are also found utahite, elaterite, wartzilite, albertite, ozokerite, and asphaltic limestones and sandstones. To prevent the accumulation of fire-damp in uintaite mining, the walls are frequently washed with a copious deluge of water, to lay the dust. H. Hirsching found that numerous samples of uintaite contained high percentages of light carburetted hydrogen compounds, free hydrogen, and free oxygen. Uintaite or gum asphaltum is wholly soluble in benzine (petroleum spirit), turpentine, and other hydrocarbon solvents; it contains 56·46 per cent. of volatile matter, 43·43 per cent. of fixed residue, and 0·10 per cent. of ash. Uintaite may be used as a base for black varnishes, paints, and japans, as an insulating and roofing material, and as a dip for pipe or wire to prevent oxidation and the corroding action of acids. More recently, it has been used as a dressing for leather, and in one form it furnishes a good imitation of patent leather, which it is practically impossible to crack. Utahite is partially vulcanised, and is only soluble to the extent of 40 per cent., the remainder being fixed bitumen. Albertite has the same composition as uintaite, but is wholly insoluble. Elaterite (or mineral caoutchouc) is partially vulcanised asphaltum, and is stated to be the most elastic substance known. Ozokerite (or mineral wax) is used for making tapers. An artificial ozokerite is prepared by a secret process of the Assyrian Asphaltum Company from uintaite by-products.

The asphaltic limestones contain a maximum of 72 per cent. of soluble bitumen and 11 per cent. of fixed bitumen, but the general average is 22 per cent. of soluble bitumen; the remainder is magnesian limestone and silica. The average percentage of total bitumen during 1896 was 29·70. This asphaltum contains no other organic matter, thus differing from the Trinidad and other species. Utah asphaltum is stated to be eminently suitable for street-paving purposes, although, according to some authorities, a mixture of asphaltic sandstone and asphaltic limestone gives a more lasting, finer grained, and better pavement.—A. S.

XIV.—TANNING, LEATHER, GLUE, SIZE.

Fermentation Phenomena in Tan Liquors. F. Andreasch. Imp. Research Laboratory, Vienna, Der Gerber, 23,536, 3.

Sources of Nitrogen.—The importance of these has been mentioned in the bacteriological portion of the research. It has been noted that (1) the amount of lactic acid in unused liquors, even after long standing (except sumac, myrabolams, and divi-divi liquors), is small; (2) in old liquors which have been used over and over again, it is large. The author finds the difference to be due entirely to the amount of nitrogenous nutriment furnished to the bacteria. The N was estimated in the principal tanning materials by Will and Varentrapp's and by Kjeldahl's methods; in the latter H_2SO_4 only was used—no additions; it gave slightly higher values than W. and V.'s method, but the difference was not important. In the table given first on page 748, the nitrogen is reckoned on the dry substance.

It must be borne in mind that only part of the nitrogenous matter is soluble, and that some of this is precipitated by the tannin. The amount of soluble N in all the materials was therefore estimated (Table A) and fermentations conducted in the following media:—(1) Original tannin infusion, (2) the same, with addition of glucose, (3) peptone, (4) potassium phosphate and magnesium

Tanning Material.	Per Cent. of Nitrogen.	
Valonia	0·582	0·771
Knoppern	0·787	0·927
Myrabolams	0·490	0·451
Divi-divi	0·914	0·936
Sumach	1·171	1·230
Willow bark	1·040	1·220
Mimosa bark	..	0·570
Oak bark	0·465	0·532
Pine bark	0·481	0·550
Hemlock bark	0·407	0·316
Quebracho wood	0·202	0·277
Oak wood	0·474	..

sulphate, (5) sugar and peptone. The inoculating material consisted of four lactic acid organisms: a bacterium, a bacillus, a micrococcus, and a yeast, all isolated out of tan liquors. The results of the fermentations by the bacterium and the yeast are given in Table B. The non-tannin in all the solutions was made equal. The amount of N in the liquors is seen to be much smaller than in the original material. Slightly more nitrogen was found in the liquors prepared by cold extraction. The concentration of the liquors made no difference to the amount of N dissolved; *i.e.*, 100 parts of the material yield the same amount of soluble nitrogenous compounds whether 25, 50, or 100 grms. per litre be used.

The table shows the influence of the amount of N on the quantity of lactic acid produced. The amount of acid in all the materials is very small, and far less than met with in practice. The peptone used was free from albumins and albumoses.

Table A.

Name of Tanning Material.	Original Tanning Material contains				Nitrogen in Extract.	Nitrogen in 100 parts Tan-Liquor.	——
	Tannin.	Non-tannin.	Nitrogen.	Nitrogen in Soluble Form.			
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	
					0·3900	0·0168	Extracted cold, 100 grms. tanning material per litre.
Sumach	23·24	19·95	1·171	0·1497	0·3544	0·0153	Extracted hot, 100 grms. per 100 c.c.
					0·3421	0·0074	Extracted hot, 50 grms. per 100 c.c.
					0·3429	0·0038	Extracted hot, 25 grms. per 100 c.c.
Valonia	28·14	15·22	0·582	0·0521	0·1201	0·0052	.. .. ..
Knoppern	28·82	8·65	0·787	0·0444	0·1185	0·0044	.. .. ..
Myrabolams	30·23	15·66	0·490	0·1090	0·2375	0·0109	Extracted hot, 100 grms. original tanning material per litre.
Oak bark	9·88	7·21	0·465	0·0323	0·1774	0·0032	.. .. ..
Pine bark	12·08	12·52	0·481	0·0513	0·2083	0·0051	.. .. ..
Hemlock bark	10·71	7·46	0·407	0·0372	0·2048	0·0037	.. .. ..

Table B.

Name of Tanning Material.	Original Liquor contains per 100 c.c.				Lactic Acid per 100 c.c. Liquor.					
					Original Liquor; no Addition.	Addition per 100 c.c. Liquor of				Inoculating Material.
	Tannin.	Non-Tannin.	Sugars reckoned as Glucose	Nitrogen.		0·5 grm. K_2HPO_4; 0·5 grm. $MgSO_4$.	1 grm. Glucose.	0·25 grm. Peptone.	1 grm. Glucose 0·5 grm. Peptone.	
	Grm.	Grm.	Grm.	Grm.	Grm.	Grm.	Grm.	Grm.	Grm.	
Valonia	0·9242	0·500	0·0946	0·0018	0·0219	0·0220	0·0250	0·4843	0·8044	I.
					0·0252	0·0281	0·0261	0·0821	0·7937	II.
Knoppern	1·6655	0·500	0·0191	0·0024	0·0221	0·0234	0·0258	0·4800	0·7955	I.
					0·0210	0·0244	0·0231	0·0173	0·7977	II.
Myrabolams	0·9652	0·500	0·2028	0·0035	0·0661	0·0678	0·0655	0·4371	0·8211	I.
					0·0685	0·0688	0·0670	0·1744	0·8000	II.
Sumach (Sicilian)	0·5825	0·500	0·1486	0·0042	0·0809	0·0841	0·0854	0·1900	0·8265	I.
					0·0842	0·0822	0·0855	0·1311	0·8155	II.
Oak bark	0·6855	0·500	0·1900	0·0021	0·0278	0·0291	0·0270	0·4777	0·7900	I.
					0·0243	0·0275	0·0254	0·1721	0·7812	II.
Pine bark	0·4826	0·500	0·1662	0·0021	0·0279	0·0275	0·0293	0·4855	0·8002	I.
					0·0260	0·0243	0·0275	0·1437	0·8105	II.
Hemlock bark	0·7185	0·500	0·0704	0·0024	0·0310	0·0300	0·0285	0·4932	0·7973	I.
					0·0275	0·0255	0·0261	0·0654	0·7916	II.

I. = Lactic acid bacterium. II. = Lactic acid yeast.

—J. T. W.

[*Glove*] *Leather Dressing, Employment of Canine Excrement and Putrefied Urine in.* E. d'Huart. Bull. Assoc. Belge des Chimistes, **11**, [2], 47—58 (this Journal, 1897, 687).

A. Canine Excrement.—The action of this material is essentially destructive, attacking the amorphous organic matter of the cutaneous tissue; but, being due to the vital activity of contained microbes, and not to the chemical substances present, cannot therefore be imitated by means of mixtures of the latter, but only by bacteriological cultures of the said organisms.

B. Putrefied Urine.—This liquor acts as a mordant for the dyes applied to the glove-leathers, and for this purpose may be efficiently replaced by a solution containing 15 to 20 grms. of ammonium carbonate—or their equivalent in potassium or sodium carbonate—per litre. Since, however, the influence of the various alkali carbonates on the *shade* of colour produced by a dye differs, it is found that to satisfactorily supersede urine, the solution must contain both fixed and volatile alkalis, the desired effect being obtained by the use of 10 to 15 grms. of sodium carbonate and 10 grms. of ammonium carbonate per litre.—C. S.

PATENTS.

Leather-Board and Material suitable for being pressed into form for Decorative and other Purposes, or for use in Substitution for Leather, Floorcloth, and the like; Treatment of Waste Substances for. T. H. Cobley, Dunstable. Eng. Pat. 13,096, June 13, 1896.

Waste fibrous material and the wax, oil, albuminous and extractive matter derived from cotton-seed and shell are pulped together, boiled with silicate of soda, and the whole precipitated by addition of milk of lime. The alkaline liquor is then drained off from the precipitate. Waste leather or other animal substance is separately treated with sulphurous acid, and then added and thoroughly mixed with the first-obtained pulp, together with tanning agents or chromium compounds. The mass is then made into boards, or pressed, or moulded as desired.—C. O. W.

Gelatinous Resilient Composition, applicable for Wheel Tyres and other Purposes. D. Forbes and J. S. Weymouth, Bristol. Eng. Pat. 18,864, Aug. 26, 1896.

Glue is melted with water and subjected to heat, and to 76 parts of it 3 parts each of sulphur, sulphate of barium, a saturated solution of alum, and collodion are added. To the cold mixture are then added 3 parts each of sulphuric acid, glacial acetic acid, nitric acid, and formic acid. To soften the mixture, glycerin may be added. After setting, the mixture can be moulded into tyres and other articles. —C. O. W.

Tanning Hides and Skins, Impts. in. W. Clark, London. Eng. Pat. 21,210, Sept. 24, 1896.

The skins prepared for tanning, and previously slightly swelled, are steeped for three days in a bath containing, in 1,000 c.c. of water, 4 grms. of lactic acid (73 per cent.), 2·6 grms. of potassium bichromate, and 0·9 grm. of sulphuric acid. Subsequently an eight-days' treatment in a bath twice as strong and then four times as strong is given. The skins are afterwards washed, dried, and prepared as usual.—C. O. W.

Waterproof Glue, Improved Manufacture of. H. Gardner, London. Eng. Pat. 21,774, Oct. 1, 1896.

Casein, albumin, blood, or glutin are mixed with alkaline substances, such as calcium hydrate, potassium hydrate, sodium hydrate, the carbonates, silicates, borates, or phosphates of sodium or potassium. Preparations with from 15 to 50 per cent. of alkaline substance form a glue not soluble after a time at the ordinary temperature. Preparations with from 5 to 20 per cent. of alkaline substance form a glue which is applied in conjunction with heat. —C. O. W.

Myrabolams, Obtaining Extracts therefrom; Impts. in or relating to Machinery or Apparatus for Treating. S. Smithson, Heckmondwike. Eng. Pat. 12,399, May 19, 1897.

See under VI., *page* 737.

XV.—MANURES, Etc.

PATENTS.

Fertiliser, An Improved. C. Fell, Hanley, Staffordshire. Eng. Pat. 17,151, May 3, 1897.

The fertiliser is prepared from excreta and urine, which are treated with sulphuric acid and mixed with sewage sludge. The mixture is then dried. The inventor claims the production of a manure containing 4 per cent. of nitrogen and 20 per cent. of phosphate.—D. B.

Manure, A Consistent Friable; An Improved Process for making. [*Orthophosphoric Acid and Peat.*] I. Kovacs, Budapesth, Hungary. Eng. Pat. 13,811, June 4, 1897.

It has recently been shown that in many cases it is preferable to allow the phosphoric acid employed for manurial purposes to come into the ground in the form of orthophosphoric acid. Its practical use has, however, been prevented hitherto by the circumstance that it is very expensive, and also that it does not come into commerce in the form of a consistent friable artificial manure, but in the form of a liquid. This form is, however, for agricultural purposes, not only costly, but its practical utilisation encounters many technical difficulties. The inventor overcomes these defects by the addition of absorbent substances, more particularly peat. By this addition the pulpy, stringy, soft product, produced by the free phosphoric and sulphuric acid and uncombined water, is converted into an artificial, easily distributed manure.—D. B.

XVI.—SUGAR, STARCH, GUM, Etc.

[*Sugar Manufacture.*] *Checking Losses in Diffusion.* H. Pellet. Bull. Assoc. Belge des Chimistes, **11**, [2], 58—64.

The author's conclusions are that—

(1.) Provided the glucose or pre-existing reducing sugars be taken into account, the polarimeter and the copper methods will give precisely concordant results in the cases of the beet, the diffusion juices, and the spent slices.

(2.) As regards the copper methods for sugar estimations in beet and pulp, it is best to invert in the filtered liquor containing—as a result of sufficiently long, warm digestion—the whole of the crystallisable sugar.

(3.) Direct inversion of the pulp of fresh or spent slices may, however, be effected by the use of tartaric acid (1 grm. per 100 c.c.), provided the digestion at 85—90°, or even 100° C., be sufficiently prolonged to ensure the complete diffusion of the sugar and its conversion into glucose.

(4.) The final dilution of the solution from the fresh slices or the diffusion juice should be so adjusted that the quantity of sugar amounts to about 5 grms. per litre, so as to be under the same conditions as the normal cupric reagent and the titrated glucose, when their "titres" are determined.

(5.) For estimating the sugar in the spent slices by inversion, the solutions being more dilute, the Fehling's or Violette's reagent should be titrated by a solution of inverted glucose containing 1 grm. per litre (reckoned in crystallisable sugar to save calculation).

(6.) For inversions in presence of pulp, *it is absolutely essential to avoid the use of sulphuric acid.*

(7.) For the indirect (polarisation) estimation of sugar in spent pulp, the slices should be finely comminuted and strongly pressed.

(8.) By taking all necessary care to ensure the exactitude of the elements required, there will be no undetermined loss in diffusion, either in sugar or polarimeter reading, whichever method be employed—that of polarisation or that by Violette's reagent.—C. S.

Caroubin: A New Carbohydrate. J. Effront. Comptes Rend. 1897, **125**, 38—40.

From the seeds of *Ceratonia siliqua*—used as a cattle food in Portugal—the author has isolated a new carbohydrate, *caroubin*, which has, empirically, the same composition as cellulose ($C_6H_{10}O_5$). When purified, it forms a white, spongy, very friable mass, which yields, in contact with water, a syrupy liquid or jelly, 3—4 grms. of the substance being sufficient to convert 1 litre of water into a thick syrup. Caroubin is soluble in dilute HCl; the solution does not reduce Fehling's solution, and is optically inactive: but when boiled, a fermentable, dextro-rotatory, reducing sugar is formed. Caroubin appears to be widely distributed in nature; it occurs in rye and barley, and very probably enters into the composition of beer.

Caroubin may, in some cases, be with advantage used instead of gelose, for the preparation of the nutritive media employed in bacteriological work.—H. T. P.

Caroubinose. J. Effront. Comptes Rend. 1897, 309—311.

The author has isolated a syrup by boiling powdered caroubin (50 grms.) with dilute sulphuric acid (1 litre), containing monohydrate, H_2SO_4 (50 grms.). The product, which he names caroubinose, consists of a sugar ($C_6H_{12}O_6$) which has about the same reducing action on Fehling's solution as dextrose, but its specific rotatory power is lower, *viz.*

$[\alpha]_D = 24$. It ferments with yeast, forms a hydrazone $C_{12}H_{18}N_2O_5$, melting at 183° C., and an osazone $C_{18}H_{22}N_4O_4$, melting at 198° C.—T. A. L.

Sugars, Iodometric Estimation of. G. Romijn. Zeits. Anal. Chem. **36**, 349.

See under XXIII., *page* 765.

PATENTS.

Boneblack or Animal Charcoal, Process for Revivifying. J. Wetter, Strand, London. From M. Weinrich, St. Louis, U.S.A. Eng. Pat. 19,310, Sept. 1, 1896.

Refers to Eng. Pat. 4354 of 1894 (this Journal, 1895, 178), for decarbonising and revivifying boneblack without charring. In this case, the apparatus therein described may be used, the process being one of oxidation, carried on at a comparatively low temperature. The drum or vessel used is heated to 300°—400° F., a regulated current of air is passed through, and the boneblack, &c. fed regularly into one end of the drum and removed from the other. During the time it is within the drum, it is continually being raised and let fall by a revolving shaft having paddles.—R. S.

Granulation of Crystallisable Liquids, An Improved Evaporting or Vacuum Apparatus for. T. Grosse, Kiew, Russia. Eng. Pat. 27,477, Dec. 3, 1896.

See under I., *page* 719.

Glucose or Sugar, Impts. in the Manufacture of. L. P. Bauer, Peoria, Illinois, U.S.A. Eng. Pat. 15,295, June 26, 1897.

The apparatus consists of a sulphurous acid generator and means for the delivery of the gas into a charging tank containing starch sludge at the ordinary temperature. The sludge is saturated *in vacuo*, after which it passes into the converter, where it is heated under pressure to the point of conversion. The pressure is then removed from the product, and the sulphurous acid recovered in the form of solution by passing it into water.—J. L. B.

Errata.

This Journal 1897, 622, col. 2, line 7 from top. In the equation insert CH_2 at the lowest part of the formula for the potassium salt of *d*-fructose.

Ibid., page 623, col. 2, line 4 from top: for "prima seconds" read "good seconds"; and line 13 from top, for "1 sq. cm." read "1 sq. decim."

XVII.—BREWING, WINES, SPIRITS, Etc.

Re-kilning Malt, Effects of. F. Schönfeld. Wochenschr. f. Brau. **14**, 137; Zeits. f. d. Ges. Brauw. **20**, [24], 319.

To remedy a deficiency in depth of colour and aroma, a malt from moderately heavy barley with a rather low percentage of nitrogen was replaced on the kiln and exposed to a temperature of 106° C. (the original kilning heat) for 8 hours. The objects in view were to some extent attained, the colour very slightly increasing, in a degree expressed by 0·1 c.c. of decinormal iodine (original shade = 0·3 c.c. decinormal iodine), and the aroma being improved. Other changes effected were: reduction of moisture from 4·7 per cent. to 1 per cent.; extract in dry matter increased from 68·8 per cent. by 0·8 per cent. in one example and by 2·2 per cent. in another; maltose (64 per cent.) in extract reduced by 4·5 per cent. and 5·6 per cent. in two samples; considerable weakening of the diastase, manifested by an increase in the time required for saccharification from 15 minutes to 23—25 minutes; slight decrease (0·06 per cent.) of nitrogen in dry matter.

The comparatively slight influence on the colour, caused surprise, in view of there being 5 per cent. of water still present before the re-kilning, and because of the rapidity of heating (to 106° C. in 2—3 hours) practised. It is considered that a greater effect would have been produced had the acrospire been more fully developed than in the parcel in question, in which it attained to but little more than half the length of the corns.—C. S.

Yeast, The Nutrition of. C. G. Matthews. J. Fed. Inst. of Brewing, 1897, **3**, 368—400.

The author treats this subject under the following heads:—

(*a.*) General considerations respecting the saccharomycetes and their vital functions. (*b.*) *Résumé* of facts ascertained concerning the constituents of yeast. (*c.*) and (*d.*) The inorganic and organic aliment derived from brewing materials. (*e.*) Experiments and experimental methods relating to yeast nutrition. (*f.*) Some practical deductions from the above sections.

In the experimental portion, it is stated that the yeast employed was *Saccharomyces coagulatus*—an organism well adapted for the investigation, inasmuch as it can be kept clean and free from bacteria and other ferments by washing. One litre of cold-water malt extract, or worts which were of two gravities (1,030 and 1,050), were pitched with 2 grms. of washed and pressed yeast taken from stock fermentations of hopped malt wort, or of wort prepared from commercial malt extract. The proportion of hops, when used, was 10 grms. to a litre of wort. The one-litre portions of wort were shaken up with air after pitching, and kept at a constant temperature of 63° F. for 15 hours, and subsequently at 70° F. At the end of the first 24 hours, the wort was usually poured into a beaker, the yeast remaining behind being shaken up to slightly aërate it, the wort poured back, and fermentation allowed to proceed until finished. It is pointed out that the experiments were designed to show the increase of yeast and the attenuation, but not the fermentative power. This increase of yeast is considered by the author to be the best guide to the suitability of the nutrient matters in the wort, and also to their quantity, provided that there is a sufficiency of fermentable matter present to ensure the yeast making the best use of the alimentary matter.

The principal conclusions drawn from the experiments are: (1.) That the boiling and coagulation of the albuminoids in cold-water malt extract make but little difference to the yield of yeast, allowing for the gravity of the worts. (2.) That unboiled cold-water malt extracts and worts give a very unclean fermentation, and that the yeast increase is thereby restricted. (3.) The boiled worts give a healthy fermentation and a good yeast crop, even in absence of hops. (4.) Hot mashes corresponding in strength to 10 per cent. cold-water mashes, give but slightly more yeast in the same volumes, showing that the sugar of the cold-water extracts is nearly adequate to secure the assimilation of the yeast nutrients. (5.) From the experimental evidence, the influence of hop extract, whether unboiled or boiled for a short time, is in the direction of diminishing the yeast output of malt wort.

Concerning the practical considerations, the author is of opinion that freshly prepared malt of good quality probably contains sufficient mineral and nitrogenous alimentary matter for the yeast, and it is almost certain that a considerable variation in the relation of these two classes of nutriment may exist, and that the amounts of each vary considerably. A variation of the phosphates, especially those of potassium and magnesium, may prove injurious to the yeast. The organic nourishment depends more especially on the malting process, and not so much on the original proteids of the barley. A fermentation may be sluggish from an excess of yeast nutriment or a deficiency; the latter may arise from poor malts or from an excess of substitutes. In such a case the wort may be pitched with a larger amount of yeast, or else with a stronger yeast, so bringing the potential of the yeast into the proper relationship with the work which has to be accomplished by it.
—J. L. B.

Alcoholic Fermentation without Yeast Cells. Will. Zeits. für das Ges. Brauw. 1897, 363.

1. The author has attempted to prepare the ferment (zymase), but without success; he, however, was provided

with a little of the yeast sap prepared by Buchner, and found it to have the properties ascribed to it by Buchner. He thinks it is not usually present in yeast, but that probably the pressed yeast employed by Buchner was in a state of decomposition.

2. Lindner, Delbrück, and others (Wochenschr. für Brau. 1897, 364) have also attempted to prepare the ferment, but also without success. Doubt is thrown on the freedom from spores of Buchner's dried yeast (this Journal, 1897, 549), as in any preparation made by them having fermentative action, they have always found evidence of living yeast.—A. L. S.

Fermentation in Media consisting of Solid Particles. T. Schlœsing. Comptes Rend. 1897, **125**, 40—43.

It has frequently been noticed that during the fermentation of solid media—*e.g.*, the nitrification of soil, the fermentation of tobacco in the manufacture of snuff, &c.—the process comes to a standstill long before the medium has been exhausted, but may be restored to its original, or even greater vigour, by stirring or crumbling the mass. In fact, whenever fermentation has become weak, it may be accelerated by stirring the medium. At first sight it is naturally concluded that the recrudescence of fermentation is due to the aëration of the medium, brought about by agitation. The author shows, however, by experiments (with stable manure) that the acceleration produced by agitation is due to some cause unknown, and that aëration has little or nothing to do with the phenomenon, seeing that it obtains *in vacuo* just as well as in the presence of air. On the other hand, aeration unaccompanied by stirring has no effect on the fermentation.—H. T. P.

Wines, Loss of Colour in ("Casse"); Influence of Iron Compounds. H. Lagatu. Comptes Rend. 1897, **124**, [25], 1461—1462.

A ferric salt, added to a wine of good colour (in quantity not greater than might occur naturally), produces a precipitate quite similar to that in *cassé* wines.

A ferrous salt added to the same wine in bottle produces no effect, but when exposed to the air, *casse* sets in, following a course quite similar to the natural *casse*.

A ferrous salt and sulphurous acid added (the latter in such quantity as would be used to remedy a tendency to natural *casse*) to the same wine, produce no result even after prolonged exposure to air.

Analysis of wines liable to this modification, before and after the *casse*, has shown that after exposure to air almost the whole of the iron of the wine is found in the precipitate.

Probably, then, a wine subject to *casse*, contains ferrous salts, which on exposure to air (with or without the aid of an oxidising diastase) become ferric salts, and then precipitate the tannins, among which is to be reckoned the colouring matter. An oxydase may be essential: but, if so, it is to oxidise the ferrous salt, not directly to render the colouring matter insoluble. This view throws light on the influence of tartaric and citric acids on the *casse*, suggests questions as to certain changes in white wines, and as to the practice of washing with ferrous sulphate solution—problems which the author is now endeavouring to solve. (See also this Journal, 1897, 156, 347.)—J. T. D.

Volatile Acids in Wine, Estimation of. U. Gayon. Revue de Viticulture; La Bière, **5**, [6], 81, and [7], 97.

See under XXIII., *page* 766.

PATENTS.

Waste Hops and Malt, Process for Utilising, from Breweries. H. Krausz and L. Vass, Hungary. Eng. Pat. 12,958, May 25, 1897.

The liquid extracted from waste malt and hops is concentrated, and mixed with yeast in the proportion of 25 to 50 decagrms. of yeast to 100 litres of liquor, and fermented at 20° to 24° C. The fermented liquor is then distilled in a suitable apparatus. The process can be modified by the admixture of fermentable or fermented substances with the above-mentioned liquid.—J. L. B.

Yeast and Bacteria, Impts. in Propagating Apparatus for Developing Pure Cultures of. N. Bendixen, Copenhagen. Eng. Pat. 14,106, June 9, 1897.

The apparatus consists of a fermenting vessel and reserve cylinder connected by a rubber tube to enable the contents of the one to flow into the other by raising or lowering the reserve cylinder. When sufficient yeast has been grown in the fermenting vessel, a portion is run into the reserve cylinder and used for seeding the next fermentation.

—J. L. B

XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

(A.)—FOODS.

Heavy Metals in Cheese, Note on the Presence of. A. H. Allen and F. H. Cox. Analyst, 1897, **22**, 187—188.

The authors consider that the metallic lead recently found by F. W. Stoddart in a sample of Canadian cheese, was probably due to the addition of a lead salt—probably the acetate—to the cheese.

Two samples of "cheese spice," used to prevent the heaving and cracking of cheese, were examined, and it was found that one sample consisted of crystallised sulphate of zinc, whilst the other was a solution of sulphate of zinc corresponding to 38 per cent. of the crystallised salt. It was found that 99·28 per cent. of the total zinc employed, passed into the curd, whilst the remaining 0·72 per cent. remained in solution in the whey. R. Bodmer recently examined two samples of cheese containing zinc, and found that one, a pale coloured cheese, contained 0·84 grain of zinc, or 3·7 grains of the crystallised sulphate per lb.; and the other, a reddish cheese, contained 0·56 grain of zinc, or 2·5 grains of the crystallised sulphate per lb. It is stated that sulphate of copper was formerly added to cheese, and that the green mould in certain kinds of cheese has been imitated by the insertion of copper or brass skewers.

—A. S.

Caroubin: A New Carbohydrate. J. Effront. Comptes Rend. 1897, **125**, 38.

See under XVI., *page* 749.

Lard, Detection of Beef Fat in. M. Ballo. Zeits. Nahrungsm. Hyg. Waar. **11**, 193; through Chem. Centr. **68**, 392.

See under XXIII., *page* 764.

Boric Acid in Milk, Detection of, and Rapid Method of Determination. G. Denigès. J. Pharm. Chim. **6**, [2], 49.

See under XXIII., *page* 766.

PATENTS.

Yeast, Impts. in the Production of Compressed Bakers'. M. P. Hatschek, Middlesex. Eng. Pat. 9908, May 9, 1896.

Malt combs, with or without bran, or peas, lupines, &c., are mashed like grain with malt, or only steeped in water at 140° to 200° F., with an addition of a small percentage of mineral or organic acid, dependent upon the amount of infection in the raw material or water. This extract is mixed with 1 to 3 lb. of maltose or other sugar for each pound of compressed yeast to be produced. The seed-yeast is introduced into the fermenting vat containing the above-described nutrient liquid, the whole being maintained at a temperature of 85° to 87° F., and filtered air pumped in by means of a rouser or other device. Boric acid or its salts, in the proportion of 1 part to 5,000—10,000 of fermenting liquor, is added to prevent infection or the formation of undesirable organic acids. When the fermentation is finished, the wash is cooled to 72° F. by adding cold water, and allowed to stand for 10 to 12 hours, the yeast being then removed by decantation and straining through bags. It is again washed with water containing boric acid in the previously mentioned proportions, and pressed, and packed in the usual manner. This process is also applicable for the manufacture of yeast for distilling purposes.—J. L. B.

Fat or Fatty Waste, Treatment of. T. Coad, East Ham, Essex. Eng. Pat. 15,336, July 10, 1896.

All traces of decomposition and dirt are removed by mixing the waste fat with an antiseptic (preferably boric acid), extracting the liquid fat (preferably by dry heat), boiling it in a solution of gelatin, and precipitating the latter (preferably with alcohol, 60 over proof). When washed and dried, the fat is said to be fit for food.—C. A. M.

"Separated" Milk, A Method of Treating and Employing the same in the Making of Bread, Biscuit, and other Food stuffs. H. Higgins, Cambridge. Eng. Pat. 15,471, July 13, 1896.

The "separated" milk is evaporated to dryness or to a suitable consistence, and mixed with any or either of the following substances:—Flour, meal, semolina, tapioca, sago, oatmeal, gluten, cane sugar, milk sugar, lactose, glucose, animal or vegetable fat, and flavouring and colouring matters. Or these substances may be mixed with the caseinogen obtained from the "separated" milk by precipitation with an acid. Claim is also made for the use of such combined products as flour in the manufacture of bread, biscuits, and the like.—C. A. M.

Caseinogen, Method for Causing the Separation of, from "Separated" Milk, and its Preservation in a Dry State. H. Higgins, Cambridge. Eng. Pat. 16,860, July 29, 1896.

The milk is agitated with a suitable proportion of an acid, such as acetic, hydrochloric, nitric, or phosphoric acids, and the separated caseinogen washed free from acid and lactose, and dried in a suitable drying chamber.—C. A. M.

Liquid Extracts of Coffee or other Substances, Impts. in the Manufacture of, and in Apparatus for the Purpose. J. F. Duke, West Dulwich, London. Eng. Pat. 19,675, Sept. 5, 1896.

In order to avoid the necessity of concentrating the extracts by evaporation and the consequent loss of aroma and fragance, the coffee or other substance is extracted in a series of connected hermetically closed vessels by means of boiling water, which is forced by pressure from vessel to vessel throughout the series, commencing with that in which the coffee is nearly spent. From time to time the vessel nearest the boiler is removed, the others moved a stage nearer, and a new vessel with a fresh charge placed next to the receiver. —C. A. M.

Preventing the Decomposition of Animal, Vegetable, or other Alimentary Substances or Products: An Improved Method of. K. I. O'Doherty, Brisbane, Queensland. Eng. Pat. 6915, March 16, 1897. (Under Internat. Convention.)

The substances are surrounded with an atmosphere of nitrogen in closed receptacles, and maintained at a temperature of from about 28°—50° F. Similarly, in defrosting hard frozen substances, such as meat, decomposition is prevented by filling the chamber with nitrogen, and letting the temperature rise, to about 40° F., until the defrosting is complete.—C. A. M.

Milk, An Improved Process of Preserving. W. T. Murray, Southland, New Zealand; at present, Wimbledon, London. Eng. Pat. 10,873, May 1, 1897.

The milk is heated to boiling point, $\frac{1}{2}$ lb. to $1\frac{1}{2}$ lb. of glycerin with about five times the volume of water added to each 100 lb., and the whole concentrated *in vacuo* to one-fourth or one-fifth its original volume.—C. A. M.

Cocoa Husks, A New or Improved Extract of, and a Method of Preparing the same. H. Christensen and R. Hamann, both of Copenhagen. Eng. Pat. 14,624, June 16, 1897.

The husks are extracted by soaking in water or steam, then boiled with water, and the resulting liquid separated from refuse and concentrated in open pans or *in vacuo*. The theobromineous extract may be used alone or as an admixture with cocoa, chocolate, &c.—C. A. M.

(B.)—SANITATION; WATER PURIFICATION.

Garbage, Disposal by the Merz System. J. Pownall. Proc. Inst. Civil Eng. 1897, **128**, [2]; Paper No. 2962.

The Merz system is an extraction process, in which a volatile hydrocarbon solvent is used. At St. Louis, Mo., U.S.A., naphtha is used, and the products obtained are grease and a residual fertiliser of some value. The garbage is discharged from the waggons into receiving tanks, from which it is drawn as required through a door in the bottom, and, after sorting, it is fed into the driers through 12-in. feed pipes. The driers consist of long chambers, elliptical-shaped in cross section, and are supplied with steam jackets over two-thirds of the perimeter on the lower side. The garbage is agitated by a central shaft and reel, and the condition of the material is ascertained from time to time by inspection through a test hole in the rear of the apparatus. A period of four to five hours at 250° F. is required to complete the drying operation, and the vapours driven off, amounting to 65—75 per cent. of the total weight, pass to water-column condensers, the condensed liquids passing to the sewer, and the uncondensed vapours being conveyed through pipes to the retorts in the boiler furnaces, where they are consumed. The dried material is discharged automatically from the rear of the drier into a screw conveyor, and thence, by means of an elevator, is carried to the extractor room. The extractors consist of cylindrical tanks of similar construction to the receiving tanks, but with the addition of the necessary appliances for regulating the admission of the solvent and steam. The tanks have perforated false bottoms, between which and the lower faces of the tanks are placed coils, so that a gentle heat may be applied to the material under treatment. The garbage is allowed to soak until all grease, oil, and fatty matters are taken up by the solvent (two to four hours), and the solution is then drawn off. The naphtha is distilled off and condensed for further use, and the residual grease is purified. The process is said to be automatic and continuous.—A. S.

Air in the Purifying and Revivifying Houses of Gas-works, Contamination of the. H. Drehschmidt. J. für Gasbeleucht. 1897, **40**, 517—520.

Breathing the contaminated air that prevails in the purifying and revivifying houses was stated to have an injurious effect on the health of the workmen employed in those houses at the Berlin municipal gasworks. Investigations were undertaken at the Müllerstrasse works (Berlin), chiefly by Dr. H. Wolf.

The chief poisonous constituents of coal-gas are ammonia, hydrocyanic acid, sulphuretted hydrogen, carbon bisulphide, other gaseous sulphur compounds, and carbon monoxide. All but the last are absorbed by oxide of iron, and are evolved from it during revivification. They were therefore sought for in the air above the revivifying floor, at the height of the workmen's heads. In modern works only 1 to 20 grms. of ammonia per cubic metre of gas reach the purifiers. All but a trace is retained there, chiefly as carbonate. The ammonia in the air was estimated by aspirating 400 to 600 litres of air through $\frac{1}{20}$N sulphuric acid. Above the revivifying floor, 0·00002 to 0·00004 vol. per cent., and in a purifier in course of being emptied, 0·00041 vol. per cent. of ammonia were found in the air. Lehmann has observed that 0·05 vol. per cent. in air produces irritation, but that men can become accustomed to 0·03 to 0·05 per cent. In works where ammonia is nearly removed from the gas before it enters the purifiers, no injury therefore need be feared to the health of the purifier workmen from its inhalation.

Several methods of estimating hydrocyanic acid in air were tried, and proved unreliable. Eventually, estimations were made as follows:—500 to 600 litres of the air were drawn, at the rate of 50 litres per hour, through two absorption vessels containing water holding in suspension fine mercuric oxide. The water was subsequently made up to a known volume filtered, and a known volume of the filtrate removed, and treated with ammonia and zinc dust. After making up to a known volume and filtering, a given fraction of this was taken and titrated with silver solution,

according to Volhard's method (J. für Gasbeleucht. 1892, 221). Blank estimations were made, to ascertain the allowance to be made for impurities in the reagents. The air above the revivifying floor, with oxide fouled once or twice only, was found to contain 0·00066 and 0·00014 in two tests; and with oxide fouled 13 to 14 times, 0·00012 vol. per cent. of hydrocyanic acid. With foul oxide which had lain some time on the floor, only 0·00002 was found. The quantity which renders air harmful does not seem to be recorded, but Weyl states that in the cyanide industries, where the quantity in the air must be much greater than the above, cases of cyanide poisoning are very rare. Injury to health from hydrocyanic acid in the air of purifying houses appears improbable.

Lead acetate paper scarcely indicated traces of sulphuretted hydrogen even on prolonged exposure to the air above the revivifying floor. This air proved, by aspiration through heated platinised asbestos, to contain total sulphur compounds equivalent to 0·00003 vol. per cent., or 0·196 mgrm. per cubic metre of carbon bisulphide. Weyl states that 0·5 to 0·8 mgrm. per litre is not injurious.

Carbon monoxide was sought for in the air of a freshly opened purifier. Samples of the air were collected by displacement of water, and the carbon monoxide therein was estimated by drawing the air through strong solution of potash and a glowing platinum capillary into standardised solution of baryta. The results of five tests showed 0·006, 0·027, 0·038, 0·038, 0·032 vol. per cent. of carbon monoxide in the air. W. Hempel's researches show that 0·043 per cent. does not cause symptoms of poisoning. It is not a cumulative poison, as the hæmoglobin compound of carbon monoxide is not very stable.—J. A. B.

Sewage Water, Purification of, by Filtration through Peat. G. Frank. Gesundheits-Ing. 1896, 345 *et seq.*, and Proc. Inst. Civil Eng. 1897, **128**, [2], 27.

The author considers that the unsatisfactory results hitherto obtained by the filtration of sewage water through peat as a means of rendering the sewage available for manurial purposes, is probably due to the air which is always present in finely divided peat. The peat should be reduced to a fine state of division under water, and be there brought to the condition of sludge, which latter proves to be an excellent filtering material. It was found that the bulk of the impurities and germs were removed from sewage water by filtration through this material, and that the resultant peat sludge was possessed of considerable manurial value. The filtered effluent was sufficiently pure to be discharged into a river.—A. S.

PATENTS.

Sewage Purification for Country Houses, Impts. in. A. H. Constable, Penhurst, Kent. Eng. Pat. 19,056, Aug. 28, 1896.

The sewage is discharged through two underground tanks having V-shaped bottoms, and the upper portions of both having a division or tongue in the centre at right angles to the direction of flow. A chamber between these tanks contains a small water-wheel or turbine, a receptacle for lime, and a screw for mixing the lime with the effluent. The crude sewage flowing into the first tank and under the partition, deposits most of its suspended matter. When the liquid has risen to a certain height, some of the clear upper portion is discharged, by means of a siphon, through the intermediate liming chamber into the second tank, in which the lime precipitate deposits, and the clarified effluent flows away.—L. A.

Tanks for the Precipitation of Sewage and Foul Liquids, Impts. in and relating to. The Magnetite Sewage and Water Purification Company, Ltd., and G. H. Skelsey, Manchester. Eng. Pat. 21,235, Sept. 25, 1896.

The improvements are intended to facilitate the removal of the sludge. The lower part of the tank is formed into two or more parallel chambers having sloping sides, which conduct the sludge to a point at which a branch pipe opens, through which the sludge can be drawn off. Each branch pipe communicates through a valve with a main pipe, so that the chambers can be emptied singly or together. The sewage flows through the tank from chamber to chamber, and is deflected by suitable baffles and partitions.—L. A.

Sewage or other Liquids, Impts. in Apparatus for the Treatment of. D. Cameron, F. J. Commin, and A. J. Martin, Exeter. Eng. Pat. 23,012, Oct. 17, 1896.

The improvements comprise (1) Means for discharging the contents of a tank along an extended line, consisting of a slotted or perforated pipe placed at right angles to the line of flow: (2) Means for the removal of mud, consisting in sloping the floor of the tank to a line or lines along which slotted or perforated discharge pipes are laid, and providing other perforated pipes for flushing the mud towards the discharge pipes; (3) Making the precipitating tanks wedge-shaped, the inlet for sewage being at the narrow end and the outlet at the broad end, thus avoiding eddies and reducing the rate of flow towards the outlet, so as to promote the subsidence of the precipitate; (4) Automatic mechanism for discharging the effluent from a tank intermittently on to a filter bed, consisting of a bucket at the end of a lever which descends when full, and raises the discharge valve; also automatic gear discharging the contents of a number of tanks in rotation; (5) Automatic mechanism for distributing the effluent evenly over the surface of a filter bed by delivering it in successive fractions through a number of separate channels distributed over the surface of the filter bed, consisting of a water-wheel and gear, which causes valves connected with the different channels to open and shut in rotation, or a revolving basin which discharges successively into channels radiating from the basin as a centre.—L. A.

Sewage and the like Precipitating and Filtering Tanks, Impts. in. J. E. Bennett and H. Beresford, Manchester. Eng. Pat. 1391, Jan. 19, 1897.

The precipitating tank is provided with a number of inclined partitions, which conduct the precipitate or sludge into hopper-shaped depressions at the bottom, from which it can be drawn off through siphon pipes. Suspended in the water at the far end of the precipitating tank is a smaller tank or box, the bottom of which is a filter or strainer, through which the effluent rises into the box and overflows through a flexible pipe into another small tank suspended outside the precipitating tank. The two small tanks are connected by a chain, which passes over two pulleys; and, by means of suitable automatic mechanism when the outside tank has become full of clear effluent, it descends, raising the filter tank and causing the clear liquid in it to flow back through the filter into the main tank, thus detaching the solid matter which has accumulated on the under side, and cleansing the filter. When the outside tank has descended a sufficient distance, a valve in the bottom is automatically opened and the liquid is discharged. The filter tank, which is then the heavier, descends again to its former position.—L. A.

Treatment of Water or other Liquids with Powdered Materials, Impts. in Apparatus for the. Maignen's Filtre Rapide and Anti-Calcaire Company, Ltd., and A. A. Ellis, London. Eng. Pat. 5823, March 4, 1897.

In existing apparatus, where mechanism actuated by a water-wheel is made to discharge from a receptacle a given quantity of dry powder at regular intervals into the water to be treated, it is found that the quantity of powder discharged gradually increases as the receptacle gets empty, because the resistance to the movement of the stirrer decreases. To remedy this defect, the receptacle is made in the form of a tall cylindrical box having several orifices one above another, each provided with a regulating shutter. Inside the box is a vertical spindle having ejector arms, opposite to each orifice, actuated by the water-wheel. When the box is quite full of powder and the resistance to the movement of the stirrer greatest, the powder is ejected from every orifice; but as the level of the powder falls and the decreased resistance causes the speed of the stirrer to increase, the number of orifices from which the powder is discharged becomes less, and by a proper adjustment of the

sizes of the openings the amount of powder ejected may be made exactly proportional to the quantity of water passing over the wheel.—L. A.

Sterilising, Disinfecting, and Similar Purposes, and for cultivating Bacteria; Impts. in or relating to Apparatus for use in. E. Fournier, Paris. Eng. Pat. 7888, March 26, 1897.

The apparatus consists of a boiler or digester having a loose cover fixed in position by means of hinged bolts and thumbscrews, with an adjustable safety valve and warning whistle. It contains water, which is heated by a spirit lamp with two burners—one large, for rapidly heating up, and the other smaller, for maintaining the desired temperature. Both burners have arrangements for adjusting the size of flame. The articles to be sterilised are placed in receptacles which fit loosely within the digester, and are either closed for dry sterilisation, or of gauze for direct steam contact. The temperature depends upon the pressure, which may be several atmospheres and is regulated by the safety valve. In using the apparatus for fumigation with formaldehyde, &c., a special inner vessel is used, in which the disinfectant vapours are produced under pressure and ejected through a pipe and cock having two independent passages—one enabling steam to pass from the digester for the purpose of heating the tube conveying the disinfectant vapours, the other passage being in communication at one end with the vapour vessel and at the other end with a ball, in which condensed vapours may collect, and with a flexible continuation, the end of which may be introduced into any suitable aperture for distributing the disinfecting vapours.
—L. A.

(C.)—DISINFECTANTS.

Paraformic Aldehyde as an Antiseptic. B. H. Paul and A. J. Cownley. Pharm. J. 1897, **59**, 107.

Since the solid polymer of formaldehyde is readily converted into the liquid state by simply heating with water, it is suggested that, for convenience in handling and transport, it might, with advantage, be prepared commercially in this condition. It was found that, when boiled with water under an inverted condenser, in the proportion of 4 : 10, solid paraformaldehyde was converted into the liquid condition in about two hours. Under pressure in a sealed tube, the time taken to effect this transformation was rather longer. Paraformaldehyde is fairly volatile in the air at ordinary temperatures, about 50 per cent. volatilising in 24 hours.

For the quantitative determination of formic aldehyde in either condition, the authors find the gravimetric determination of the amount of hexamethylene tetramine formed on the addition of ammonia to formaldehyde, yields the most satisfactory results.

The ammonium compound is evaporated until viscid, and then dried to constant weight under the desiccator. On exposing an aqueous solution of formaldehyde to the air until it dries up, there appears to be formed, not a true para compound, but a mixture somewhat resembling the substance $(CH_2O)_2$ described by Tollens and Mayer as being formed when formic aldehyde is evaporated over sulphuric acid. The authors found that this body melts at about 131° C., and is more soluble in water. The polymer obtained by adding sulphuric acid to liquid formaldehyde melted at 170° C. The variation in the melting point ascribed to paraformaldehyde is probably due to the presence of an admixture of these two bodies.—J. O. B.

PATENTS.

Formic Aldehyde, Impts. in the Production of Vapours of, and Apparatus for the same. J. J. A. Trillat, Paris, France. Eng. Pat. 20,773, Sept. 19, 1896. (Date claimed under Internat. Convention, Feb. 25, 1896.)

A process and apparatus are claimed for the production of formaldehyde vapours from the solution, without polymerisation, by heating in an autoclave with a neutral salt (calcium chloride) to a pressure of three or four atmospheres and then discharging the vapours.—A. C. W.

Dressing or Wash for Hop and other Plants, Improved Compositions for use as. B. S. Dyer, London. Eng. Pat. 9566, May 5, 1896.

Picric acid (½ to ¾ lb.), soft soap (5 lb.), and "liver of sulphur" (¾ lb.) are made into a paste with a little water and diluted with more water to 100 galls. The proportions may be varied.—L. A.

Destroying Insects on Plants, Impt. in Preparations for, and for Similar Purposes. E. C. Hughes, Manchester. Eng. Pat. 16,773, July 29, 1896.

The preparation claimed, is a mixture of nicotine with ground mineral substances (sand, silicious earth, &c.), and is used in a dry state for the purpose of fumigation and also for mixing with the soil.—L. A.

Formic Aldehyde Vapour, Improved Means of Distributing. La Société Chimique des Usines du Rhône, Lyons, France. Eng. Pat. 20,622, Sept. 17, 1896.

The claim is for the means of distributing formaldehyde vapour, which consists in mixing the aldehyde solution with a liquid of very low boiling point (such as ethyl chloride) without chemical action on the aldehyde, and in enclosing the mixture in suitable vessels provided with a jet.—A. C. W.

XIX.—PAPER, PASTEBOARD, Etc.

Millboard for Bobbins. Papier Zeit. 1897, **22**, [29], 1018.

For the making of bobbins which do not scale or split, the process given is as follows:—Sheets of millboard to make the required thickness are pasted together with a mixture of starch paste and 30 per cent. of water glass, slightly dried, punched out, and treated with a quick-drying oil. The discs for making the bobbins thus obtained, are subjected to hydraulic pressure for about 24 hours, by which they are rendered very hard. They are then turned in a lathe. No intense heat is used, and the discs thus made, it is said, do not split.

The millboards used are not sized at all, and the paste used should be spread as evenly as possible, the pasted boards being well dried.—S. P. E.

Paper, Mechanical Testing of. S. Ferenczi. Zeits. angew. Chem. 1897, 436.

In order to test the capacity of a sample of paper to bear rubbing against itself and to resist crumpling, Pfuhl has devised a mechanical arrangement. The strip of paper is fastened at one end on the outside of a metal cylinder which rotates within a semi-circular sheet of rubber clamped to a similarly shaped metallic casing. Into the space between the rubber and the case, air is driven with a pump, and the pressure is registered by means of a gauge. As the inner cylinder is rotated, the paper doubles back on itself and is squeezed between the two surfaces, the operation being either continued till the strip is destroyed or the air pressure increased until the same effect is attained. Tested in this instrument, the six classes into which the Prussian Paper Testing Institute divides paper according to its power of resisting hand-rubbing, will bear pressures of (1) over 70, (2) 18—70, (3) 26—48, (4) 16—26, (5) 6—16, and (6) ½—6 cm. of mercury when the saturation of the atmosphere is 65 per cent.; but the inventor prefers not to use the pump above 48 cm., but rather to repeat the rotation of the cylinder.

Schopper has also constructed an apparatus for the same purpose, in which a strip of paper 115 mm. long and 16 mm. wide is stretched by springs horizontally at a tension of 0·5 to 1 kilo. At the middle of its length it passes between two pairs of guide rollers, and through a slot cut in a bar of metal 5 mm. thick, which is driven backwards and forwards at right angles to the paper by means of a hand-wheel and eccentric pin. The number of transverse thrusts, recorded by a counter, which the sample will bear before breaking, varies from 0 to 800 on the above scale.—F. H. L.

PATENTS.

Paper Pulp and the like, Impts. in or relating to the Manufacture of Articles from. E. C. de Segundo, Middlesex. Eng. Pat. 15,601, July 14, 1896.

Hydraulic moulds, having fixed and movable sections and operating normally to the surface of the pulp, are used to form articles from pulp, &c. For ornamented articles the faces of the moulds are grooved or sunk as required. The pulp may be heated whilst under pressure by having the moulds steam-jacketed. The water from the pulp is got rid of by perforating the moulds, or by forming the faces of the mould with wire-gauze laid over grooves or escape channels. An important feature is that the guides of the sections are not allowed to present sharp angles to the surface of the article being formed, but are bevelled off or otherwise arranged so as to form clearances. For hollow articles, collapsible cores may be used.

Starch or other binding material may be used with the pulp, the heating of which would firmly bind the material together.—S. P. E.

Paper, Cardboard, and Similar Materials; Impts. in and connected with Superficially Coloured. C. H. Voigt, Leipzig-Plagwitz, Germany. Eng. Pat. 12,301, May 18, 1897.

The improvement consists in the use of a new composition to serve as "binding material" in place of the size, consisting mainly of glue, hitherto employed to mix with the pigments. By the use of this new composition it is said the otherwise necessary sizing of the paper previous to the application of the film of colour, may be dispensed with, and the subsequent printing of several colours on the same sheet, is facilitated. The composition is prepared as follows:—

About 100 parts of starch or other mucilaginous vegetable substance, such as agar-agar or carragheen, and about 2 per cent. of solid alkali hydrate are mixed with 300—500 parts of water, and subjected to pressure at a temperature ranging between 110°—200° C. until a uniform mass has been produced. The colouring matter is subsequently incorporated in this compound, to which wax or soap may be added if required.—I. S.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

Monazite. H. B. C. Nitze. J. Franklin Inst. 1897, **144**, 127—133.

The development of incandescent gas lighting caused an increase in the output of Carolina monazite sand from 130,000 lb. in 1893 to 1,900,000 lb. in 1895. The output fell, however, in 1896 to 17,500 lb., owing to the increased competition of the Brazilian field, where the sand occurs naturally concentrated on the beach, requires no cleaning, and may be loaded directly into ships and carried as ballast. Monazite is essentially a complex tribasic phosphate of cerium, lanthanum, and didymium, with variable percentages of thoria and silica. The two last-named are believed by some to be present in combination, whilst others assume that the thoria is present as a constituent of the phosphate. The percentage composition of four samples from the United States ranged as follows:—P_2O_5, 26·1 to 29·3; Ce_2O_3, 29·9 to 37·2; La_2O_3, 26·6 to 31·6; ThO_2, 1·5 to 14·2; SiO_2, 0·3 to 2·8; and H_2O, 0·2 to 0·7 per cent. The value depends mainly on the percentage of thoria (as that element is chiefly required for incandescent lighting), and this varies from 4 to 7 per cent. in the best Carolina grades. The mineral usually crystallises in monoclinic tables, short columns, or small needles, from 0·05 to 1 in. long, but occasionally attaining to a length of 5 ins. It is brittle, has a hardness of 5—5·5, and a specific gravity of 4·6—5·3; the lustre is resinous to waxy, bright in fresh crystals, but weathering dull, or even becoming coated with a light brown earthy substance. When pure it is transparent, but it is translucent or opaque in the more impure specimens, and it has a yellow, brown, or greenish-yellow colour. It occurs in granitic rocks and varieties of gneiss, and has been found in apatite, cyanite, and vein quartz. The main constituents of granite may all contain monazite as intergrowths, but it is generally confined to the felspar. Zircon is almost always concomitant. In the United States it is found only in North and South Carolina, within an area of about 2,000 sq. miles. Most of the stream deposits have been worked for gold. The gravel beds are about 1 to 2 ft. deep, and rarely exceed 12 ft. in width; they contain from almost nothing up to 1 or 2 per cent. of monazite. In Brazil the principal monazite (beach) sand is on the island of Alcobaca, to the extreme south of Bahia, but the mineral is also found in the gold and diamond placers of Bahia, Minas Geraes, Rio de Janeiro, and Sao Paulo. It also occurs in the placers of Rio Chico (U.S. of Colombia), of Bakakui (Sanarka river, Ural mountains), and in Siberia.

In Carolina the monazite is obtained by washing the sand in sluice-boxes, after the manner of placer gold. These boxes are 8 ft. long by 20 in. wide and deep, and require the service of two men—one to charge the gravel on to a perforated plate at the upper end, the other to agitate the contents with the aid of a gravel-fork or perforated shovel, in order to float off the lighter sands. The boxes are cleaned out periodically, and should yield about 40 to 70 lb. of cleaned monazite each *per diem*. Two or more boxes may be used in succession at different levels. Magnetite is eliminated from the dried sand by means of a large hand-magnet, but many heavy minerals (zircon, garnet, rutile, ilmenite) cannot be separated by these methods, and there may be 30 to 35 per cent. of foreign matter left even in good samples of washed monazite. The grade may often be improved up to 85 per cent. monazite by a system of winnowing, &c. But the cost of such processes is considerable, and the gain is not always commensurate. The author has produced a clean product with only an insignificant loss in the tailings by jigging, and then passing the jig concentrates over a Wetherill magnetic separator. Any gold in these concentrates could be recovered by amalgamation in riffle boxes. The working of decomposed rock *in situ* is not usually remunerative, on account of the small proportion of monazite present, unless the rock be at the same time gold-bearing.—W. G. M.

Monazite in Idaho. W. Lindgren. Eng. and Mining J. 1897, **64**, 69. From the Amer. J. of Science.

A sample of sand washed from the lake beds near Idaho City, consisted of: Ilmenite in sharp hexagonal crystals, but no magnetite; zircon, also in extremely sharp crystals of a slightly brownish colour; and abundant yellow or greenish-yellow grains rarely showing crystallographic faces; the refraction and double refraction of this mineral were very high, the hardness not much over 5. The ilmenite was eliminated by the electro-magnet, and an analysis by W. F. Hillebrand, of the remaining powder containing about 70 per cent. of the yellow mineral, showed that it was a phosphate of the cerium metals, the approximate amount of the oxides of the latter being 48 per cent.; in these about 1·2 per cent. of thoria was found. Another sample from the alluvial gold-washing in Wolf Creek, Placerville, when cleaned from quartz, &c., appeared as a heavy dark sand, consisting of a black iron ore (ilmenite), rounded crystals of red garnet, sharp crystals of zircon, and irregular grains of a dark yellowish-brown mineral with a waxy lustre, sometimes showing crystallographic faces. There was practically no magnetite present. On a qualitative analysis of the sand, W. F. Hillebrand found present phosphoric acid, cerium metals, and thorium. The yellowish mineral is therefore in all probability monazite. Although the monazite occurs in considerable quantity, it is doubtful whether the mineral can be profitably extracted, except possibly as a by-product obtained from the gold washings.—A. S.

Arsenic, Some Soluble Compounds of. G. G. Henderson. Brit. Pharm. Conference, Aug. 1897. Pharm. J. 1897, [1416], 146.

Methods of preparation are described of the soluble combinations of arsenic with the acid tartrates and citrates of the alkali metals. These compounds are analogous to tartar emetic, the antimony being replaced by arsenic.

Sodium arsenio-tartrate, $AsO.NaC_4H_4O_6.2\frac{1}{2}H_2O$, crystallises from water in groups of colourless prisms, from 50 per cent. alcohol in colourless plates. It loses its water of crystallisation when heated to 105° C., or when left to stand over sulphuric acid in a vacuum, but the dried salt may be heated for several hours at about 200° C. without decomposing. It is easily soluble in water, and the solution, which is very poisonous, has a sweet taste and is of an acid reaction. The physiological action of this salt is practically the same as that of "liquor arsenicalis." Ammonium arsenio-tartrate crystallises in small lustrous needles, readily soluble in water, but less stable than the sodium salt. The potassium salt forms a white crystalline powder, which is decomposed by water into arsenious oxide and potassium hydrogen tartrate. The arsenio-citrates form colourless crystals, easily soluble in water and fairly soluble in dilute alcohol; these salts decompose when heated in aqueous solution.

—A. S.

Conium Maculatum. Note on the Pharmacy of. E. H. Farr and R. Wright. Brit. Pharm. Conference, Aug. 1897. Pharm. J. 1897, [1416], 136.

The authors prepared standardised products of conium and solutions of its alkaloids for physiological examination. A fluid extract of dried unripe fruits was prepared by successive percolation, and was standardised so as to contain 2·5 per cent. of total alkaloidal hydrochlorides. A corresponding solution of the mixed alkaloids of the same fruits was prepared in a similar manner, the alkaloids being extracted from the percolate; this solution was made up to the same strength as the preceding one. Succus fruct. conii was obtained by maceration and pressing, using first rectified spirit and then a mixture of spirit and water, and the extract was found to contain 0·7 per cent. of total alkaloidal hydrochlorides. Solutions were also prepared of pure conine hydrochloride, conhydrine hydrochloride, and of pseudoconhydrine hydrochloride.—A. S.

Conium Maculatum. Certain Preparations and Active Priciples of; Note on the Action of. W. Findlay. Brit. Pharm. Conference, Aug. 1897. Pharm. J. 1897, [1416], 137. (See preceding abstract.)

The author, on examining the preparations described in the preceding abstract, found that conine and the mixed alkaloids are the most toxic, the lethal dose being, for conine, 37 mgrms. per kilo. of body weight; for the mixed alkaloids, 39 mgrms.; for conhydrine, not less than 257 mgrms.; and for pseudoconhydrine, above that quantity. The fluid extract appears to be as active as conine. The results obtained with the succus were uncertain, on account of its low percentage of alkaloids.—A. S.

The Aloïnes. E. Leger. Comptes Rend. 1897, **125** 185—188.

From different species of aloes, crystalline substances have been extracted; to these, names have been given recalling the country of origin. According to Groenwold (Arch. der Pharm. 1890, 115), barbaloïne (Barbadoes) and curaçaloïne (Curaçao) are identical; whilst Tilden (Journ. Chem. Soc. 1872, 205; 1877, 264) considers barbaloïne, socaloïne, and zanaloïne as probably identical.

The author extracted Barbadoes aloes with acetone and glacial acetic acid, and from the residue extracted the aloïne by methyl alcohol, some being also obtained from the acetone solution. After drying *in vacuo* over sulphuric acid, the analytical results accorded best with the formula of Groenwold, $C_{16}H_{16}O_7 + H_2O$. Barbaloïne crystallises from water with $3H_2O$ in yellow needles. A diacetyl derivative was obtained. When barbaloïne once re-crystallised from methyl alcohol is again allowed to separate from that solvent, the mother-liquors on concentration deposit yellow plates; these contain 14—15 per cent. of water and consist of a new aloïne isomeric with barbaloïne. The existence in the same aloes of these two isomers crystallising with 1 and 3 molecules of water, probably explains the differing content in water of crystallisation found by certain authors. Socaloïne and zanaloïne may be mixtures of these two aloïnes in varying proportions.

—A. C. W.

Pilocarpine and Pilocarpidine, Isomerism of. A. Petit and M. Polonovski. J. Pharm. Chim., 6 ser., **6**, (1), 8.

From the results of their previous researches the authors have been led to the conclusion that pilocarpine and pilocarpidine are isomeric forms of the same body, and therefore to doubt that the transformation of pilocarpine into pilocarpidine is accompanied by the production of methylic alcohol, as stated by Hardy and Calmels and others (this Journal, 1887, 675). Further experiments confirm their views, and show that, although aqueous solution of caustic soda affects the transformation of pilocarpine into pilocarpidine, no trace of methylic alcohol is formed during the process, and therefore that there can be no elimination of a methyl group. It is found also that the hydrochloride of pilocarpine, if fused for a few minutes at 200° C., is wholly transformed into pilocarpidine hydrochloride without any appreciable loss in weight. After this treatment the salt has all the physical and chemical properties of pilocarpidine, thus proving that pilocarpine is transformed into pilocarpidine without any chemical alteration, and that the change of characters is due entirely to molecular rearrangement, the nature of which has not been at present determined.—J. O. B.

Pilocarpine and Pilocarpidine Chloroplatinates, Further Note on. A. Petit and M. Polonovski. J. Pharm. Chim., 6 ser., **6**, (1), 11.

The following further particulars concerning the chloroplatinates of pilocarpine and pilocarpidine are given. Pilocarpine chloroplatinate occurs as a bright yellow precipitate, which crystallises from boiling water in large orange lamellæ, readily soluble in boiling water, but only slightly so in cold water. In water at 23° C. the solubility is 0·8 in 100. The salt contains no water of crystallisation, and melts at 213° C.

Pilocarpidine chloroplatinate has the same crystalline form as the preceding salt, but is much less soluble in boiling water, and has just half the solubility in cold water, the figure for 23° C. being 0·4 in 100. It, too, is anhydrous. It melts between 222° C. and 224° C. The determination of the platinum gave precisely the same figures from both salts.

—J. O. B.

"Loretin," m-Iodohydroxyquinoline Sulphonic Acid, Notes on. Ad. Claus and S. Baumann. J. für prakt. Chem. **55**, 17.

The authors combat the prevalent theory that loretin $(C_9H_4N.(OH)I.HSO_3)$ and its combinations owe their germicidal action as surgical dressings to the liberation *in situ* of iodine in a nascent condition. No instance of the characteristic eruption produced by free iodine has been recorded during the whole period, extending over 15 years in which loretin has been employed, nor has iodine ever been detected in the urine of patients treated with it. The authors find that loretin compounds are quite stable, the dry salts always requiring to be heated to over 200° C. before decomposition sets in. The only instance in which free iodine is liberated at normal temperatures is when the aqueous solution is exposed to sunlight—a condition which never occurs in clinical practice. The only change which the loretin molecule appears to undergo in ordinary use would seem to be the combination of a molecule of water with the hydrogen atom, and the simultaneous production of hydrogen peroxide and hydriodic acid, the latter being immediately recombined; or it may be that the loretin molecule, as a whole, without undergoing decomposition, combines with septic matter and renders it harmless.

The authors have prepared neutral and basic salts of calcium, strontium, barium, and magnesium, with loretin. The normal calcium salt prepared by precipitating a solution of a normal loretinate of an alkali with calcium chloride, forms a heavy orange crystalline precipitate, which, from its property of adhering to gauze or wool, renders it suitable for the preparation of antiseptic dressings.

By pouring an almost saturated alcoholic solution of calcium chloride on to the surface of a $1-1\frac{1}{2}$ per cent. solution of sodium loretinate, and leaving them in contact, calcium loretinate crystallises out in fine red rhombic prisms,

which have trichroic properties. When heated, these lose their characteristic colour and become yellow. Basic calcium loretinate is obtained by treating a very dilute solution of basic alkaline loretinate with calcium chloride solution. The basic lime salt slowly forms cream-coloured needles.

The strontium salts closely resemble those of calcium, but the normal barium salt is much more soluble than either of them, and crystallises in red needles. Basic barium loretinate forms greenish-yellow needles. The normal salt of magnesium was unstable and very sensitive to sunlight; it crystallises in opaque, flat prisms. Basic magnesium loretinate was more easy to isolate, being more stable. It forms yellow prisms containing 5 mols. of water. —J. O. B.

Citronella, Commercial Oil of. J. C. Umney and R. S. Swinton. Brit. Pharm. Conference, Aug. 1897. Pharm. J. 1897, [1416], 138—139.

The authors contradict the statement that the differences in specific gravity, optical activity, and other properties, between the oil of citronella derived from native sources in Ceylon, and that distilled by two English firms there, is due to adulteration with a wood oil. It was found that these differences are due to the presence in the oil distilled by the natives, of a sesquiterpene, to the amount of 37 per cent., which does not distil over in a current of steam like the products of the English firms, and which raises the specific gravity; and also of a highly active terpene which raises the optical rotation. The presence of these constituents reduces the odour value and impairs the solubility in alcohol. —A. S.

Petroleum, Medicinal. [Presence of Sulphur Compounds.] F. C. J. Bird. Brit. Pharm. Conference, Aug. 1897. Pharm. J. 1897, [1416], 153—154.

White petroleum (sp. gr. about 0·885) can be obtained more free from taste and odour than the lighter oils, but most of the commercial oil, much of which is sold as chemically pure, contains a certain proportion of sulphur compounds, probably introduced during the process of bleaching. White petroleum jelly is frequently a mixture of ceresine and white oil, and contains the impurities of the latter. Very pale jellies, which are true non-crystalline petroleum residues, also usually contain sulphur due to the bleaching process. Yellow oils, though generally free from sulphur, are in many cases so tainted with a "paraffine" flavour as to be unfit for internal use. The yellow petroleum jellies are the purest as far as sulphur is concerned. It was found that, as far as could be traced with certainty, the Russian oils contained sulphur, whilst the American oils were free. The reduction test for sulphur with zinc and hydrochloric acid is recommended as convenient, easily applied, and delicate. 1 drm. of the sample, and 0·5 drm. of absolute alcohol, and 15 minims of pure hydrochloric acid are shaken in a test tube with a piece of pure zinc. The presence of sulphur is indicated by the brown or black coloration produced on a strip of paper, moistened with subacetate of lead, which is suspended in the upper part of the tube.—A. S.

Hypophosphites. C. T. Tyrer. Brit. Pharm. Conference, Aug. 1897. Pharm. J. 1897, [1416], 150.

See under XXIII., *page* 764.

Chloral Hydrate Reaction with Ammonium Sulphide. J. Lesinsky and C. Gundlich. Amer. Chem. J. 19, 603.

See under XXIII., *page* 765.

Digitalis, Determination of Active Principles of. C. C. Keller. Ber. Deutsch. Pharm. Ges. 7, 125; through Pharm. J. 4, [1413], July 24, 1897, 62.

See under XXIII., *page* 767.

Vanillin-Para-Phenetidine. C. Goldschmidt. (Ger. Pat. 91,171.) Pharm. Centralh. 38, 353.

The patentee obtains a combination of paraphenetidine with vanillin by heating the two bodies together to about 140° C. The constitution is stated to be—

$$C_6H_3(OH)OCH_3.CH:NC_6H_4OC_2H_5.$$

When recrystallised from hot water it melts at 97° C. It is said to be hypnotic and anti-neuralgic as to action. It is also soluble in water.—J. O. B.

Tetra-Iodo-Ethylene (Di-Iodoform), Preparation of. Biltz. Ber. 1897, 30, [10], 1200; through Pharm. Zeit. 42, [52], 448.

Tetra-iodo-ethylene, which has been recommended as an iodoform substitute on account of its freedom from odour, may be prepared as follows:—50 grms. of iodine are dissolved with heat in a saturated solution of iodide of potassium and the solution cooled to 0° C.; small pieces of coarsely powdered calcium carbide are now cautiously added, in small quantities at a time, the solution being kept well stirred. The reaction is represented by the equation—

$$C_2Ca + 4I = C_2I_2 + CaI_2$$
$$C_2Ca + 6I = C_2I_4 + CaI_2.$$

Tetra-iodo-ethylene forms yellow non-volatile needles melting at 187°. It is less soluble than the accompanying di-iodo-acetylene, which occurs in colourless volatile needles, which are readily soluble in most solvents. The author states that di-iodo-acetylene is a powerful germicide, in which property it is only surpassed by the extremely poisonous cyanogen iodide. Tetra-iodo-ethylene has the same action on wounds as iodoform, but it is an odourless substance.—J. O. B.

Iodoformin and Iodethylformin, Preparation of. Bocquillon Limousin. Méd. nouv. 1897; through Pharm. Cent. 38, 457.

Trillat prepares iodoformin, $C_6H_{12}N_4I_4$, by first mixing together ammonia and formaldehyde in molecular proportions and then adding an alcoholic solution of iodine, or a solution of iodine in potassium iodide, to the solution of hexamethylene tetramine so formed. Iodoformin falls as a yellowish-brown crystalline pulverulent precipitate, which is collected, washed, and dried. It is insoluble in the cold in water, alcohol, ether, benzene, or chloroform; soluble in acetone, and sparingly dissolved by boiling alcohol. Boiling water decomposes it into iodine and formaldehyde, and it is decomposed in a similar manner by long standing at 40° C., with weak alkaline solutions. It contains 80 of iodine.

Iodo-ethylformin, $C_6H_{12}N_4.(C_2H_5I)_2$, is obtained by the action of ethyl iodide on a weak alcoholic solution of hexamethylene-tetramine. It crystallises in long, slender, colourless needles readily soluble in water, the solution being almost tasteless. It is sparingly soluble in alcohol, and insoluble in ether and in chloroform. Sodium carbonate decomposes it, with the formation of sodium iodide, a small quantity of ammonium carbonate, and formaldehyde. Strong acids also liberate formaldehyde from it. It has been prescribed for internal use by Bardet as a substitute for alkaline iodides.—J. O. B.

Iodoform, Decomposition of, by Light. G. Fleury. J. Pharm. Chim. 1897, 6, [3], 97.

The author confirms the prevailing opinion that when solutions of iodoform are exposed to the action of light, decomposition, accompanied by the liberation of free iodine, only takes place up to a certain point. This he attributes to the fact that the reddish-brown solution of iodine so formed, prevents the passage of the ultra-violet rays, and so obviates further decomposition. It is found that when an ether-alcohol solution of iodoform, to which an excess of reduced silver is added, is placed in a white glass flask and exposed to sunlight, either direct or diffused, in the course of a few days practically the whole of the iodoform is decomposed, and the amount of silver iodide found approximates to the theoretical quantity of iodine contained in the iodoform. It is evident, therefore, that when the iodine liberated by the action of sunlight on iodoform is removed as it is formed, the decomposition is virtually complete.—J. O. B.

Bitter Fennel, Oil of. E. Tardy. J. Pharm. Chim. 1897, 6, [3], 98.

The oil of cultivated bitter French fennel is found to contain: (1.) A dextro-rotatory terpene. (2.) Phellandrene-

(3.) Cymene. (4.) Fenchone (termed by the author "fenone"). (5.) Estragol. (6.) Anethol. (7.) Anisic aldehyde. (8.) Anisic acetone. (9.) Anisic acid. (10.) An undetermined crystalline body having the empirical formula $C_{13}H_{14}O_2$, the constitution of which has not yet been ascertained.

The oil obtained from fennel cultivated at Grasse had a specific gravity at 0° C. of 1·007; the rotation index for the 100 mm. was a_D + 18° 20′. The anisic acid was removed by washing with aqueous potash, after which, on treatment with sodium bisulphite, the aldehydes and ketones were precipitated. These were set free by treating the purified precipitate with potash, and the oily liquid thus obtained was submitted to fractional distillation. The two chief fractions distilled between 246°—250° C. and 260°—265° C. The former was anisic aldehyde and the latter anisic acetone; on oxidation with permanganate in aqueous solution, this gave anisic and acetic acids. It appears to have the constitution $C_6H_4(OCH_3)(CH_2.COCH_3)$. In fractionating these bodies a crystalline substance came over between 270°—280° and formed a butter-like mass, which, when recrystallised from chloroform, melted at 213° C. The author assigned to it the formula $C_{13}H_{14}O_2$. It is possible that it is a coumarin-like body, formed by the action of the anisic and acetic acids, liberated during distillation, on the aldehydes. The other bodies enumerated above were obtained from the residual oil, after treatment with bisulphite, and were separated from each other by fractional distillation, and identified by their specific reactions.—J. O. B.

Certain Arums, Active Principles of. A. Hébert and F. Heim. Comptes Rend. **124**, 1368.

The plants examined were *Arum maculatum*, *A. italicum*, and *Arisarum vulgare*. In all, the active principles were found to be in very small quantity. They consisted of—

(A.) An *alkaloid* obtained in the proportion of 0·005 per mille. This was isolated in the form of a brown oily volatile liquid having an odour of mice, strongly alkaline in reaction, and fuming when approached by the stopper of a hydrochloric acid bottle. In chemical and physical properties it closely resembled conine; it differed from that alkaloid, however, in its physiological potency, the effects produced on the living organism being similar to those of conine, but less intense. It is, however, an active poison; 100 kilos. of the fresh plants yielded from 4 to 5 grms. of this body.

(B.) A *saponin*, found in the greatest quantity in the resting tubers, where it amounted to 1 per mille, although it was detected in small quantities in all parts of the plant. The toxic action of this saponin is said to be less marked than that of other sapotoxins,. When hydrolysed, it gave glucose and a sapogenin.

The authors were unable to obtain any evidence of the presence of hydrocyanic acid, which has been reported by Jorissen to be present in these plants.—J. O. B.

Oil of Turpentine [French], Detection of Rosin Oil in. A. Aignan. Comptes Rend. **124**, 1367.

See under XXIII., *page* 764.

PATENTS.

Piperazine and Polybasic Acids, The Manufacture of Acid Salts of, and the Production therefrom of Double Salts of Lithia and Piperazine. A. Koelliker, Beuel-on-Rhine, Germany. Eng Pat. 18,981, Aug. 27, 1896.

Piperazine forms acid salts with oxalic, tartaric, and citric acids; these acid salts form neutral double salts with lithium. The compounds mentioned, are claimed as new and useful products.—A. C. W.

Vinyldiacetonalkamine, The Production of a, and of Anæsthetics therefrom. Chemische Fabrik auf Actien vorm. E. Schering, Berlin, Germany. Eng. Pat. 20,697, Sept. 18, 1896.

The vinyldiacetonalkamine of Fischer (Ber. **17**, 1794) is a mixture of two bases, melting at 138° and 161°—162°; these may be separated by crystallisation of their salts or the bases themselves. The former alkamine is claimed as a new product, together with its derivatives.—A. C. W.

Vanillin, Impts. in the Manufacture of. La Société Chimique des Usines du Rhône, Lyons, France. Eng. Pat. 21,106, Sept. 23, 1896.

The separation of vanillin from mixtures with *m*-methoxysalicylic aldehyde, by treatment with calcium hydroxide (or other hydroxides) in solution or suspension, is claimed. On agitation, vanillin dissolves in the alkaline solution; the isomer is precipitated as calcium salt. The two compounds are recovered by acidifying, and extracting with ether.
—A. C. W.

Iron for Medicinal Purposes, The Manufacture of Improved Preparations of. E. Dolle, Bremen, Germany. Eng. Pat. 9701, April 15, 1897.

The manufacture of iron preparations which do not possess the unpleasant flavour of iron, by precipitating chloride of iron with phosphate of soda and combining the precipitate with one or more of certain essences and tinctures (cardamon, orange, vanilla, &c.), is claimed.—A. C. W.

Hydroxylated Ethers of Phenol [Guaiacol, &c.], Process for the Extraction of, from Mixtures. L. Lederer, Munich, Germany. Eng. Pat. 12,964, May 25, 1897.

The hydroxyphenol ethers form crystalline compounds with potassium carbonate; these are permanent in air, but partly decomposed by water, the presence of potassium carbonate preventing the decomposition. They are soluble in hot alcohol and benzene, and crystallise out on cooling. The patent claims the use of this property for the extraction of such ethers from mixtures, by treatment with potassium carbonate or its solution, or by treatment of solutions of the potassium phenolates with carbonic acid. The potassium carbonate compound is precipitated from aqueous solution by ether, and decomposed by acids.
—A. C. W.

Phenetidine-sulphonic Acid and its Salts, as also of Acetyl Derivatives of the latter [Antipyretics], Manufacture of. F. Hoffmann, La Roche and Co., Basle, Switzerland. Eng Pat. 14,375, June 14, 1897.

The process for the production of the phenetidine-sulphonic acid, which consists in the treatment of phenetidine with concentrated or fuming sulphuric acid, and purification by conversion into salts, and precipitation of the acid by the addition of mineral acid, the production of the salts by neutralisation or double decomposition, of the acetyl derivatives by heating these salts with acetic acid or anhydride, and finally the acid, its salts, and their acetyl derivatives as new compounds, are claimed. The acetyl derivatives are antipyretics easily soluble in water.
—A. C. W.

Theobromine, Impts. in the Manufacture or Preparation of. C. F. Boehringer and Soehne, Mannheim, Germany. Eng. Pat. 14,610, June 16, 1897.

In this patent the manufacture of theobromine is claimed "by transforming 3.7-dimethyl-6-amino-2.8-dioxypurine, by treatment with phosphorus oxychloride, into 3.7-dimethyl-6-amino-2-oxy-8-chloropurine, reducing the latter to 3.7-dimethyl-6-amino-2-oxypurine, and then by treatment with nitrous acid, converting it into theobromine."—A. C. W.

Trichloropurine, A Process for the Production of. C. F. Boehringer and Soehne, Mannheim, Germany. Eng. Pat. 14,612, June 16, 1897.

The process consists in heating 2.6-dichlor 8-oxypurine with phosphorus oxychloride.—A. C. W.

Isoeugenol and Derivatives thereof [for preparing Vanillin], Impts. in the Manufacture or Preparation of. C. F. Boehringer and Soehne, Mannheim, Germany. Eng. Pat. 14,611, June 16, 1897.

The patent claims "converting eugenol into isoeugenol in such a way that the free hydroxyl group of the eugenol is

protected by the introduction of the phosphoric acid radicle," and modifications of this process in which eugenol is converted into eugenol phosphoric acid by the action of phosphorus oxychloride, and the latter heated with alcoholic potash, or heated with aqueous alkali, which transforms it into isoeugenol phosphoric acid, this being converted into isoeugenol by the action of heat on the acid salt of an alkali metal.—A. C. W.

Sweetening Substance [Methylbenzene Sulphonic Imide], A New or Improved, and a Process for its Manufacture. E. R. Savigoy, London. Eng. Pat. 4467, Feb. 19, 1897.

Methylbenzene sulphimide is claimed as a new sweetening substance, and the process for its manufacture, which consists in saponifying toluene cyansulphamide with "dry potash," precipitating the methyl benzene sulphonic imide by the addition of sulphuric acid, and recrystallisation from xylene, after which it is finally obtained in a pure form by the addition of dimethylbenzene.—A. C. W.

[Saccharine.] o-Anhydrosulphamine-benzoic Acid from p-Sulphamine-benzoic Acid, Process for Separating. R. Barge, Stassfurt, Germany. Eng. Pat. 15,009, June 21, 1897.

The inventor claims the processes for the separation of the two sulphamine-benzoic acids, which consist in precipitating a basic copper salt of the para compound from a solution of the neutral salts of both, and in precipitating the copper salts together and dissolving out the *o*-sulphamine-benzoic acid by sodium hydroxide or carbonate. In the first case sufficient caustic soda may be added before precipitation to neutralise the free acid formed.—A. C. W.

XXI.—PHOTOGRAPHY.

Commercial Egg Albumin, Estimation of. P. Carles. J. Pharm. Chim. 1897, 6, [3], 102.

See under XXIII., *page* 767.

PATENTS.

Chromo-Photography, Impts. in and relating to. — Valentin Vaucamps, Paris. Eng. Pat. 20,898, Sept. 21, 1896.

Films are made by superposing sensitised layers tinted differently—in the order of the colours of the spectrum if for photography in natural colours—in any other order if for fancy pictures. One such layer is partially set by artificial cooling before the next is poured on, perfect adhesion without sensible mixture being thus secured. Such a film is exposed under a negative, red side next the negative, and, when developed, the layers are eaten away to different depths, the red being left insoluble under the light parts of the negative, while the blue layer only is affected (and hence laid bare by the removal of the superposed red and yellow by the developing solution) under the dark parts. When fixed, a picture in natural colours, visible by reflected light only, however, is obtained. To procure a picture visible by transmitted light, the fixed film (on paper) is transferred to a perfectly plane glass plate, red side downwards. The result of this is, so to speak, to transfer the reliefs from the front to the back of the film, and by grinding down these reliefs till the film is of uniform one-layer thickness, we get a picture showing by transmission the same colours which the untouched film showed by reflexion. Pictures can be mechanically multiplied by placing the finished fixed film and a similarly prepared but unsensitised film in a reproducing machine, similar to that used for musket stocks or similarly shaped pieces of woodwork.

—J. T. D.

Dishes or Baths employed in Developing and like Photographic and Chemical Operations. W. S. Wain, Ripley, Derbyshire. Eng. Pat. 21,495, Sept. 29, 1896.

An ordinary developing dish with a broad, flat bar crossing the middle of its under side, on which the dish rests. The width of this bar is so arranged that when the dish and its contents are tilted, the centre of gravity is just beyond the bar, towards the raised end. Under these circumstances, when let go, the dish rocks backwards and forwards for a while, and ultimately comes to rest in a horizontal position. Instead of the broad bar, two parallel narrow ribs (corresponding with the edges of the bar), or two short webs (corresponding with the ends of the bar), or four points (corresponding with the corners of the bar) may be used.

—J. T. D.

Sensitised Photographic Plates and Films, Impts. in. M. Levy, Berlin. Eng. Pat. 10,098, April 22, 1897.

Thin glass or celluloid plates are coated on both sides with a sensitised film. Several of these, if thin enough, may be used superposed. The time of exposure is thus shortened. Though intended originally for photographing by X-rays, the device may be used for ordinary photography.—J. T. D.

Sensitive Plate or Film, A New, and a Method of Producing, Developing, Fixing, and Toning the same. T. C. Porter, Eton. Eng. Pat. 12,922, May 25, 1897.

A gelatin or other film is immersed in solution of ferric ammonium citrate or other ferric salt for three minutes, and is then thoroughly and rapidly washed. After drying, the film is exposed under a negative, developed by potassium ferricyanide, and fixed by hydrochloric acid. The positive is then treated with very dilute ammonia solution till the greenish-blue tint has acquired a sufficient violet tinge. The pictures so obtained, are suitable for lantern slides exhibiting ice and snow scenery, sea-views, &c.—J. T. D.

XXII.—EXPLOSIVES, MATCHES, Etc.

Annual Report, for the Year 1896, of H.M. Inspectors of Explosives.

The inspectors have, happily, to chronicle only one death due to fire or explosion during manufacture.

Note is made of the continued growth of the trade in explosives, the number of factories increases—five having been licensed during the year, and several existing ones have increased their area.

In regard to the importation of nitroglycerin compounds 1,259,200 lb. were received in 1896, against 830,070 in 1895, and this amount is greater than during any previous year, with the exception of 1889, when 1,325,950 lb. were imported. The quantity of dynamite, which fell to 23,000 lb. in 1894, but then revived to 56,000 lb. in 1895, has further risen to more than three times that amount in 1896. There has been a large increase in the importation of carbonite, and it now heads the list of imported nitroglycerin explosives.

Of detonators, 9,780,000 were imported, showing that a great percentage of this trade still remains in foreign hands. There are, however, six factories in this country in which detonators are made.

While collodion cotton now appears on the list of authorised explosives, it is exempt from the conditions imposed on explosives "when it is in solution in alcohol and ether, or wet, or saturated with methylated spirit and enclosed in air-tight cases." With regard to the particular form of collodion cotton which is used in the manufacture of xylonite and kindred articles, certain relaxations in the conditions of manufacture have been granted when the collodion cotton contains not more than 11·11 per cent. of nitrogen.

Dr. Dupré's report is given in full, as usual, and in it he says that "the extremely high character shown last year among explosives proper has, however, unfortunately, not been maintained." Fourteen samples, of which 10 were gelatin dynamite No. 2, were rejected, against none in the preceding year. He also notes a falling off in the activity of inventors, as only five new explosives were submitted for approval.

Attention is again called to the danger as well as illegality of using chlorate and sulphur in coloured fires, and even of using sulphur and strontium nitrate—instances of such a mixture taking fire spontaneously having been recorded.

An explosion of ammonium nitrate due to overheating in the course of the manufacture of nitrous oxide (laughing-gas) took place in Kensington, and Dr. Dupré considers it

worthy of note "in connection with the often reiterated assertions of some manufacturers that certain explosives containing ammonium nitrate cannot be exploded by heat or fire."

An interesting accident, unattended with loss of life or personal injury, occurred at the Arklow Factory during the nitration of a charge of glycerin. When there were about 60 lb. of nitroglycerin formed, the charge fumed off, and although the connections to the drowning tank were immediately opened, it was subsequently ascertained that no nitroglycerin was among the water and acids in the drowning tank; consequently the whole of the estimated charge of nitroglycerin (60 lb.) must have fumed off in a few seconds. The cause of the accident was attributed to a small leak subsequently discovered in one of the cooling coils.

A destructive fire occurred in March at the Cotton Powder Works near Faversham, and several interesting points were noted: one, "that some 5,000 to 6,000 lb. of guncotton in water, which were exposed to the full effect of the fire, were practically uninjured, although in some cases the vats and vessels containing it were burnt away: and an even more interesting and important fact was the burning away inexplosively of over 3,000 lb. of guncotton (containing about 30 per cent. of water) made for the War Office and contained in barrels of $27\frac{3}{4}$ lb. each."

Short accounts are given of the principal accidents and outrages which occurred at home and abroad; also of the more important explosions and fires due to petroleum; and, in the latter connection, accidents are described which took place when the inside of tanks were being painted with a solution of coal-tar in crude low-flashing tar oil. Such substance ought to have been labelled "highly inflammable."

Attention is drawn to the Order, in virtue of which, after January 1, 1897, it will be unlawful to use in certain parts of coal mines any explosive other than a "permitted explosive," a list of which is given. "It is proposed to establish a Government testing station at Woolwich, at which, from time to time, explosives which may be submitted for addition to the 'permitted list,' or modifications of explosives already on that list, can be tested as to their non-liability to ignite inflammable gas or coal-dust, or admixtures of the two, and their suitability and safety for use in coal mines."—W. M.

PATENTS.

Explosives, Impts. relating to the Manufacture of. G. Kynoch and Co., and A. T. Cocking, both of Witton, Warwick. Eng. Pat. 22,190, Oct. 6, 1896.

This invention relates to an apparatus for drying cordite and the effectual recovery of the solvent employed in its manufacture. The cordite is spread on metallic and perforated removable trays, which rest on ledges in metallic connection with the walls of the vacuum drying chamber, consisting of a cylindrical cast-iron vessel surrounded by a water-jacket, the latter being heated to any desired temperature. "The acetone or other solvent vapour is driven off by the heat transmitted by conduction from the hot-water jacket and the vapour is drawn away from the vacuum chamber into a condenser." The air escaping from the condensing plant may be conveyed through a water scrubber. Drawings accompany the specification.—W. M.

Matches, A Non-Ignitible Coating applied to Common Lucifer Match Boxes and Finger Ends of. L. A. Thomas, Yarm-on-Tees, Yorkshire. Eng. Pat. 24,816, Nov. 6, 1896.

The inventor proposes to reduce "the danger from fire in storage or carriage of lucifer matches," by treating the matches and the containing boxes and wrappers with a solution of silicate of soda.—W. M.

Explosives, Impts. in the Manufacture of. F. Volpert, Dortmund, Germany. Eng. Pat. 14,196, June 10, 1897.

The inventor claims increased safety for explosives used in coal mines by the addition to them of "sulphur, pyrosulphur, polysulphur, and polythionic acid salts as well as analogous acids of silicium, phosphor, selenium, tellurium, and antimony, including meta-acids, also the salts of barium, wolfram, molybdenum and tin acids." "The simultaneous use of the cyanogen compounds is also to be recommended."—W. M.

Nitrocellulose, Impts. in the Manufacture of. A. Liedbeck, Stockholm, Sweden. Eng. Pat. 14,690, June 17, 1897.

The inventor obviates the disadvantage of want of uniformity in the product resulting from the ordinary method of nitrating poor qualities of cellulose, by allowing the beating and nitration to proceed simultaneously. "The acid mixture is introduced into the beater, which is then set in motion, the cellulose being introduced by degrees through a feeding aperture in the hood until the whole quantity weighed off has been fed in."

When the mass has acquired the fineness desired, it is freed from excess of acid centrifugally, and then washed.
—W. M.

Matches. Impts. in. J. T. Maguire, Waterloo, Liverpool. From Diamond Match Co, Chicago. Eng. Pat. 14,989, June 21, 1897.

The object of this invention is to render the match shank uninflammable by means of sulphate of ammonia or other suitable salt.—W. M.

Explosive Compounds, Impts. in the Manufacture of. Hudson Maxim, Surrey. Eng. Pat. 15,499, June 29, 1897.

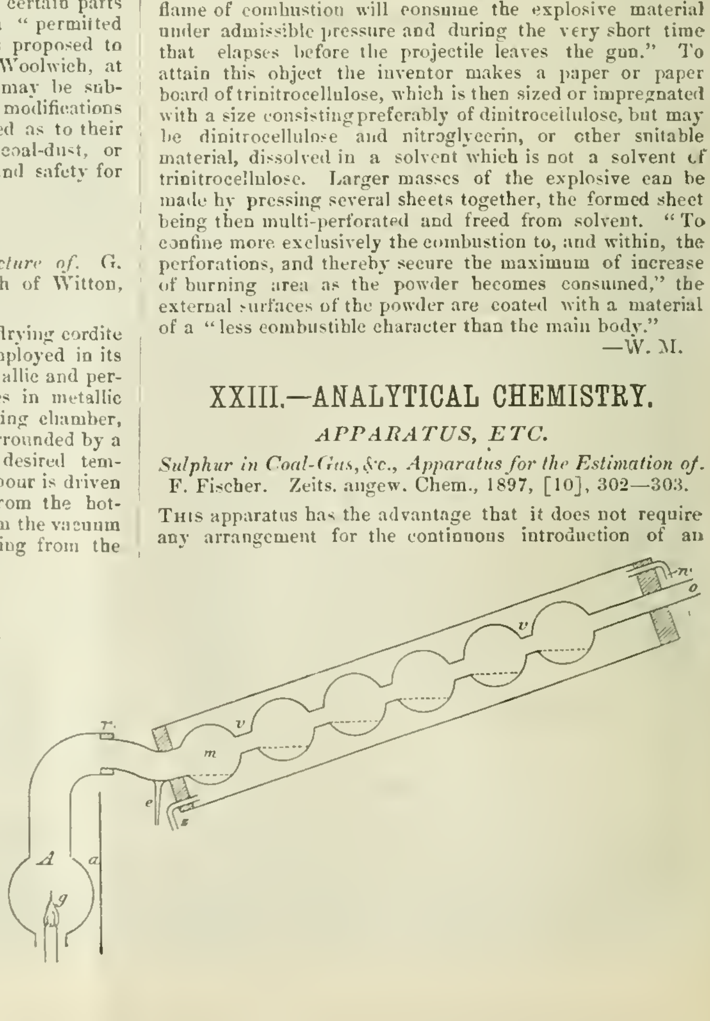
The object of this invention is to overcome the great difficulty experienced with pyroxylin explosives, for use in "guns of large calibre on account of the density of a colloid of guncotton and the small extent to which the flame of combustion will consume the explosive material under admissible pressure and during the very short time that elapses before the projectile leaves the gun." To attain this object the inventor makes a paper or paper board of trinitrocellulose, which is then sized or impregnated with a size consisting preferably of dinitrocellulose, but may be dinitrocellulose and nitroglycerin, or other suitable material, dissolved in a solvent which is not a solvent of trinitrocellulose. Larger masses of the explosive can be made by pressing several sheets together, the formed sheet being then multi-perforated and freed from solvent. "To confine more exclusively the combustion to, and within, the perforations, and thereby secure the maximum of increase of burning area as the powder becomes consumed," the external surfaces of the powder are coated with a material of a "less combustible character than the main body."
—W. M.

XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

Sulphur in Coal-Gas, &c., Apparatus for the Estimation of. F. Fischer. Zeits. angew. Chem., 1897, [10], 302—303.

This apparatus has the advantage that it does not require any arrangement for the continuous introduction of an



absorbing agent for the sulphur compounds evolved, and it is also suitable for the determination of sulphur in burning oils, &c. The gas is burnt within A at the rate of 25 or 30 litres (1 cub. ft.) per hour, and the current of air is regulated so that the gases issuing at o still contain 4 or 6 per cent. of oxygen. During the combustion of 50 litres, some 50 c.c. of weak sulphurous and sulphuric acid condense in the bulbs r and gradually run out through c into a small beaker placed underneath, protected from the heat of the flame *g* by the asbestos sheet *a*. The condensed liquid is either titrated direct, or after oxidation with hydrogen peroxide by means of decinormal alkali; or for greater security, 20 c.c. of the standard soda are introduced into the bulbs through *o* before beginning the test. The sulphuric acid may also be estimated gravimetrically. The joint at *n* consists of an asbestos ring, or the whole apparatus may be constructed in one piece.—F. H. L.

Mercury Pump, A New; Without Taps and Movable Joints. H. Henriet. Comptes Rend. 1897, **125**, 22—23.

In this pump, the lower extremity of the working bulb is connected by a descending tube, partly of glass, partly of rubber, to a mercury reservoir, which latter can be raised or lowered by a chain and pulley in the usual way. The tube just below the working bulb, is branched. The branch ascends vertically for some distance, is bulbed at the top, then again descends, and is finally bent slightly upwards, terminating in a horizontal portion, to which the vessel to be exhausted is attached. At the lower bend, connection is made with a doubly branched tube, both limbs of which descend and terminate in a trough of mercury. One of these limbs is graduated and serves as a pressure gauge, as also for the introduction of gas into the pump and attached vessels. By means of the second, which terminates in a length of rubber tube, connection may be made with an auxiliary water-pump. Returning to the working bulb —its upper portion is drawn out to a capillary tube, which is bent downwards and terminating in a mercury trough forms the outlet of the pump. In operation, the mercury flowing out of the working bulb, presently uncovers the branch tube leading to the exhaust; subsequently, when the mercury is caused to re-ascend, the gas that has passed into the bulb is expelled through the upper capillary tube. —H. T. P.

Hand-Jigger, An Experimental. P. W. Duffield. Eng. and Mining J. 1897, **64**, 68, from the Inst. of Mining and Met.

In the concentration of ores, the estimates made by means of hand tests on the finely ground material are sometimes unsatisfactory, and the author suggests the use of a hand-jigger, in which quite small quantities of ore can be treated and their behaviour during the process actually observed. The concentrates cannot by means of such a machine as that described, be automatically discharged in the form of a finished product, but any ore which is coarser than the screen used, can be subjected to the pulsating action communicated to the water by a plunger, and the result observed from time to time by employing a glass tube in the ore compartment. A screen as fine as 80 mesh can be used in this jigger with advantage and it is provided with an adjustable stroke, the speed being subject to the will of the operator. Although clean material is not actually delivered apart from the gangue, the separation is so indicated that some estimate can be formed of the duty which might be expected from actual working on a large scale.—A. S.

Phosphates and Platinum. W. G. Stratton. Brit. Pharm. Conference, Aug. 1897. Pharm. J. 1897, [1416], 157.

The author calls attention to the fact that when phosphates are heated in a platinum vessel in the presence of carbon, a marked erosion and eventually an absolute fusion of the metal may result.—A. S.

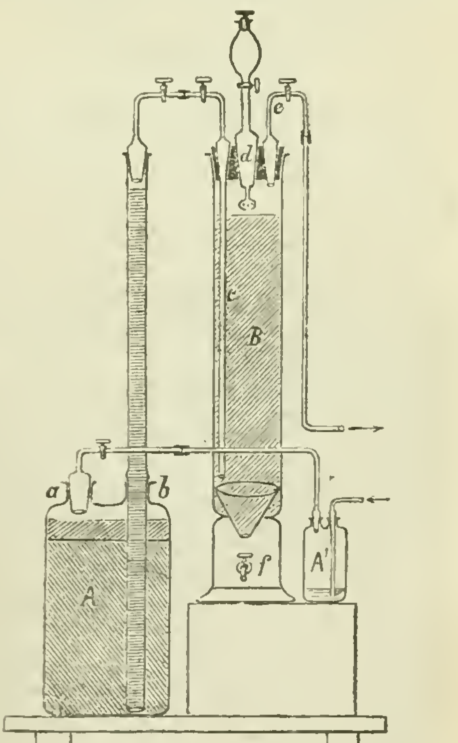
Ampère-Meter, Thermal. C. Camichel. Comptes Rend. 1897, **125**, 20—21.

The apparatus comprises a delicate thermometer, the bulb of which is surrounded by a slightly wider tube of glass, the annular space between the two being filled with mercury, and the whole suitably screened from air currents. The current to be measured is passed through the mercury jacket for 30 seconds and the elevation of temperature noted. The apparatus is calibrated by comparison with a standard ampère-meter. Its scale is open and regular; and according to the author, the increase of temperature corresponding to a given current remains constant when the *initial* temperature ranges from 15°—28° C. The instrument is not liable to get out of order, and is especially suitable for checking the indications of the ampère-meters in distributing stations. In a voltmeter, constructed on the same principle, the dilatation of a column of mercury of high resistance is measured.—H. T. P.

Apparatus for Purifying Gases, New. A. J. J. Vandevelde. Chem. Zeit. **21** [44], 445.

This apparatus has been devised to obviate the inconveniences attending the ordinary forms, due to the high resistance offered to the gases and the necessity for the frequent renewal of the sulphuric acid, &c. employed in drying.

A 2-litre Woulff's bottle A is fitted with a tube *b*, containing soda lime, and is then packed with solid potash covered with a layer of unslaked lime. The gas, after passing through a small preliminary dryer A′ (a Woulff's bottle containing sulphuric acid), enters A through the hollow stopper *a* and diffuses through the contents, being



thereby freed from carbon dioxide. Issuing through *b*, and traversing the tube c, the gas enters the drying vessel B, filled with clean, dry, broken glass and concentrated sulphuric acid (the latter introduced through the hollow stopper *d*, with rose jet attached), where it is dried, and leaves the apparatus *viâ* the tube *e*.

The advantages of this arrangement are that the bulk of the moisture is removed in A′, and thus the contents of A will remain in working order longer, especially since the mouth of *b* is not obstructed by incrustations. In addition to this, A is conveniently arranged for re-filling, as is also B, the spent acid from which can be drawn off through a tap *f* at the base, and replaced by a fresh charge through the funnel tube *d*. This should be done each time the apparatus is used.—C. S.

Caustic Potash and Soda, Commercial, Purified by Alcohol. [*Analysis.*] E. Wurmann. Zeits. Anal. Chem. **36**, 379.

See under VII., *page* 737.

INORGANIC CHEMISTRY.—QUALITATIVE.

Disulphuric Acid, Colour Reaction for. E. Barral. J. Pharm. Chim. 1897, **6**, (3), 104.

Powdered para-dichloride of hexachlorobenzene, $C_6Cl_6Cl_2$ (1 : 4), gives a magnificent reddish-violet coloration when dissolved in sulphuric acid containing disulphuric acid. On the addition of water or on exposure to the air, this colour slowly disappears as the disulphuric acid is converted into ordinary sulphuric acid. The colour thus produced is not due to impurities in the acid, since it is obtained by exposing a mixture of C_6Cl_8 and pure synthetic SO_3 to the action of moist air.

The reaction may be employed for the volumetric determination of disulphuric acid. To a known quantity of Nordhausen acid, a little of the powdered C_6Cl_8 is added, and the mixture titrated with ordinary sulphuric acid containing one-tenth its weight of water, until the coloration disappears. In discoloured Nordhausen acid the end of the reaction is somewhat difficult to perceive, otherwise the process works extremely well.—J. O. B.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Water Analysis, Applicability of Ammonium Carbonate in. A. Rössing. Zeits. Anal. Chem. **36**, 359—369.

It is asserted to be better, after evaporating a water and igniting the residue, to re-convert any calcium oxide formed from carbonate, by using water containing carbonic acid instead of by means of ammonium carbonate, as alkali chlorides are partly changed by the latter into carbonates.

The author evaporated 25 c.c. of solutions of sodium chloride and potassium chloride, containing 5 grms. per litre, to dryness, moistened with ammonium carbonate, dried, ignited and weighed, and found no loss in the residue even on repeating the treatment. A solution of sodium sulphate also suffered no loss.

A solution of calcium sulphate prepared from gypsum and containing per 100 c.c., sulphate corresponding to 0·1304 grm. of SO_3, after repeated evaporation with ammonium carbonate, drying and igniting gently, then contained sulphate equal to 0·01235 SO_3, the residue weighing 0·167 grm. instead of 0·220, indicating a considerable loss of sulphuric acid by volatilisation. Magnesium sulphate also showed considerable loss, and the residue dissolved in hydrochloric acid with effervescence.

A mineral water was prepared containing calcium bicarbonate, calcium and magnesium sulphates and sodium chloride. The residue obtained on evaporation was very gently ignited and then moistened, in one case with ammonium carbonate and in the other with water through which a stream of carbonic acid had been passed, dried on the water-bath and again gently ignited. The residue treated with ammonium carbonate had diminished in weight, while that treated with water containing carbonic acid remained constant.

It was also found that using water containing carbonic acid when magnesium carbonate was present, or using sodium carbonate in the presence of magnesium chloride, avoiding ammonium carbonate and keeping the temperature low, enabled the operator to obtain fairly constant results. The magnesium carbonate obtained, contained 25 per cent. of CO_2; it was therefore a basic carbonate, and the result is low from loss of carbonic acid. The addition of a definite quantity of potassium sulphate in the presence of magnesium chloride, as proposed by Tillmann, appears a much better method, the double salt $MgSO_4,K_2SO_4$ being formed and potassium chloride.

The addition of dilute sulphuric acid, recommended by Fresenius, to determine the mineral salts in the form of sulphates cannot give exact results in the possible presence of calcium and magnesium salts, since to convert the alkali bisulphates formed, ammonium carbonate must be used. Although the ammonium carbonate is added in the solid form and not in solution, it is found to cause loss of sulphuric acid, but the error in this case is small, and may be neglected in water analysis. The use of solid ammonium carbonate instead of water containing carbonic acid is, however, not advisable, as calcium oxide formed by ignition in the deeper layers of the residue may fail to be reconverted into carbonate.—L. J. de W.

[*Silicates, Analysis of.*] *Separation of Soluble Active Silica from Quartz.* G. Lunge and C. Millberg. Zeits. angew. Chem. 1897, 393 and 425.

In view of Michaëlis's statements to the effect that a solution containing 10 per cent. of Na_2O does not act upon quartz, even during several hours' boiling (this Journal 1895, 1065), and that soluble silica cannot be completely dissolved in sodium carbonate, the authors have submitted a large number of different varieties of silica to a lengthy investigation, the results of which are indicated below. Caustic alkalis cannot safely be employed to separate quartz from soluble silica as they dissolve too much of the former. Hot alkali carbonates can only be used with perfect accuracy after the powder under treatment has been freed from the most finely-divided portions by levigation, for if any appreciable amount of impalpable dust be present, some quartz is rapidly dissolved, even by 5 per cent. sodium carbonate on heating to the temperature of the water-bath; but assuming the material to have been prepared by the ordinary process of grinding, followed by shaking through the finest canvas, the error will probably not exceed 0·1 per cent. or 0·2 per cent.—the soluble silica being returned too high. All danger of the particles of the quartz itself penetrating the paper towards the end of the filtration may be avoided by the use of "hardened" papers, coupled with the saturation of the liquid and wash water, with common salt.

The diminution in the power of quartz to resist attack by caustic and even carbonated alkalis when it has been reduced to a truly impalpable powder, is far greater than has previously been recognised; and it is a matter of considerable interest, especially in the case of cement, to accentuate the importance of thorough grinding in order that the chemical properties of the crystalline silica may be fully utilised. Trass and puzzolana do not contain any free amorphous silica; but their active constituents consist of zeolitic silicates, the most notable one being a compound of sodium and aluminium resembling analcite, which is entirely soluble in 30 per cent. caustic potash on the water-bath.

It accordingly follows from the foregoing observations, that in the analysis of silicates, if the sample be reduced to the finest possible powder, treated with hydrochloric acid, and dried at 110° C., the silica derived from the silicates decomposed by the acid may be readily separated from the quartz and the unattacked silicates, by digestion for 15 minutes with 5 per cent. sodium carbonate, the error not exceeding + 0·2 per cent.—F. H. L.

Nitric Acid, Quantitative Determination of, by Electrolysis. K. Ulsch. Zeits. für Elektrochem. 1897, **3**, 546—547.

If a dilute solution of nitric acid or a solution of a nitrate in water be electrolysed with platinum electrodes in presence of sulphuric acid, and of a sufficiency of copper sulphate, the nitric acid is reduced completely to ammonia, and copper is deposited at the cathode. This reduction has been made the basis of a quantitative method by G. Vortmann. Some years ago, the author published details of a similar method, in which an iron-copper couple was used in a sulphuric acid solution to reduce the nitric acid, and the volume of gas liberated gave the data for the quantitative determination.

The author has recently experimented with a modification of Vortmann's method, in which a platinum plate covered with spongy copper is used as cathode, and no copper sulphate is used in the cell. Such cathodes, however, quickly lose their reducing power. Further experiments showed that the physical characteristics of the surface of the copper

cathode determined its reducing action, and led to the adoption of the following method:—A cylindrical spiral of copper wire (1·4 mm. diameter) is made by winding 2 metres in length of the wire, upon a glass tube of 15 mm. diameter. One end of the wire is bent into the line of the cylinder axis, and is left projecting about 15 cm., to serve as the conducting end outside the cell. The spiral is gently stretched until it measures 70 mm. in length. A straight platinum wire, 1 mm. thick and 20 cm. in length, is used as anode. A test tube 20 mm. wide by 17 cm. long is used as the electrolytic cell, and is closed by a rubber stopper, through which both anode and cathode ends pass.

The anode hangs in the centre of the spiral, and both must nearly touch the bottom of the tube. Before each determination, the copper spiral is heated in a Bunsen flame, to faint redness, and is immediately quenched in cold water. The solution used for the author's experiments contained a known amount of potassium nitrate; 20 c.c. were used for each test. This volume contained in each case 10 c.c. of normal sulphuric acid. Two accumulators coupled in series, gave the E.M.F. (4 volts), demanded.

The current density was reduced to 1·47 by means of a resistance; the surface of cathode immersed, was calculated from the known dimensions of the wire and spiral. The reducing action of a copper spiral cathode, prepared by heating as described, is so perfect, that for long no hydrogen bubbles are visible; and in a special experiment it was proved that 90 per cent. of the nitric acid had been reduced before bubbles appeared. The heat of the reduction causes the solution to gain in temperature. This is favourable to the reaction. The electrolysis must be continued for 10 minutes after the moment when hydrogen bubbles first become visible at the cathode. The ammonia produced is determined by titrating the solution for the excess acidity, and by calculating the ammonia equivalent to the loss of acid found.

The author gives a table, showing that in ten experiments conducted under proper conditions as regards excess of sulphuric acid, the average variation from the correct result was only ·029 per cent., and the average time required for the electrolysis was only 36 minutes. The method therefore excels all others in speed and accuracy. Unfortunately the presence of other salts, *e.g.*, chlorides, though as mere impurities, in the solution renders the results untrustworthy; and the use of this method is therefore confined to those cases where pure solutions are under examination.

—J. B. C. K.

Iron Ores. Notes on the Determination of Insoluble Phosphorus in. C. T. Mixer and H. W. Du Bois. Franklin Inst., 1897, **144**, 137—138.

The usual method of determining the phosphorus in the insoluble residue from an iron ore, by fusion with sodium carbonate is impracticable where many analyses are to be made. Hydrofluoric acid treatment led to no saving in time, and the heating to redness with sodium carbonate, although successful with low silica ores, was useless with those containing much silica, on account of the fusion of the mass. Ignition at a red heat with calcined magnesia extracted all the phosphorus, but simple calcination without any basic addition was equally successful. In the end, the simplest method found to be quite accurate, was as follows:—1·5 grm. of ore is dissolved in 25 c.c. of HCl (sp. gr. = 1·1), and the solution is evaporated to a syrup. After dilution, it is filtered into an Erlenmeyer flask, and the filter with the residue is ignited in a platinum crucible. The residue is then broken up with a platinum rod, and ignited for several minutes at a red heat, removed, cooled, and boiled in a beaker for 5 minutes with water containing a few drops of hydrochloric or nitric acid. After filtering, the solution is neutralised with ammonia and precipitated as phospho-molybdate, the precipitate being titrated by Handy's modification of the sodium hydroxide method. The solution from the insoluble residue, may be mixed with that containing the soluble phosphorus prior to precipitation, but it is better to treat the two separately.—W. G. M.

Metals, Electrolytic Methods of Analysis of. L. Wolman. Zeits. für Elektrochem. 1897, **3**, 537—545.

The results may be summarised as follows:—

Pure Solutions.—The determination of copper in acid or ammonium oxalate solution, of lead as peroxide in nitric acid solution, and of silver in potassium cyanide or nitric acid solution, can be carried out with both speed and accuracy, and in these respects the electrolytic methods are quite equal to the purely chemical methods.

As regards iron and manganese, the electrolytic methods are far behind the chemical methods, especially in respect to the time demanded.

Mixtures.—The separation of two or more metals is possible in those cases where the one metal is not deposited in acid solutions. This method gives most satisfactory results in the case of solutions containing zinc and copper, nickel and copper, and cadmium with zinc.

The separation of zinc and nickel by Vortmann's method, which consists in using an alkaline sodium-potassium tartrate solution offers great advantages as regards speed, but the method leads at present to inaccurate results.

The separation of lead as lead peroxide at the anode in solutions made strongly acid with nitric acid, from zinc, silver or copper is complete. The deposition of the other metals on the cathode, in such strongly acid solutions is however too slow to be satisfactory, and in the case of silver the deposit is spongy. It is suggested by the author that this difficulty may be met by dissolving the cathode deposit, after all the lead is separated at the anode, removing the excess of acid by evaporation, and by then repeating the electrolysis with this less strongly acid solution, for separation of the zinc, silver, or copper.

In such cases, however, it may probably be quicker to separate the metals by chemical methods, and to use the electrolytic deposition only for their final separation from pure solutions.—J. B. C. K.

Bronze and Brass, Analysis by Electro-chemical Methods. A. Hollard. Comptes Rend. 1897, **124**, [25], 1451—1453.

Bronze.—Determination of Copper.—Attack 50 grms. with 25 c.c. of nitric acid of specific gravity 1·32 (36° B.) and 15 c.c. of strong sulphuric acid, as described in a former paper (this Journal, 1897, 66). Dilute to 350 c.c., heat nearly to boiling, keep at this temperature till any deposit of tin clots and leaves a perfectly clear solution, then immerse the cone and spiral, and electrolyse as directed (*loc. cit.*).

Determination of Tin.—Evaporate the liquid, after removing copper, till only a few drops of sulphuric acid remain; take up with hydrochloric acid and water, and precipitate the tin with hydrogen sulphide; wash the precipitate with sodium chloride solution, dissolve in yellow ammonium sulphide, and evaporate to dryness on the water-bath; treat the residue with 9 grms. of potassium chlorate dissolved in water, and excess of hydrochloric acid; evaporate to dryness and dissolve residue in 30 c.c. of hydrochloric acid diluted with water; filter; add and dissolve 30 grms. of pure ammonium oxalate, heat to about 90°, and electrolyse with a current of about 0·7 ampère. The whole of the tin will be down in about 12 hours as a firmly adhering deposit. (These deposits all adhere better if the surface of the cone be roughened by depositing zinc on it, dissolving off with nitric acid, and dipping the cone into melted potassium hydrogen sulphate several times in succession.)

Determination of Zinc.—Boil the filtrate from the precipitated tin sulphide to expel hydrogen sulphide; evaporate on the sand-bath till only a few drops of sulphuric acid remain: dissolve the residue in water; neutralise with ammonia; add 15 c.c. of a 10 per cent. solution of ammonium citrate, 13·8 grms. of ammonium acetate (or its equivalent in acetic acid and ammonia), and 3 c.c. of glacial acetic acid. About 12 hours' electrolysis, with a current of 0·6 ampère, will deposit the zinc completely. Iron in the bronze may deposit partly with the zinc, and must then be determined by permanganate in the solution of the zinc from the electrode.

Lead is determined in bronze exactly as in commercial copper (this Journal, 1897, 164). The stannic oxide formed

during the attack with nitric acid, need not be removed; it is simply allowed to settle.

Brass.—Copper, zinc, and all impurities are determined exactly as above, or as in the two papers above referred to. —J. T. D.

Arsenic, Separation from Antimony. O. Piloty and A. Stock. Ber. **30**, 1649—1655.

Arsenic, dissolved in strong boiling hydrochloric acid into which is led hydrogen sulphide, is completely volatile. The weighed mixture of arsenic and antimony salts is placed in a round-bottomed flask of 300 c.c. capacity and 100 c.c. of strong hydrochloric acid added. Through the cork pass two tubes. One dips under the acid, and is connected above, by a T-piece, with generators of hydrochloric acid gas and of sulphuretted hydrogen, the tube communicating with the latter being furnished with a pinch-cock. The other is a delivery tube, and dips into 250 c.c. of water acidulated with hydrochloric acid, contained in a 600-c.c. Erlenmeyer flask which stands in ice. The solution is now heated to boiling, a rapid stream of hydrochloric acid led in, and two bubbles per second of hydrogen sulphide passed. The liquid turns milky from sulphur, but clears again. In half an hour or more, nearly all the liquid has distilled over; the hydrogen sulphide is now shut off, and, after a minute or so, the flame removed and the T-piece disconnected. After cooling, the arsenic sulphide is collected on a filter on a porcelain filter-plate, then (with the portion adhering to the flask and the delivery tube) dissolved in as small a quantity as possible of dilute caustic potash solution, in a 500-c.c. round-bottomed flask. Bromine in excess is now added, the whole acidified with dilute hydrochloric acid, and boiled till all free bromine is driven off. The liquid is transferred to a 500 c.c. Erlenmeyer flask, and kept at 70° for three hours whilst hydrogen sulphide is passed through; the stream is kept up while cooling, and the liquid allowed to stand for 12 hours. The arsenic pentasulphide is collected in a Gooch crucible, washed successively with water, absolute alcohol, pure carbon bisulphide, alcohol, and anhydrous ether, dried at 105°, and weighed.

The antimony solution in the distillation flask is filtered, diluted, precipitated by hydrogen sulphide, and the sulphide treated in the same way as the arsenic pentasulphide. This simplicity in the determination of the antimony is the great advantage which this method of separation presents over the distillation with a ferrous salt.

The authors are busied with a process for the separation of tin from antimony and arsenic.—J. T. D.

Hypophosphites. C. T. Tyrer. Brit. Pharm. Conference, Aug. 1897. Pharm. J. 1897, [1416], 150—152.

The author considers that the characters and tests of the U.S.P. are superior to those of the B.P., which are unsatisfactory and indefinite. The following volumetric method for the quantitative determination of hypophosphites is stated to be more accurate and generally applicable than those hitherto published:—One grm. of the sample is dissolved in water, and 5 per cent. barium chloride solution added in slight excess. The precipitated sulphite, sulphate, phosphite, and phosphate are filtered off, and may be weighed or preserved for subsequent testing. Copper sulphate solution, 10 per cent., is added to the filtrate in excess, with 5 c.c. of concentrated sulphuric acid, and the whole is then boiled for 10 minutes. The precipitate of metallic copper, together with a proportion of protoxide present, and some barium sulphate, is washed by decantation, dissolved in 30 per cent. nitric acid, sodium carbonate added until a slight precipitate is formed and then acetic acid in excess. Excess of potassium iodide is added, and the liquid titrated with thiosulphate solution, using starch solution to indicate the end reaction. If sulphuric acid be added in large excess, only one molecule of copper is precipitated, as compared with two molecules in presence of a small amount of acid. When applying this method to the iron salt, the latter is dissolved in 20 per cent potassium citrate solution and well diluted before reduction.

The odour of sulphuretted hydrogen, which is sometimes developed by hypophosphorous acid and the syrups of the hypophosphites, is attributed to the use of charcoal as a filtering medium. The trace of free sulphuric acid usually contained in hypophosphorous acid is reduced to sulphuretted hydrogen by boiling with charcoal, and even in the cold, on long standing. Traces of phosphate, phosphite, and sulphate are always present in hypophosphites and the sodium compound usually contains more phosphite than the other salts. A trace of carbonate should always be present in the potassium and sodium compounds: first, to ensure the absence of calcium, and secondly, to prevent the generation of a distinctive odour on keeping; this odour is stated to be probably due to spontaneous decomposition, resulting in the development of gaseous P_2H_4. Barium hypophosphite should always be tested for nitrates, this impurity occurring from the use of imperfectly prepared barium hydrate. Mixtures of barium nitrate and hypophosphite are stated to be particularly explosive. Arsenic should be tested for in all hypophosphites, being a possible impurity in badly refined phosphorus.—A. S.

ORGANIC CHEMISTRY.—QUALITATIVE.

Quinones, General Reaction for Aromatic. S. Blumenfeld and P. Friedlaender. Ber. **30**, 1464—1465.

Under ordinary conditions aromatic quinones react with phenols, forming phenoquinones or quinhydrones. These are direct addition products, are highly coloured, and are easily decomposed into their components. Thus benzoquinone and hydroquinone yield quinhydrone, whilst quinone and phenol yield phenoquinone. In presence of sulphuric acid, however, equimolecular proportions of a quinone and a phenol yield colourless condensation products, but, according to analysis, without elimination of water. For instance, one molecular proportion of α-naphthoquinone is dissolved in a small quantity of acetic acid, somewhat more than one molecular proportion of pyrogallol is added, and then a few cubic centimetres of dilute sulphuric acid. A precipitate is formed which dissolves on heating, and the solution becomes colourless. On diluting with water, the condensation product separates in a crystalline form. It dissolves easily in alcohol, ether, glacial acetic acid, and acetic ether, but is insoluble in water, petroleum ether, and chloroform. It has the composition $C_{16}H_{12}O_5$, and melts at 240°—246° C. Similar products are obtained from benzoquinone and β-naphthoquinone on the one hand, and resorcinol and α-naphthol on the other.—T. A. L.

Lard, Detection of Beef Fat in. M. Ballo. Zeits. Nahrungsm. Hyg. Waar. **11**, 193—197; through Chem. Cent. **68**, 392.

Pure lard, when melted and allowed to cool, solidifies with a perfectly even surface, whilst in the case of beef fat the surface is broken up or cracked, and air is enclosed in the fat. The author, in conjunction with S. Glöckner, proposes to measure the amount of enclosed air, and in this way to determine the quantity of beef fat present in mixtures of this fat and lard. The melted fat is placed in a glass vessel and allowed to thoroughly solidify. The whole apparatus is then filled with water. 100 c.c. of chloroform are now run into the vessel from a funnel fitted on to a burette, which in turn is ground into the neck of the vessel. The displaced water is allowed to run out through a side tube in the neck of the glass vessel. The tap connecting the funnel to the burette is then closed. As the fat is dissolved in the chloroform, the liberated air rises into the burette, and may be measured. Pure lard solidifies free from air, but when adulterated with more than 9 per cent. of beef fat, air is always found in the fat; even 3 per cent. may be detected. Pure beef fat gives off 6·5 to 8·8 c.c. of air; pure lard, none; lard containing $33\frac{1}{3}$ per cent. of beef fat, 3·2 to 5·3 c.c. of air; and lard with 9 per cent. of beef fat, 0 to 3·1 c.c.; calculated on 100 grms. of fat.—W. P. S.

Oil of Turpentine (French), Detection of Rosin Oil in. A. Aignan. Comptes Rend. **124**, 1367.

The author has previously shown that since French oil of turpentine, the product of *Pinus maritima*, is levo-rotatory, and rosin oil is dextro-rotatory, a simple determination of the optical activity is sufficient to detect any considerable

admixture of the one with the other (Comptes Rend. **109**). It is now found that by distilling off the greater part of the oil and determining the optical rotation of the residue, a very small admixture of rosin oil can be detected by the lowering of the optical rotation of this residue. Thus, by distilling 250 c.c. of oil of turpentine, containing 3 per cent. of added rosin oil, until the residue measured 70 c.c., the rotation of this for 200 mm. was only -36°·21, whilst that of the same amount of residue from a like quantity of pure unmixed oil was—51°·5. It was found that by conducting the distillation under reduced pressure, a dextro-rotatory residue was obtained when only so small a quantity as 0·5 per cent. of rosin oil was added. J. O. B.

Tannin and Gallic Acid, Action of, on Quinoline Bases. O. de Coninck. Comptes Rend. 1897, **125**, 37—38.

The author has investigated the reactions furnished by lepidine, quinoline, and quinaldine with tannin and gallic acid under varying conditions, and finds that they closely resemble those yielded by the pyridine and hydrogenised pyridine bases, as also many volatile alkaloids, under the same conditions. (This Journal, 1897, 470.)—H. T. P.

Chloral Hydrate: Reaction with Ammonium Sulphide. I. Lesinsky and C. Gundlich. Amer. Chem. J. **19**, 603—606.

To 10 c.c. of a solution of chloral hydrate (8 grms. to 100 c.c. of water), 5 c.c. of yellow ammonium sulphide are added, and the whole quickly mixed. A precipitate is formed, quite suddenly, but after the lapse of an interval of time, more or less considerable according to the temperature (at 1° C., 44 seconds; at 65° C., 3 seconds). The precipitate is yellow or pinkish, becoming rapidly darker when heated on the water-bath; and the filtered solution, extracted with ether, yields an oil with an odour of walnuts.

The authors suggest that this reaction may be of value in the commercial examination of chloral hydrate. They are investigating it further, both as to the nature of the products and as to the cause of the retarded precipitation. —J. T. D.

ORGANIC CHEMISTRY.—QUANTITATIVE.

Ultimate Organic Analysis, Simplification of. M. Dennstedt. Ber. **30**, 1590—1597.

A combustion tube 80 cm. long is charged at its middle with platinum black (prepared by the author from pyridine chloroplatinate) 6—8 cm. long, kept in place by discs of fine platinum gauze at either end. It is supported on a strip of asbestos cardboard in a sheet-iron tray, itself supported on five or six movable uprights of sheet iron, which serve at once as supports and as shields against radiation from one part of the tube to another. A cover of bent sheet iron lined with asbestos serves to keep in the heat when necessary. A couple of Teclu burners heat the platinum black to low redness at the beginning and throughout the combustion, and all other heating is done by ordinary Bunsen burners, for the most part turned low. The usual arrangements are made for burning the substance in a stream of oxygen, and for collecting the water and carbon dioxide formed. Care must be taken to have always an excess of oxygen, and to conduct the combustion cautiously until the substance is completely carbonised, after which no particular care is needed.

In front of the platinum black come two silver boats filled with molecular silver, heated to 200—300°, and then two boats with lead peroxide, heated to 150°, each of these having been weighed. Any halogen in the substance is caught by the silver boats. Any sulphur is partly burnt to sulphuric acid, which also is completely caught by the silver boat, and partly to sulphur dioxide, which is absorbed by the lead peroxide; whilst, if the substance contain nitrogen, any of this which may be converted into NO_2, is also absorbed by the lead peroxide. The procedure when all these may be present, is as follows (the simplifications, where some are known to be absent, will be obvious):—The increase of weight of the silver boat is due to halogen $+ SO_4$: the boat and contents are digested in a stoppered tube with dilute potassium cyanide solution, the filtered solution acidified with hydrochloric acid, and boiled till all HCN is expelled; in the filtrate from the silver chloride, the SO_4 is determined by barium chloride: the amount of this subtracted from the increase of the silver boat, gives the halogen. The increase of the lead peroxide is $SO_2 + NO_2$: the contents of the boat are extracted with 33 per cent. alcohol and made up to 100 cc., and the lead nitrate in an aliquot part is determined by evaporation; the corresponding NO_2 in the whole, subtracted from the increase in the peroxide boat, gives the SO_2, from which, along with the SO_4 in the silver boat, the sulphur is calculated. Proofs of the accuracy of the work are given:—In a test analysis, *p*-bromophenylurea, $CS(NH.C_6H_4Br)_2$, gave—

	H	C	S	Br
	2·59	40·31	8·29	41·45
calc.	2·80	40·44	8·31	41·67

Not only are chlorine and sulphur thus determinable in one operation with carbon and hydrogen, but no special combustion furnace is needed and no special room; little time is taken up, and the combustion tubes last out many operations.—J. T. D.

Sugars, Iodometric Estimation of. G. Romijn. Zeits. Anal. Chem. **36**, 349—359.

Having shown (this Journal, 1897, 366) that oxidation by iodine in alkaline solution affords a convenient and exact method for the determination of formaldehyde, and that other aldehydes are also attacked, the author has attempted to extend the method to estimation of the sugars.

The oxidation of the reducing sugars in alkaline solution by various reagents, even by atmospheric oxygen, proceeds readily, although it is doubtful whether the reaction is a simple one. If, however, this were so, and if glucose were oxidised to gluconic acid according to the equation—

$$CH_2OH.(CHOH)_4.CHO + 2I + 3NaOH =$$
$$CH_2OH.(CHOH)_4.CO.ONa + 2NaI + 2H_2O,$$

each c.c. of $\frac{1}{10}$ N iodine solution acted on would thus represent 9 mgrms. of glucose. It is found that in the presence of free alkali the decomposition proceeds further, so that it is better to supply the necessary alkali as sodium and ammonium carbonate, potassium bicarbonate, trisodium phosphate, and borax, all of which were tried. Although in presence of borax the action is slow, the reagent has the advantage of sharply differentiating glucose and levulose, or, generally, aldoses and ketoses. On mixing borax with solution of iodine in potassium iodide, the brown colour becomes only slightly lighter; there is consequently only a small quantity of hypoiodite and iodate formed. The solution, however, acts on thiosulphate, as Topf found (*ibid.* **26**, 137), with alkali carbonates and bicarbonates; that is, less thiosulphate is necessary for the reduction than for the acidified solution, and the difference is only partly made up on acidifying by the iodine set free from the iodate formed. A part of the thiosulphate is therefore oxidised to sulphate. From solutions titrated alkaline, the iodine set free on acidifying, was very small in fresh solutions, and even on warming to 60° or 70° C., or in old solutions, amounted to only 12—30 per cent. of the quantity obtained on titrating the previously acidified solution. The mixed solution may therefore be kept without fear of a great decrease in titre. It is best prepared by dissolving the borax in a portion of the water by warming, and after the solution is cool, adding the concentrated iodine solution and making up to a definite volume. The solution used by the author contained, in 25 c.c., 1 grm. of borax, and iodine sufficient to require for its decolorisation, after acidifying, 30—33 c.c. of $\frac{1}{10}$ N thiosulphate solution. If 25 c.c. of this solution is mixed with a definite quantity of a solution of glucose, it is at first quickly reduced, and then more and more slowly. On acidifying and titrating with thiosulphate, the remaining iodine is determined and the difference calculated to sugar. The results are found to be influenced by the temperature, by time, and by quantity of sugar. 25° C. was chosen as the most suitable temperature. The test is made in a narrow-necked flask closed with a glass stopper. After the sugar solution and the borax-iodine solution are introduced by a pipette, the stopper is fastened by wire and a drop of water placed round the rim, and the flask is placed in a thermostat. After being heated

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for a definite time, 1·5 c.c. of hydrochloric acid is added, of sp. gr. 1·126, and the iodine titrated. The values obtained by using 35—50 c.c. of solution and acting for 16—22 hours on 154—164 mgrms. of sugar varied from 97·76 to 99·47 per cent. of the sugar taken. The reaction may be regarded as complete in 16—22 hours, for 20—24 hours later only about 2 per cent. more sugar was found. On great dilution the results are higher, due to the hydrolytic dissociation of the borax; for when this is prevented by the addition of boric acid, the action is greatly retarded.

Under similar conditions, levulose is scarcely attacked. Herzfeld (Zeits. f. Rübenzuckerind. 35, 967) states that, although levulose is more sensitive to alkalis than glucose, it exhibits greater resistance to oxidising substances in alkaline solution; but a sharp distinction of this kind was not to be expected. At 25° C., in 17½ to 19 hours, from 2·23 to 3·22 per cent. of the quantity of levulose taken was found; in 40 to 42½ hours, 5·04 and 5·60 per cent. At higher temperatures glucose is more quickly oxidised, but levulose is also more strongly attacked. It is possible that at lower temperatures a still sharper distinction might occur, but the determination would then require too long a time, and it would be inconvenient to maintain a lower temperature than 25° C. in the summer months. The behaviour of nearly all the simple sugars and some compound sugars were examined, towards borax-iodine. It was found that the aldoses—galactose, mannose, arabinose, xylose, and rhamnose—behaved in the main as glucose does. The results appear to show small variations in oxidisability, and it is possible that different isomers are differently attacked; but the difference is insignificant, and requires further investigation with more abundant material. Chitosamine chloride, which Lobry de Bruyn and Alberda van Eekenstein have shown easily passes into a derivative of levulose, is oxidised by borax-iodine as an aldose. Sorbose suffers as little oxidation as levulose. The compound sugars are more strongly attacked than was to be expected from their constitution. For maltose and lactose this is not very surprising; but saccharose, raffinose, and stachyose, which do not reduce alkaline copper solution, are strongly oxidised; consequently it is not possible to conclude as to the presence of aldoses from reduction alone.

Among other organic substances examined, those forming iodoform caused a strong reduction, acetaldehyde being a surprising exception. No separation of iodoform was observed in the case of ethyl alcohol, but much with lactic acid and acetone. The simultaneous formation of oxalic acid from lactic acid affords an excellent reaction for identifying that substance. Glycerin and mannitol were also strongly attacked, owing to their capability of forming compounds with boric acid. The behaviour of formic acid is also worthy of notice. In the short time required for the determination of formaldehyde, no irregularities indicating further oxidation were observed; but, on this account, borax-iodine is unsuitable for the determination of formaldehyde.

Various mixtures of sugars were now tested. It was found that the presence of levulose produces no alteration in the oxidation of glucose. Mixtures of these sugars may therefore be determined as pure glucose. Cane sugar acts otherwise, but the difference is small, as with five times as much saccharose as lactose, the result was only increased by 2 per cent. Milk sugar may thus also be tested in the presence of cane sugar.

Mannitol, however, greatly retards the oxidation of glucose—a phenomenon in accord with the increase in the conductivity of boric acid on the addition of mannitol observed by Magnanini (*ibid.* 30, 467); and glycerin appears also to form a similarly easily oxidisable compound.

Although the above method thus allows of a convenient and exact determination of aldoses, either alone or mixed with ketoses and other bodies, it is not immediately applicable in practice, as the glycerin in wine and uric acid in urine, would cause errors in estimating the sugar.

—L. J. de W.

Volatile Acids in Wine, Estimation of. U. Gayon. Revue de Viticulture; La Bière, 5, [6], 81—82, and [7], 97—100.

In the normal (alcoholic) fermentation of grape-must, not more than 20 to 25 centigrms. of volatile acids, expressed in monohydric sulphuric acid, are produced per litre; but as the yeast becomes weaker, the microbes obtaining access quickly increase the (volatile) acidity, as shown by determinations made by the author in two vats of 1896 wine, wherein this acidity was raised from 0·12 grm. per litre (in the absence of microbes) to 0·36 grm. within 8 days in the one case, where 35 to 40 microbes were detectable in the microscopic field, and to 0·45 grm. in the same time in the second sample, which revealed 50 to 60 microbes. In this instance the usual increase of *total* acidity noticeable in diseased wines was masked by the precipitation of tartar under the influence of alcohol and reduced temperature (29° C. maximum). That the injurious ferments do not always make their appearance at the same stage is demonstrated by the fact that in the first sample, 22·7 grms. of sugar were still present per litre at the time of infection, whereas in the second sample there was only 1 grm. per litre unconsumed, the fermentation being at an end and the wine only left to allow the colour to deepen. The date of the appearance of the microbes varies according to a number of circumstances, including temperature, weather, maturity of the grapes, dimensions of the fermenting vat, &c., infection being noticed in 1896 on the 16th—17th day, from the 9th to 12th day in 1880 and 1888, and on the 4th day in 1891. In 30 samples of 1896 wine examined by the author, only a single one was destitute of microbes, and this was found to contain only 0·2 grm. of volatile acidity per litre, the figures for the others ranging from 0·3 up to as much as 0·6 grm. in Gironde wines, and even to 0·8 grm. in a Narbonne wine.

The Bordeaux wines are all infected with germs capable of producing disease in the environment, and these continue to develop in bottle, despite rackings, finings, and mixings; but in the earlier stages, the volatile acids they excrete do not always spoil the flavour of the wine, a transient improvement being effected in some cases until the limit of 1 grm. per litre is exceeded.

The influence of temperature on the maturing of wine is exemplified by an 1887 Medoc wine, part of which was stored in a cool, and the rest in a warm cellar; the former, after 8 years in store, being quite sound, and only containing 0·92 grm. of volatile acidity; whereas the second had "turned," and gave 2·31 grms. per litre. Very similar results were obtained with a mixture of 1890 and 1891 vintages under the same conditions.

The percentage of volatile (acetic) acid has also been found to increase concurrently with the progress of mannitic fermentation.

For the estimation of these acids, the following modification of the Pasteur method is recommended as being quicker than the original, and capable of performance on a smaller sample.

Ten c.c. of the wine and 20 c.c. of water, are distilled in a tubulated retort, connected with a glass condenser. When 20 c.c. of distillate have passed over, an equal quantity of water is poured into the retort, and a second 20 c.c. of distillate collected, this procedure being repeated until the distillate is no longer appreciably acid. The successive fractions collected, are then titrated with calcium hydroxide solution (titer 4·7), and the acidity calculated from the amount of reagent consumed. The results agree with those obtained by the Duclaux method, which requires a larger quantity of substance, but is more expeditious in cases where the wine is rich in volatile acids; on the other hand, the modified Pasteur method gives an earlier indication of the acidity present, the quantity contained in the first distillate representing approximately one-half of the total.—C. S.

Boric Acid in Milk, Detection of, and Rapid Method of Determination. G. Denigès. J. Pharm. Chim. 6, (2), 49.

If to a milk containing boric acid, some phenolphthaleïn be added, and then decinormal soda till a very faint rose tint is produced, and afterwards a little neutral glycerin (about one-fourth the volume of the milk), it will be noticed that the rose tint at once disappears. In order to restore the original colour, it will then be necessary (especially if to assist the process, as much alcohol be added as was added

of glycerin) to add an amount of the alkali solution serving as a precise measure of that amount which, on the one hand, the borated milk alone would absorb, and that, on the other which an equal volume of an aqueous glycerin solution of boric acid of the same strength would require. It will be visible how on this basis a method may be devised for the rapid estimation of boric acid in milk.

Two flasks, each containing 20 c.c. of the sample of milk, are placed on a white background, and to one of them a few drops of phenolphthalein solution are added, followed by sufficient $\frac{1}{10}$ N.NaHO solution to give a barely perceptible rose tint, readily observed by comparing with the other flask. 10 c.c. of a mixture of equal parts of alcohol, 90 per cent. and of glycerin, is then run in. If the rose tint persist, borax may be considered as absent; if it be present, the red coloration disappears and the mixture is titrated with $\frac{1}{10}$ N.NaHO solution until it is brought back. The number of c.c. employed in this second titration is noted and the amount of boric acid calculated with the following correction for the action of the lactose in the titration. When N = number of c.c. employed, $N - 0{\cdot}15$ = number of grms. per litre of boric acid added. This correcting figure has been established by actual experiment, but it only holds good where the amount of boric acid does not exceed 3 grms. per litre, and when the lactose (as in normal milk) ranges from 40 to 50 grms. per litre. When the amount of boric acid exceeds this figure, the sample must be diluted with an equal volume of water and sufficient milk sugar added to bring the amount up to 40—50 grms. per litre. When borax has been used as a preservative, half a c.c. of $\frac{1}{10}$ N HCl solution must be added before the first neutralisation.

—J. O. B.

Digitalis, Determination of Active Principles of. C. C. Keller. Ber. deutsch. Pharm. Ges. **30**, [7], 125; through Pharm. J. **59**, [1413], July 24, 1897, 62.

The author advocates the following method for the determination of digitoxin in foxglove leaves. This substance is freely soluble in alcohol and chloroform, and is precipitated by water and dilute alcohol, also by tannin, but not by basic lead acetate. It can be washed out with chloroform from acid or alkaline solutions. Digitalin and digitonin are almost insoluble in chloroform. The leaves are extracted by percolation with 70 per cent. alcohol, until the residue from 3 or 4 c.c. redissolved in acid water, gives no precipitate with tannin. The alcohol is distilled off, the residue dissolved in water and precipitated by basic lead acetate, filtered, washed, excess of lead removed with sodium sulphate and the clear filtrate, made faintly alkaline with ammonia, is washed out with several successive portions of chloroform. The solvent is evaporated, the residue dissolved in 3 c.c. of chloroform, 7 c.c. of ether, and 10 c.c. of petroleum spirit are added, when the digitoxin is precipitated in flocks. For weighing, this precipitate may be redissolved in hot alcohol, and the solution evaporated in a tared vessel, or the petroleum spirit may be decanted off, and the digitoxin weighed in the form of powder. A solution of digitoxin in glacial acetic acid containing ferric chloride, when floated upon strong sulphuric acid, gives at the line of contact, a dark zone and gradually the acetic solution becomes dark blue. This reaction is given by $\frac{1}{10}$ mgrm. of digitoxin.

The aqueous solution from which digitoxime has been removed by chloroform contains digitonin, which may be precipitated with tannin. The author was not able to isolate digitalein which he regards as a mixture of digitonin with traces of digitoxin and of digitalin. He concludes that the leaves, like the seeds, contain digitalin, digitoxin, and digitonin, but that the proportions of each are somewhat different, good leaves containing more digitoxin than the seeds. The amount present in the leaves, is, however, very variable, ranging from 0·26 to 0·62 per cent. Pharmaceutical preparations were found to vary still more, and the author advocates their standardisation by the amount of digitoxin contained in them.—J. O. B.

Commercial Egg Albumin, Estimation of. P. Carles. J. Pharm. Chim. 1897, **6**, [3], 102.

Two grms. of the sample are dissolved in a small quantity of water and gradually diluted, with constant stirring, to 200 c.c. If free from coagulated albumin this solution will be clear. 100 c.c. of this are taken and 35 c.c. of a 1 per cent. pure tannin solution added, and a pinch of powdered potassium bitartrate. The mixture is well shaken, then thrown on a plain filter, and the clear filtrate divided into two equal parts in twin tubes. To one a few drops of solution of transparent gelatin is added; to the other a like quantity of tannin solution. If both remain clear, the egg albumin is pure and of good quality. If the gelatin solution cause a precipitate, it shows that the egg albumin is weak and of poor quality, either from a portion having coagulated by overheating in the course of manufacture, or by the fraudulent addition of non-coagulating matter. On the other hand, if the tannin solution give a precipitate, it is due to the presence of gelatin, which has about four times greater precipitating power for tannin than egg albumin; but, since it is not coagulated by heat, it is worthless for a majority of technical purposes, such as clarifying liquids, for which egg albumin is used. A further test is to heat 100 c.c. of the solution gradually on the water-bath. In this way the whole of the egg albumin will be coagulated, and the filtrate from the pure substance will give no precipitate with tannin; gelatin, however, is not so coagulated, and will give a copious precipitate with that reagent. In this filtrate also, dextrin, gums, and other adulterants, if present, will be found.—J. O. B.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Precipitated Chalk, Dimorphism of. M. Adler. Zeits. angew. Chem. 1897, 431.

In precipitating calcium carbonate, the speed with which the trimetric crystals of arragonite change into the rhombohedra of calcspar depends partly on the temperature of the liquids, but rather on that of the ammonium carbonate solution than on that of the calcium chloride; and below 32° C., the more concentrated the alkaline liquid the more permanent are the rhombs. This is due to the dissociation of the ammonium carbonate, for, as Rose pointed out, in an atmosphere of carbonic acid gas only calcspar is produced. If it be necessary to work with dilute solutions, and it be desired to obtain arragonite, the temperature should be kept as low as possible, and must not exceed 22°. The colder the liquids the smaller and more stable are the rhombic crystals. Increased pressure also assists the conversion, for while the rhombs will bear a pressure of 2 atmospheres without change, on raising it to 4 or 5, rhombohedra begin to be produced, and at 6 atmospheres the precipitate feels gritty between the fingers. Dilution of the calcium chloride with calcium sulphate solution, instead of plain water, causes the arragonite to be as permanent as though it had been thrown down from concentrated liquids. If the chalk be precipitated by means of sodium carbonate, it will bear a temperature of 100° without alteration; but in the event of using the ammonium salt, it should be dried as quickly as possible at or below 60° C. in a current of air.—F. H. L.

Nitrates in Presence of Ether, Action of Dilute Nitric, Sulphuric, Hydrochloric, and Phosphoric Acids on. C. Tanret. Bull. Soc. Chim. 1897, 497—503.

When two liquids insoluble or only slightly soluble in each other are shaken together, and one of them contains a substance soluble in either, the two liquids divide the substance in such a manner that the quantities dissolved in the same volume of each are in a constant ratio. Berthelot and Jungfleisch have characterised this as the *coefficient of distribution*. In the case of an aqueous solution of an acid shaken with ether, the coefficient varies from 1:91 to 1:130 for tartaric acid and from 1:1·2 to 1:1·23 for acetic acid, whilst for dilute sulphuric, hydrochloric, and phosphoric acids, it is as low as from 1:2700 to 1:500. Thus, owing to the great difference in their coefficients of distribution it is possible to separate for the greater part, organic acids from these three mineral acids. Nitric acid, however, in a liquid rich in ammonium nitrate, passes over into the ether like an organic acid, but when the ether is shaken with water the acid is taken up by the latter, and hence nitric acid has different coefficients according as to whether

ammonium nitrate be present or not. As an outcome of this observation and by acting on alkaline nitrates with dilute acids, the author's experiments have led to the following conclusions:—Since sulphuric, hydrochloric, and phosphoric acids liberate nitric acid from nitrates, the presence of a small quantity of these free acids is easily detected in a liquid, if after having saturated it with an alkaline nitrate, it is extracted with ether and the latter is then shaken with water, when the nitric acid which passes into it can be readily characterised.—T. A. L.

Sulphantimonites of Silver. Pouget. Comptes Rend. 1897, **124**, [26], 1518—1520.

Potassium sulphantimonite, with silver nitrate in dilute solution, gives a black precipitate of SbS_3Ag_3. If the potassium sulphantimonite be strong, and in sufficient excess, this black precipitate, formed in the first instance, is converted rapidly into a yellow crystalline precipitate of SbS_3Ag_2K. This salt can also be obtained by the action of potassium sulphantimonite on previously formed silver sulphantimonite. It is decomposed by alkaline sulphides with formation of silver sulphide, and by the action of water is broken up, slowly in the cold, more rapidly when heated, into potassium and silver sulphantimonites.

Whatever the concentration or the temperature of the solution of potassium sulphantimonite, addition of a sufficiency of silver nitrate determines the formation always of the black precipitate, which, when so formed is not of constant composition, containing always more Ag_2S than corresponds with the formula SbS_3Ag_3, but which is always free from potassium.

The salt SbS_4AgK_2 has not yet been obtained by the author.—J. T. D.

Petroleum in the Cavities of Fossils, Occurrence of. F. C. Phillips. Proc. Amer. Phil. Soc. **36**, [154], 121—126.

A case of irregular deposition of oil and bitumen in coral, occurs in a quarry at Williamsville, N.Y. Owing to the quantity present and the apparent absence of sediment above the coral, it does not seem as though the oil could have been formed *in situ* from the comparatively small amount of vegetable matter in the coral cells at any one time during the growth of the reef. The cause of infiltration may probably be a displacement by water of oil formerly distributed in the adjacent (limestone) rock.—C. S.

New Books.

Manuale del Chimico e dell' Industriale. Raccolta di Tabelle, di Dati Fisici e Chimici e di Processi d' Analisi Tecnica ad uso dei Chimici Analitici e Technici, dei Direttori di Fabbriche, dei Fabricanti di Prodotti Chimici, degli Studenti di Chimici, ecc., ecc. Del Dr. Luigi Gabba, Prof. Ord. di Chimica Tech. nel R. Instit. Techn. Superiore e nella R. Scuola Super. d' Agricolt. di Milano. Seconda Ediz. Ampliata. Milan: Ulrico Hoepli, Editore-Libraio della Real Casa. 1898. Price L. 5·50. London: H. Grevel and Co., 33, King Street, Covent Garden.

This little work, resembling Biedermann's Chemiker-Kalender, is furnished with data and tabulated matter bearing reference to the various branches of Chemical Industry, as well as to weights and measures, temperatures, specific gravities, &c. The book contains preface, table of contents, 128 pages of subject-matter, and an alphabetical index. The general plan is as follows:—I. Money System and Weights and Measures. II. Physical Data. III. Chemical Data. IV. Applied Chemistry: i. Chemical and Technical Analysis of Water. ii. Analysis of Minerals and Metals. iii. Analysis of Alloys. iv. Analysis connected with the Alkali Industry. v. Composition and Analysis of Glass. vi. Analysis connected with the Ceramic Arts. vii. Mortars and Cements. viii. Fuels and Illuminants. ix. Fatty Matters, Waxes, and Stearin. x. Oils, Fats, &c. xi. Analysis of Mineral Lubricating Oils. xii. Analysis of Resins and Varnishes. xiii. Soaps, and Data relating to Saponification. xiv. Textile Fibres. xv. Agricultural Products, &c. xvi. Food Analysis, &c. xvii. Dyestuffs. xviii. Sugars, Starches, &c. xix. Tanning. xx. Explosives. xxi. Analysis of Urine, &c. xxii. Photography. xxiii. Chemical Data on Hygiene. xxiv. Various Data.

La Fabricazione dell' Acido Solforico, dell' Acido Nitrico, del Solfato Sodico, dell' Acido Muriatico. Del Dr. V. Vender, Chimico Consulente a Milano, già Chimico e Direttore di Fabbriche. Milan: Ulrico Hoepli, Editore-Libraio della Real Casa. London. H. Grevel and Co., 33, King Street, Covent Garden. 1897. Price L. 3·50.

Small volume, pocket-book size, like the preceding, containing subject-matter, data, and tables, &c., with reference to the Manufacture of Sulphuric Acid. There are 312 pages of subject-matter, illustrated with 107 woodcuts, and an alphabetical index of authors and inventors. The work is divided into four sections, as follows:—Manufacture of Sulphuric Acid. Manufacture of Nitric Acid. Manufacture of Hydrochloric Acid. Tables.

Leghe Metalliche ed Amalgame Alluminio, Nichelio, Metalli Preziosi e Imitazioni Bronzo, Ottone, Monete e Medaglie, Saldature. Dell' Ingegnere I. Ghersi. Milan: Ulrico Hoepli, Editore-Libraio della Real Casa. London: H. Grevel and Co., 33, King Street, Covent Garden. 1898. Price L. 4.

Small volume, pocket-book size, like the preceding, containing subject-matter covering 405 pages, and furnished with curve diagrams, tables, &c., and alphabetical indexes of subject-matter and authors' names. The sub-divisions of the text are as follows:—I. Alloys in General. II. Binary Alloys. III. Alloys of Aluminium, &c. IV. Nickel Alloys. V. Amalgams. VI. Gold Alloys. VII. Silver Alloys. VIII. Platinum Alloys. IX. Alloys for Coinage and Medals. X. Bronzes. XI. Ternary Alloys, &c., Delta Metal, Fusible Alloys, Solders, &c. XII. Appendix.

Die Untersuchung, der Schmiermittel und Verwandter Produkte der Fett- und Naphta-Industrie. Von Dr. D. Holde, Vorsteher der Abth. für Oelprüfung an der königl. mech.-tech. Versuchsanst. zu Charlottenburg. Berlin: Verlag von Julius Springer. 1897. Price M. 7. London: H. Grevel and Co., 33, King Street, Covent Garden.

8vo volume containing 256 pages of text, illustrated with 59 wood engravings, and an alphabetical index of subject-matter. The treatise deals with the following subjects:—A. Lubricants and their Requirements. B. Lubricating Arrangements, &c. C. Composition, Properties, and Production of Lubricating Oils. D. General Methods of Testing: i. General Properties. ii. Physical Tests. iii. Chemical Tests. E. Special Tests. i. Of Vegetable and Animal Oils and Fats. ii. Of Mineral Lubricating Oils. iii. Of Mixtures of Mineral and Fatty Oils. iv. Testing Lubricants of Considerable Consistency. v. Testing Oils for Tool Machinery. F. Appendix.

Chemisch-technisches Repertorium. Uebersichtlicher Bericht über die neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der technischen und industriellen Chemie, mit Hinweis auf Maschinen, Apparate und Literatur. Herausgegeben von Dr. Emil Jacobsen. 36. Jahrgang. 1897. Erstes Halbjahr. Erste Hälfte. Mit in den Text gedruckten Illustrationen. Berlin: R. Gaertner's Verlagsbuchhandlung, Hermann Heyfelder, S.W., Schöneberger strasse 26. London: H. Grevel and Co., 33, King Street, Covent Garden.

This number, for the first half-year of 1897, treats of the following subjects of applied chemistry:—Building Materials, Cements, and Artificial Stones. Dyestuffs, Colours, and Calico-Printing. Fats, Oils, Illuminants, and Fuels. Fermented Liquors. Tanning, Leather, and Glue Manufacture. Textiles. Glass and Earthenware. Wood and Horn. India-Rubber and Gutta-Percha. Cements and Agglutinants. Lakes, Varnishes, and Paints. Metals.

Trade Report.

TARIFF CHANGES AND CUSTOMS REGULATIONS.

(*From the Board of Trade Journal.*)

Belgium.

Tariff Modifications.

The *Moniteur Belge* of the 22nd August last, publishes the provisions of a law which passed the Chamber on the 6th August last. The principal articles of the above law are as follows:—

Art. 1. The import duty on tea is abolished.

Art. 2. Beetroots must pay an import duty of 1 fr. per 1,000 kilos. from 1st July 1898.

Art. 3. Sec. 1. The final paragraph of the first section of the first article of the law of the 16th April 1887 is to read as follows:—

"Syrups and molasses of a total saccharine strength of less than 50 per cent., 18 frs per 100 kilos."

Sec. 2. The second section of the article cited above is to read as follows:—

"Refined sugar, known as 'vergeoises,' brown sugar, or bastard sugar are assimilated to raw sugars as far as the import duties are concerned. Syrups and molasses of a total saccharine strength of 50 per cent. or more, as well as 'melades,' are assimilated to raw sugars of the 4th class."

Art. 4. Sec. 1. The importation, manufacture, transport, storing, or sale of saccharin and similar substances are forbidden. This prohibition applies to products containing saccharine or similar substances.

Sec. 2. By substances similar to saccharine are meant chemical products having a sweet taste, but of no value as an article of food.

Sec. 3. Exception to the above articles will be made in favour of saccharin and similar substances imported by pharmacists for medicinal purposes.

Sec. 4. The Minister of Finance will determine the conditions and formalities to be observed by pharmacists benefiting by Sec. 3.

The abolition of the duty on tea will come into force on 1st January 1898.

The alteration in the duty on syrups and molasses came into force on 23rd August last.

Venezuela.

Tariff Modifications.

The following modifications in the Customs tariff of Venezuela are published in the *South American Journal* of 24th July last:—

The following is a list of such articles as enter duty free:—Mineral waters, which formerly paid 4 cents per kilo.

The following, second class, pay 2 cents per kilo.:—Liquid carbonic gas, elbow tubes, glass dust.

Third class, 5 cents per kilo.:—Asbestos, Muscovado sugar, formerly prohibited; gum arabic, formerly 25 cents per kilo.; butter, excluding oleomargarine; chemical products for curing skins, which formerly paid 25 cents per kilo.

Fourth class, 15 cents per kilo.:—Refined sugar, formerly prohibited, harness oils, oil cloth, naphtha, lard.

Fifth class, 25 cents per kilo.:—Alcohol of every sort except cane above 22° Cartier, which will pay duty corresponding to the sixth class, 50 cents per kilo.

Seventh class, 1 dol. per kilo.:—Matches, formerly in this class, are prohibited.

New Customs Tariff of Cape Colony.

An Act to amend the Customs Union Tariff Act of 1889 came into force on the 1st July last, and the following are some of the rates of duty levied thereunder:—

Schedule A.

Class I.—*Special Rates.*

No.	Article.	Rate.	£	s.	d.
1	Ale, beer, and cider, all kinds of strength exceeding 2 per cent. of proof spirit: (*a.*) In vessels of not greater content than one imperial quart	Per imp. gall.	0	1	6
	(*b.*) In other vessels, bulk or wood	"	0	1	3
2	Acetic acid	"	0	3	0
4	Blasting compounds, including all kinds of explosives suitable and intended for blasting, and not suitable for use in firearms.	Per lb.	0	0	3
7	Cement	Per 100 lb.	0	0	6
23	Gunpowder and other explosives suitable for use in firearms	"	0	0	6
		and 9 per cent. *ad val.* in addition).			
28	Ochre (commonly known as "Kaffir ochre").	Per 100 lb.	0	5	0
29	Oils: (*a.*) Paraffin and kerosene	Per imp. gall.	0	0	3
	(*b.*) Anthracene, brick, coal- or gas-tar, cocoanut, cotton seed, hemp seed, palm and palm kernel, shale, and any other kinds, being in a crude and unrefined state, for making antifriction grease, candles, or soap, or burning as fuel in oil engines, not including fish-oil.	10 % *ad valorem.*			
	(*c.*) Essential and perfumed	20 % "			
	(*d.*) Other than above, including fish-oil	Per imp. gall.	0	1	0
34	Salt, rock	Per ton	0	2	0
35	Salt, common, not including refined or table salt.	"	0	5	0
36	Soap, not including toilet soaps and soap powders and extracts.	Per 100 lb.	0	4	2
38	Spirits: (*a.*) Perfumed	Per imp. gall.	1	0	0
	(*b.*) Liqueurs and cordials exceeding 2 per cent. of proof spirit.	"	0	15	0
	(*c.*) Other sorts, exceeding 2 per cent., but not exceeding the strength of proof by Sykes' hydrometer, and so on in proportion for any greater strength.	"	0	15	0
39	Sugar: (*a.*) The produce of the cane, not refined, golden syrup, molasses, saccharum, and treacle.	Per 100 lb.	0	3	6
	(*b.*) Not cane, and all refined sugars	"	0	5	0
47	Vinegar, of standard strength, fit for immediate use as such (*i.e.*, requiring no more than 40 grms. of $KHCO_3$ to neutralise 1 oz. troy): (*a.*) In bottles or other vessels of the capacity of not more than one imperial quart.	Per imp. gall.	0	1	6
	(*b.*) Do., do., in larger vessels or in bulk.	"	0	0	6
	(*c.*) Concentrated extract or essence, of greater strength than above.	"	0	3	0

Class II.—*Free.*

No.	Article.
58	Asbestos packing and boiler composition.
68	Brass and copper in bars, unmanufactured.
79	Gambier, myrobolans, sumach, and other dyestuffs for leather (in bulk).
82	Fireclay and firebricks.
84	Flowers of sulphur and other substances (in bulk) for destroying diseases in animals or plants.
87	Glue.
88	Guano and fertilisers generally.
92	Lead.
93	Leather in the piece, all kinds.
99	Papers for printing or lithographic purposes.
102	Potash or soda, carbonate, bicarbonate, caustic, crystals, and silicate (in bulk).
108	Resin and carbonate of ammonia.
113	Sheep dip, powder, and dipping tanks.
119	Tin and zinc.
123	Vaccine virus, and toxin.

Class III.—*General: Ad Valorem 9 per Cent.*

131	All goods, wares, or merchandise not elsewhere charged with duty, and not enumerated in the free list, and not prohibited to be imported into the Union, shall be liable to a duty of 9 per cent. *ad valorem.*

Class IV.—*Special: Ad Valorem* 20 *per Cent.*

The following articles shall be liable to a duty of 20 per cent. *ad valorem*:—

135 Extracts and essences of all kinds used as food, flavouring, or perfumery, including saccharine.
136 Fireworks of all descriptions.
137 Patent and proprietary chemicals, drugs, or medicines, and all medicated foods.
138 Perfumery, cosmetics, powders, and soap or other preparations for toilet use, and soap powders and extracts.

New Tariff of Japan.

U.S. Cons. Reps., Sept. 1897, 91.

The United States Minister at Tokyo reports that a statutory Tariff Bill regulating the Customs dues to be levied in all cases where Japan is not bound by treaty stipulations, was passed by the lower house of the Diet on the 17th March, and has received the Imperial assent.

The following are taken from this law:—

(*a.*) *Tariff annexed to Protocol of the Anglo-Japanese Treaty signed at London, July* 16, 1892.

	Ad Val. Per Cent.		Ad Val. Per Cent.
Caoutchouc, manufactures of	10	Oil, paraffin	10
Cement	5	Paint in oil	10
Glass, uncoloured	8	Paper, printing	10
„ stained	10	Sugar, refined	10
Indigo, dry	10	Saltpetre	5
Lead	5	Tin, block	5
Leather, sole	15	„ plates	10
„ other kinds	10	Wax, paraffin	5
Mercury	5	Zinc, pig	5
		„ sheet	7½

(*b.*) *Tariff annexed to the Protocol of the Japanese-German Treaty* (*Official Gazette, Nov.* 20, 1896).

	Ad Val. Per Cent.		Ad Val. Per Cent.
Lead, pig	5	Dyes and paints:—	
Chemicals, drugs, and medicines:—		Anilin and alizarine dyes	10
Phosphorus, red	10	Logwood extract	10
Subnitrate of bismuth	10	Paints in oil	10
Bromides	10	India-rubber manufactures	10
Quinine	8	Leather, sole	15
Chlorate of potash	10	„ other sorts	10
Dynamite	10	Papers	10
Iodide of potassium	10	Paraffin oil	10
Nitrate of potash	5	„ wax	5
Salicylic acid	10	Portland cement	5
		Zinc, block	5
Window glass, plain	8	„ plates	7½
„ stained	10	Sugar, refined	10

(*c.*) *Tariff as passed by Bill of Imperial Parliament and published in the Official Gazette.*

Class I.—Dutiable.

Section I.—Scientific Apparatus.

	Ad Val. Per Cent.
Crucibles	10
Chemical apparatus	10
Photographic apparatus	15

Section II.—Foods and Drinks.

Mineral waters	10
Salt, crude	10
„ refined	15

Section IV.—Drugs, Chemicals and Medicines.

Under this head 71 articles are mentioned, the rates being 10 per cent., except for alcohol, 40 per cent., and musk, natural and artificial, 15 per cent.

Section V.—Dyes and Paints.

Under this head 30 articles are mentioned, the rates being 10 per cent., except for gold, silver, and platinum fluids, which are charged 15 per cent.

Section VI.—Glass.

Window glass, plain	10
„ stained	15
Plate glass	20
Looking-glasses	25
All other glassware	20

Section VIII.—Skins.

Leather, sole	15
„ other kinds	15

Section IX.—Metals.

Antimony	5
Brass	10
Copper, block and coin	5
„ other kinds	10
German silver	10
Lead, pig	5
„ pipes	10
Mercury	5
Nickel	5
Platinum, block	5
„ bars, wires	10
Solder	5
Tin and zinc, block	5
„ plates	10

Section X.—Oil and Wax.

Candles	15
Oils	10

Section XI.—Paper.

Paper	15
Ink	15
Sealing wax	15

Section XII.—Sugar.

	Ad Val. Per Cent.
Sugar, ordinary	5
„ loaf	20
„ candy	25
„ molasses	10
„ syrup	10

Section XIII.—Textiles.

Oilcloth and linoleum	15

Section XV.—Liquors.

Beer and stout	25
Brandy, gin, and whisky	40

Section XVI.—Miscellaneous.

Asbestos plates	10
Blasting gelatin and other similar explosives	15
India rubber and gutta percha:—	
Raw	5
Sheets	10
Worked	20
Celluloid, sheets	10
„ worked	20
Portland cement	5
Coke	15
Dynamite	15
Fireworks	30
Glue	5
Gunpowder	15
Guncotton	15
Lard and tallow	10
Matches	20
Oil paints and photographs	25
Asphalt and tar	5
Plaster of Paris	5
Graphite	5
Porcelain and earthenware	20
Pulp for paper making	5
Sandal wood	10
Soap, toilet	20
„ other	10
Perfumeries	30

Class II.—Free of Duty.

Bone ashes.
Guano.

Class III.—Prohibited.

Drugs, chemicals, medicines, beverages, and provisions of impure nature, to be regarded injurious under laws and orders.
Articles infringing the law of the Empire relating to patent designs, trade marks, and copyrights.
Opium.

EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

The Phosphate Concessions in Algeria.

Bd. of Trade J., Sept. 1897, 338.

A despatch, dated 5th August last, has been received at the Foreign Office from Sir Edmund Monson, Her Majesty's Ambassador at Paris, enclosing copy of the report laid before the Chamber of Deputies by the Commission appointed to examine a Bill for the working of the phosphate beds in Algeria.

This document commences with an exhaustive account of the present condition of the phosphate trade, and then goes on to examine the steps necessary for regulating the Algerian workings.

The Commission are of opinion that, as France possesses a sufficient supply of phosphates for all her wants, there is no need to monopolise for her use any of the Algerian produce; and they are in favour of steps being taken to promote the development of this industry as a source of wealth to the colony. They consider, therefore, the proposal of the Government to establish an export duty of 2 frs. per ton as unnecessary.

The Commission also come to the decision that all the phosphate beds in the extreme south of Algeria should belong to the State.

Although no express statement is made, it would appear that, as far as the Commission are concerned, there is no intention to interfere with the concessions granted to Messrs. Crookston Brothers, of Glasgow, and the Constantine Phosphate Company, of Leith, but no information is to hand as to the actual state of their case, which, from the last reports, was still under the consideration of the Conseil d'Etat.

Decline in the Bavarian Glass Trade.

Bd. of Trade J., Sept. 1897, 336.

A despatch, dated 31st July last, has been received at the Foreign Office from Mr. V. Drummond, Her Majesty's Minister at Munich, reporting a serious decline in the Bavarian glass trade with America.

At Furth, one of the most important manufacturing towns in Bavaria, the glass exports to America, which amounted in 1891 to 380,000*l.*, were last year only 185,000*l.* Since 1893, the year of the commercial crisis, Bavarian glass manufacturers have had to cut down the working power of their establishments, which, even with this diminished power, they have been keeping up at a great sacrifice in the hopes of better times. The new American tariff, however, not only dissipates the idea of any improvement, but makes it clear that the Bavarian glass industry is placed in a serious position, and the preservation of the United States market, which formerly took more than three-fourths of the whole Bavarian looking-glass trade, is now a question of life for it.

Establishment of a Sugar Refinery at Lourenço Marques.

Bd. of Trade J., Sept. 1897, 338.

A despatch, dated 4th August last, has been received at the Foreign Office from Sir H. G. MacDonell, Her Majesty's Minister at Lisbon, stating that, according to the *Diario de Noticias*, the Royal Commissioner of Mozambique has sanctioned the concession granted by the Governor of Lourenço Marques to Mr. Johannes Bang for the establishment in that district of a sugar refinery, advantage being taken of the produce of the same for the distillation of alcohol.

The alcohol thus produced will pay a duty of 27 reis per litre on exportation, or of 260 reis a litre when destined for local consumption.

Foreign Competition in Canada.

Chem. and Druggist, Sept. 25, 1897.

The following extracts from the statistics appended to the recent Blue Book relate to the most important chemicals:—

Bleaching Powder.—Britain is squeezing out America, but France is appearing as a competitor. The development of alkali manufacture in the States since 1894, however, may again have altered the proportions in the past three years. In 1884 Canada bought 21,305 dols. worth in the States and 36,846 dols. from Britain; in 1894 the States supplied her with only 5,995 dols. worth, and the mother country with 43,877 dols. Other nations are practically out of this business.

Borax.—In this article Britain has taken complete possession of the field. In 1884 Canada bought 15,000 dols. worth from the States and the same amount from Britain. Five years afterwards the proportions were 12,341 dols. and 5,108 dols.; and in 1894 Britain supplied 35,887 dols., the States only 2,303 dols. This is a growing business, and worth watching.

Bottles (including Carboys and Demijohns).—Defeat of the States by Germany, with the incidental outsqueezing of Britain and France. Belgium and Austria are also getting more than a look-in in the Canadian bottle-trade. Here is the proof:—

—	1884.	1889.	1894.
	Dols.	Dols.	Dols.
From United Kingdom	86,027	26,134	22,215
„ U.S.A.	253,232	59,174	77,097
„ Germany	22,742	49,870	95,214
„ France	9,973	3,163	3,616

Acids.—Britain has lost a little ground during the past decade. The importation of many acids, such as sulphuric, has ceased almost entirely since 1884, because Canada now manufactures these goods for herself; but the principal loser by this process of emancipation has been the United States, not Britain. In acetic acid the total importation of Canada has fallen from 19,101 dols. in 1884 to 9,228 dols. in 1894. The share of Britain has decreased from 3,984 dols. to 1,354 dols., of U.S.A. from 4,140 dols. to 479 dols. But Germany has kept her lead—7,900 dols. in 1884, 7,395 dols. in 1894. Of sulphuric acid Canada imported 18,033 dols. worth in 1884, and only 1,648 dols. in 1894.

Alum and Aluminous Cake.—The balance of imports has been shifted from Britain to the United States. Here are the figures:—

—	1884.	1889.	1894.
	Dols.	Dols.	Dols.
From United Kingdom	17,329	16,538	11,572
„ U.S.A.	5,137	5,428	13,507
„ Germany	..	1,431	310

Of late years Belgium has also shipped parcels of alum.

Cream of Tartar.—The subjoined figures speak for themselves:—

—	1884.	1889.	1894.
	Dols.	Dols.	Dols.
Great Britain	57,683	94,392	36,997
United States	24,192	14,421	15,573
France	46,639	50,432	50,564
Total	128,514	159,245	103,134

Phosphorus.—Britain holds her own in this trade, while America is falling off. No other countries participate in the business, but as time goes on Canada will probably manufacture all her own requirements. The figures are:—

—	1884.	1889.	1894.
	Dols.	Dols.	Dols.
From United Kingdom	9,058	7,960	8,308
„ U.S.A.	3,092	122	1,059

Potashes.—Britain is rapidly losing ground in this department, but it must be added that the imports of potash salts into Canada are decreasing all round. Of saltpetre the Dominion imported 38,219 dols. in 1884, but only 16,978 dols. in 1894. In the former year Britain supplied 19,950 dols., Germany (together with Holland, whose saltpetre exports must have been of German origin), 17,879 dols. worth. In 1894 the figures were:—Britain, 3,662 dols.; United States of America, 4,081 dols.; Germany, 9,235 dols. In crude muriate and bichromate of potash we are also losing trade, and Germany is stepping in. The figures were:—

—	1884.	1889.	1894.
	Dols.	Dols.	Dols.
From United Kingdom	35,898	16,730	15,290
„ U.S.A.	2,666	12,600	8,268
„ Germany	..	..	5,494

Other potash salts are very little imported.

Soda-salts.—In the department of soda-salts Britain has always held the lead among the providers of Canadian requirements. But it may be noted with satisfaction that while the annual imports of the Dominion in this branch of business are growing slowly, they are more and more falling into British hands. The United States are distinctly losing ground. Look, for instance, at the figures relating to soda-ash, nitrate, caustic, crystals, and silicate:—

—	1884.	1889.	1894.
	Dols.	Dols.	Dols.
From United Kingdom	201,128	204,974	241,893
„ U.S.A.	114,416	55,254	93,484
„ other countries	..	20	1,205

And in bicarbonate of soda we have the field practically to ourselves:—

——	1884.	1889.	1894.
	Dols.	Dols.	Dols.
From United Kingdom	27,205	26,133	44,949
" U.S.A.	1,541	2,110	1,737

No other countries compete.

Soaps.—The Canadian Customs divide soaps in five great classes, which, in order of value, are as follows:—(1) Soap powders, sapolio, &c.; (2) Perfumed or toilet soaps; (3) Common brown and yellow soap; (4) Castille and white soap; and (5) Common soft soap, saddlers' soap, and silver soap. In Class 1 the U.S. hold the field. In 1894 they provided 49,723 dols. worth out of a total of 53,517 dols. Britain supplying the rest. This is a pity, for this soap-powder business has practically sprung up in the last 10 years. But in toilet soap Britain leads, and is steadily gaining ground, rather at the expense of the United States. The figures are interesting:—

——	1884.	1889.	1894.
	Dols.	Dols.	Dols.
From United Kingdom	14,109	15,936	19,896
" U.S.A.	25,029	20,196	21,323
" France	2,007	899	1,588

There are practically no other competitors.

In Class 3 (common soaps) Britain has completely taken the place of the United States, as will be seen by the following figures:—

——	1884.	1889.	1894.
	Dols.	Dols.	Dols.
From United Kingdom	9,701	6,874	35,220
" U.S.A.	28,067	6,307	5,219

Class 4, Castile soap, is in the hands of France, Marseilles being the headquarters of this industry, which depends upon cheap olive oil for its success. The imports are increasing, and in 1894 were worth 17,170 dols. Class 5 is a small one, but here also Britain is displacing the States:—1884, United States of America, 4,366 dols.; Britain, 498 dols. 1894, United States of America, 1,240 dols.; Britain, 1,889 dols. The total soap imports of Canada in 1894 were worth about 160,000 dols.

Instruments.—There is a rapidly-growing import business in surgical, optical, and philosophical instruments, but in these branches, in which ten years ago Britain led the way, she has been hopelessly outstripped by the United States and Germany. The figures are melancholy reading:—

——	1884.	1889.	1894.
Surgical instruments:—	Dols.	Dols.	Dols.
From United Kingdom	6,875	12,678	8,609
" U.S.A.	5,558	11,578	25,596
" Germany	..	1,083	10,020
" France	..	1,851	1,379

and in optical instruments (including spectacles, microscopes, and telescopes) our discomfiture is still worse:—

——	1884.	1889.	1894.
	Dols.	Dols.	Dols.
From United Kingdom	23,277	18,287	16,762
" U.S.A.	29,289	30,483	69,608
" Germany	1,729	4,011	8,707
" France	10,639	16,339	16,985

In india-rubber goods much the same sort of thing is going on. Our defeat in this branch of business is nothing short of a disgrace to this country. We held the market when it was small. Now that it is worth 200,000 dols. a year at least, we have been practically thrown out.

Patent Medicines.—The imports of patent medicines into Canada equal in value those of surgical and philosophical instruments. But while we have lost our grip of the last-named business, the British patent medicine still has a fair hold on the affection of the Canadians, though it yields the palm to the American nostrum. Of patent medicines the imports were:—

——	1884.	1889.	1894.
	Dols.	Dols.	Dols.
From United Kingdom	27,386	43,078	36,740
" U.S.A.	113,713	118,199	141,620
" France	4,419	11,421	11,383

GENERAL TRADE NOTES.

The Canadian Leather Industry.

Bd. of Trade J., Sept. 1897, 354.

The *Monetary Times* (Toronto) states that the export of Canadian leather is a trade of recent growth, but if it continues to develop as it has done in the past 10 years, it will soon become of national importance. The annual value of sole and upper leather exported from Canada during the last decade is as follows:—

Years.	£	Years.	£
1887	92,000	1892	211,000
1888	62,000	1893	180,000
1889	130,000	1894	328,000
1890	151,000	1895	265,000
1891	181,000	1896	397,000

The Chilian Nitrate Industry.

Bd. of Trade J., Sept. 1897, 350.

According to the *Chilian Times* the production of nitrate in 1896 amounted to 23,832,434 quintals, and the exportation amounted to 24,066,189 quintals. The principal countries to which nitrate is exported from Chile are Germany, United Kingdom, France, United States, and Holland.

The following table shows the world's consumption of nitrate for the last seven years:—

Years.	Spanish Quintals.	Years.	Spanish Quintals.
1890	20,244,523	1894	22,750,901
1891	20,969,785	1895	23,858,193
1892	20,194,852	1896	24,604,150
1893	20,153,406		

Consequently, since 1891 to 1896 there has been an increase in consumption over 1890 of 4,359,627 quintals, or close upon 200,000 English tons.

Mineral Industry of Bosnia-Herzegovina in 1896.

Bd. of Trade J., Sept. 1897, 348.

The *Revue d'Orient* states that, according to official returns, the mineral production of Bosnia-Herzegovina in 1896 was as follows:—

——	1896.	1895.
	Tons.	Tons.
Copper ore	4,003	1,776
Iron ore	23,213	12,735
Chrome ore	442	706
Manganese ore	6,821	8,145
Lignite	222,724	195,496
Copper	206	105
Iron, raw	10,120	2,569
Cast goods	1,039	1,080
Salt (kitchen)	13,720	12,758

The Production of Petroleum.

Bd. of Trade J., Sept. 1897, 267.

The following particulars, derived from various official and other sources, show the state of the industry in some of the principal petroleum-producing countries:—

Russia.—According to a recent report of H.M. Consul at Batoum, the total quantity of naphtha produced by the wells of the oil fields in the neighbourhood of Baku during the past year was in excess of that of the year preceding; the figures being 6,229,060 tons for 1896, against 6,085,431 tons in 1895, or an excess for 1896 of 143,629 tons.

The quantity of crude oil produced by the flowing wells was 1,403,218 tons in 1896, against 1,817,704 tons in 1895; and by pumping wells 4,825,842 tons in 1896, as compared with 4,267,727 tons in 1895.

The naphtha-producing territories at Grozni, in the Northern or Cis-Caucasus, in Galicia, Java, Japan, and Peru, are slowly but surely coming into prominence. Hitherto the Baku mineral oil industry has had to compete with only one serious rival, *i.e.*, America, but now the following fresh factors must be counted with, namely:—

The discovery of oil in the district of Grozni, which took place in 1893, and is annually assuming wider proportions. It is stated that there are at present seven firms at work there, obtaining oil in unlimited quantities, which, although it may probably not be of such good quality for refining purposes as the oil produced by the wells on the shores of the Caspian Sea, is nevertheless sufficiently good for burning as fuel.

The extended development of the mineral oil industry in Galicia has had the effect of bringing about a sensible decrease in the quantity of distillate shipped from Batoum to Austria-Hungary during the past year, which circumstance may eventually have the same effect on the markets in Germany, where, according to reports, it is proposed to increase the import duties on mineral oils reaching that country from Russia and America, and to draw the supplies for home consumption in the shape of the crude material from Galicia, erecting in Germany the necessary works for refining purposes.

Dutch East Indies.—There is increased production of petroleum in Java, and the supplies from that island to Chinese ports are steadily increasing, although it is stated that the kerosene is of inferior quality.

For the working of Javanese petroleum, a company was formed at Amsterdam in March last as a successor to the Dordt'sche Petroleum Maatschappj, which was organised in 1887 and reorganised in 1890.

A recent issue of *Kuhlow's* reports that this company commenced operations in the district of Djaba Kota, residency of Soerabaja, and secured leases and concessions for the best oil territories on the island. It also took an interest in extensive oil lands at Langkat and Palembang on the island of Sumatra, and at Koetei on the island of Borneo.

Besides extending their operations in the residency of Soerabaja, the company, after having placed their first petroleum on the market in 1889, succeeded, in 1893, in opening up the oil fields in the residency of Renbang, which proved highly productive. The oil found there is of excellent quality, and the output permitted a rapid increase in the production of illuminating oil.

The Dordt'sche Petroleum Maatschappj intends in the first place to supply the Java and Madura markets, the consumption of which increased from about 2,500,000 cases in 1889 to 3,600,000 cases in 1896. Of the latter, 1,250,000 cases were supplied by the Dordt'sche Petroleum Maatschappj. This shows that the company can safely treble its productions before it needs to look around for outside markets. The transport facilities are very good. The company controls a fine system for handling its products, consisting of pipe lines, tank cars, reservoirs, and casing houses, which are distributed all over the island.

Statistics of the last eight years show that the Javanese production of illuminating oil has increased from 8,000 cases in 1889 to 1,250,000 cases in 1896, and the net earnings of the company from 36,697 florins to 1,923,611 florins. The production for January 1897 amounted to 108,007 cases, as against 87,577 cases for January 1896. The capital stock of the Dordt'sche Petroleum Maatschappj is 15,000,000 florins, one florin being about equal to 1*s.* 8*d.*

Roumania.—Mr. A. Percy Bennett, H.M. Acting Consul at Galatz, sent to the Foreign Office, in November 1896, a special report on the petroleum industry of Roumania, in which he says that it may be taken for granted that petroleum exists in abundance in that country.

From Turn-Severin or thereabouts, on the western frontier of Roumania, the petroleum zone can be traced at the foot of the Carpathian Hills, skirting them in their course through the country towards Bukovina and Galicia, on the north-east. Petroleum is there found in the paludinous pliocene and in the miocene formations: its existence is evident, not only by its traces on the surface, but by other indications, such as its smell and its gases. Along the whole length of the zone mentioned may been seen primitive workings of the mineral in the shape of comparatively shallow hand-dug wells. These wells, producing small quantities of oil, are scattered over an area of from 10 to 15 kiloms. From end to end of the zone there are five principal centres at which the workings have been concentrated, and at these centres have been found, collected in pockets near the surface, the largest quantities of oil of the first stratum yet discovered. These centres are:—

1st. Olt Valley, in the vicinity of Rimnic-Valcea (Valcea District).

2nd. Dimbovitza and Talomitza Valleys, in the vicinity of Tirgu-Vestei (Dimbovitza District).

3rd. Prahova and Teleagen Valleys, north of Ploiesti (Prahova District).

4th. Buzeu Valley, north of Buzeu (Buzeu District).

5th. Trotush and Tazlau Valleys, between Tirgu-Ocna to the south and Bacau to the north (Bacau District).

According to several writers, petroleum is to be found not only in the above-mentioned zone, but also in the whole of the plains down to the Danube, which would embrace a zone of, say, 100 kiloms. in width. This assertion is, to a certain extent, verified by certain and manifest signs of the existence of petroleum, but it is not known at what depth even the first stratum may be met with, as no borings for oil have ever been made in the plains, and the water wells sunk by hand there have not, up to the present, touched any oil-pocket however small. So far, at the five centres referred to above, only the first stratum has been worked to any extent. These centres comprise about 50 borings, and, say, 800 hand-dug wells. The deepest successful boring only reaches about 300 metres, and whilst the majority of the hand-dug wells range from 60 to 120 metres, none of them go below 200 metres.

The output of crude oil from these borings and wells for the year 1894–95 reached 80,000 tons.

The Consul, in his report, adds that, though petroleum has been worked in Roumania for about 25 years, yet considering the small quantity now extracted, and taking into account the extensive zone in which distinct evidence of its existence is to be met with, it is evident that the Roumanian petroleum industry is at present only in its infancy.

United States.—The year 1885 seems to have marked a new era in the history of petroleum. The importance of the Trenton limestone as an oil producer in the United States was first recognised at this time, and this year marks the discovery of the Lima (Ohio) and the Indiana fields. From the Lima field alone was derived more than one-third of all the oil produced in the United States in 1889.

The following table, taken from the report of the United States Secretary of the Interior for 1896, shows the production of crude petroleum in the United States from the year 1885 to 1895, in barrels of 42 Winchester gallons each:—

Years.	Barrels.	Years.	Barrels.
1885	21,858,785	1891	54,291,980
1886	28,064,841	1892	50,509,136
1887	28,283,483	1893	48,412,666
1888	27,612,025	1894	49,344,516
1889	35,163,513	1895	52,983,526
1890	45,822,672		

Since the beginning of operations at Titusville, Pennsylvania, in 1859, the enormous total of 709,713,403 barrels of crude petroleum have been produced in the United States. By far the largest portion of this has been produced in what is known as the "Pennsylvania and New York oil fields," these fields producing alone 516,657,260 barrels of the total of 709,713,403 barrels, or nearly 73 per cent. Ohio has produced 133,343,773 barrels, and West Virginia 37,179,604 barrels; California and Colorado have produced respectively 6,683,901 and 4,188,325 barrels, while Indiana, which did not figure as a producer of petroleum until 1889, has produced 11,341,664 barrels, more than one-third of which was produced in 1895.

Peru.—In a recent report to the Foreign Office by Her Majesty's Consul at Callao, it is stated that the petroleum industry in the north of Peru originated some 30 years ago, oil having been discovered by drilling for water on the Mancora estate, near Payta. During that period six different undertakings have been started, but so far without giving satisfactory results. Two kerosene factories exist on this coast; the kerosene produced gives a dull light, smokes, and cannot bear comparison with American kerosene, which fetches from 8 to 9 soles the case, whilst the former has to be sold at from 2 soles 50 c. to 3 soles the case of 10 gallons, and only serves for street lighting. From 4,000,000*l.* to 5,000,000*l.* have been spent on the several undertakings, without producing in any single case a fair return. Hundred of wells have been sunk, some resulting in flowing, others in pumping wells, oil being found at a depth varying from 250 to 600 feet, but all have run dry after a very short flow. Experts from the United States have carefully examined the land north and south of Payta, and have given it as their opinion that, in spite of abundant surface indications all over the district, no oil of any importance is likely to be found.

The *Manufacturer* (Philadelphia) states that the London and Pacific Petroleum Company, at Negritos, 45 miles north of Payta, has a pipe line from its wells at Negritos, and pumps the oil a distance of six miles, to Talara, on the coast, where its refinery is located, and from which point it ships its product. This company has produced 8,000,000 barrels of crude oil in the last six years, and now has a production of 200 barrels per day. One of its best wells produced 500 barrels a day six years ago, and is still making 45 barrels a day. There is but little gas in the field, and in consequence oil is used for fuel in operating the wells. Salt water is troublesome unless the wells are kept pumping continuously.

The right to drill must be secured by grant from the Peruvian Government, as it retains the mineral right in all the land in Peru. American machinery is used almost exclusively.

The Ozokerite Monopoly (Galicia).

Chem. Zeit. 21, [44], 439.

At present the syndicated mine owners control, either by ownership, option, or contract, the greater part of the shafts in the Boryslaw, Truscawiec, Dwiniacz, and Starunia districts, and produce sufficient ozokerite to supply the demand, the few outsiders being unable to produce enough to satisfy the requirements of any ceresin works, most of whom are, moreover, under engagement to buy from the syndicate alone. A few of the ceresin makers, having mines of their own, were able to make better terms than the bulk, who have to put up, as best they may, with the increased rates for raw material. The Austro-Hungarian Länder Bank, which is interested in the association, is said to be endeavouring to obtain a concession for forming a share company, to carry on the work of the syndicate.

—C. S.

Denaturing of Acetates.

Chem. and Druggist, Aug. 28, 1897, 359.

A tax of 62 frs. 50 cents is levied in Paris upon every 100 kilos. of vinegar, with a corresponding amount upon acetic acid or acetates, which thus becomes a considerable obstacle in many industries. It is now proposed to admit the acetates and acid duty free, provided they are denatured so as to unfit them for domestic use or for conversion into vinegar. Altogether six methods of denaturing are suggested, *viz.*, the addition of (*a*) 1 per cent. of Methylene Blue, ferric nitrate, copper sulphate, Rhodamine, or Victoria Blue; (*b*) 2 to 3 per cent. of copper acetate; (*c*) 3 per cent. of vitriol; (*d*) 5 grms. of soluble Naphthol Black, Blue, Red, or Yellow, per litre of acetic acid; (*e*) 7 grms. of arsenic per litre; or (*f*) 2 grms. of lead acetate per litre.

BOARD OF TRADE RETURNS.

Summary of Imports.

Articles.	Month ending 31st August	
	1896.	1897.
	£	£
Metals	1,836,201	1,572,652
Chemicals and dyestuffs	411,899	360,704
Oils	642,811	594,905
Raw materials for non-textile industries	5,036,984	5,635,240
Total value of all imports	32,480,473	33,371,385

Summary of Exports.

Articles.	Month ending 31st August	
	1896.	1897.
	£	£
Metals (other than machinery)	2,620,119	2,615,718
Chemicals and medicines	557,522	599,190
Miscellaneous articles	2,548,560	2,394,179
Total value of all exports	20,326,796	18,773,997

Imports of Metals for Month ending 31st August.

Articles.		Quantities.		Values.	
		1896.	1897.	1896.	1897.
				£	£
Copper:—					
Ore	Tons	15,055	13,053	60,743	58,556
Regulus	„	9,172	5,631	227,125	150,782
Unwrought	„	5,107	4,301	246,108	214,641
Iron:—					
Ore	„	455,218	491,411	307,998	379,908
Bolt, bar, &c.	„	6,329	6,409	49,940	53,301
Steel, unwrought	„	2,088	2,178	21,154	23,327
Lead, pig and sheet	„	15,087	11,306	165,170	136,826
Pyrites	„	34,708	48,708	60.759	79,396
Quicksilver	Lb.	2,272	.34,090	320	3,284
Silver ore	Value £	..	..	118,509	111,697
Tin	Cwt.	91,834	24,953	277,009	74,964
Zinc	Tons	5,977	5,256	102,838	89,305
Other articles	Value £	..	..	198,528	196,665
Total value of metals		..	..	1,836,201	1,572,652

Imports of Oils for Month ending 31st August.

Articles.		Quantities.		Values.	
		1896.	1897.	1896.	1897.
				£	£
Cocoa-nut	Cwt.	9,562	11,957	10,348	12,363
Olive	Tuns	813	900	28,904	28,543
Palm	Cwt.	96,850	96,573	99,270	99,116
Petroleum	Gall.	14,958,840	13,235,414	274,959	210,523
Seed	Tons	1,877	2.307	39,134	55,668
Train, &c.	Tuns	2,748	1,583	45,902	27,536
Turpentine	Cwt.	74,344	56,787	69,900	58.307
Other articles	Value £	..	..	74,394	72,849
Total value of oils		..	..	642,811	594,905

Imports of Chemicals and Dyestuffs for Month ending 31st August.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Alkali............ Cwt.	18,474	18,496	8,311	8,416
Bark (tanners', &c.) „	31,192	37,797	11,665	12,512
Brimstone .... „	8,741	47,668	2,091	11,853
Chemicals...... Value £	..	..	122,311	107,834
Cochineal .... Cwt.	272	411	1,777	2,458
Cutch and gambier Tons	2,171	2,037	42,862	29,279
Dyes:—				
Alizarin ....Value £	..	..	17,062	16,143
Anilin and other „	..	..	37,171	35,160
Indigo .... Cwt.	1,351	862	18,122	12,021
Nitrate of potash . „	26,635	22,551	20,433	16,231
Valonia .... Tons	2,422	2,673	25,325	27,306
Other articles... Value £	..	..	105,269	81,691
Total value of chemicals	..	..	411,899	360,704

Imports of Raw Material for Non-Textile Industries for Month ending 31st August.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Bark, Peruvian .. Cwt.	1,936	924	4,135	1,440
Bristles.......... Lb.	296,789	331,641	39,740	39,193
Caoutchouc ...... Cwt.	25,143	21,272	280,507	227,102
Gum:—				
Arabic......... „	3,335	7,128	6,258	14,588
Lac, &c. .... „	10,755	11,477	51,682	35,822
Gutta-percha .... „	4,748	3,238	49,219	32,249
Hides, raw:—				
Dry............ „	40,108	40,672	91,495	104,096
Wet........... „	37,099	52,249	78,125	112,778
Ivory........... „	1,126	707	41,644	31,150
Manure:—				
Guano .... Tons	813	..	3,741	..
Bones .... „	3,911	3,363	12,446	11,736
Nitrate of soda... „	938	565	7,295	4,272
Phosphate of lime „	34,278	36,621	55,377	53,885
Paraffin.......... Cwt.	47,789	62,720	46,328	53,489
Linen rags....... Tons	1,562	2,278	15,403	23,629
Esparto.......... „	14,126	15,641	57,811	68,723
Pulp of wood .... „	25,025	35,864	126,252	172,429
Rosin............ Cwt.	159,921	156,159	42,138	39,407
Tallow and stearin „	161,922	102,177	161,237	95,916
Tar ....Barrels	30,228	31,551	18,711	21,590
Wood:—				
Hewn .... Loads	314,922	332,208	696,790	762,781
Sawn .... „	970,231	1,143,801	2,129,936	2,641,104
Staves......... „	18,657	13,156	67,461	57,607
Mahogany ..... Tons	3,160	6,711	31,759	58,831
Other articles....Value £	..	..	918,414	975,423
Total value	..	..	5,036,984	5,635,240

Besides the above, drugs to the value of 75,471*l.* were imported, as against 54,778*l.* in August 1896.

Exports of Drugs and Chemicals for Month ending 31st August.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Alkali............ Cwt.	323,363	376,186	89,857	97,185
Bleaching materials „	80,993	87,085	27,699	26,921
Chemical manures Tons	35,145	50,496	135,398	174,799
Medicines....... Value £	..	..	84,215	84,732
Other articles... „	..	..	220,353	215,553
Total value	..	..	557,522	599,190

Exports of Metals (other than Machinery) for Month ending 31st August.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Brass........... Cwt.	9,264	9,048	38,437	38,470
Copper:—				
Unwrought.... „	28,791	43,456	71,041	105,772
Wrought ...... „	21,405	22,713	65,509	67,994
Mixed metal... „	11,731	15,715	29,089	37,616
Hardware...... Value £	..	..	164,123	155,043
Implements ..... „	..	..	103,959	101,299
Iron and steel ... Tons	311,596	287,719	1,917,109	1,853,705
Lead .... „	2,354	2,324	29,995	30,908
Plated wares ... Value £	..	..	27,356	29,223
Telegraph wires . „	..	..	65,176	93,475
Tin .... Cwt.	10,037	8,110	32,175	26,617
Zinc .... „	13,510	14,027	9,848	11,520
Other articles .. Value £	..	..	66,302	64,076
Total value	..	..	2,620,119	2,615,718

Exports of Miscellaneous Articles for Month ending 31st August.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Gunpowder...... Lb.	550,800	1,031,900	10,971	17,746
Military stores.. Value £	..	..	159,152	95,728
Candles.......... Lb.	1,534,600	1,831,000	22,567	24,206
Caoutchouc ..... Value £	..	..	103,071	94,363
Cement.......... Tons	25,217	36,089	43,350	60,257
Products of coal Value £	..	..	105,157	97,173
Earthenware ... „	..	..	154,087	119,448
Stoneware ...... „	..	..	19,777	12,505
Glass:—				
Plate........... Sq. Ft.	152,728	145,955	9,824	7,820
Flint........... Cwt.	7,268	7,373	16,466	15,550
Bottles......... „	57,723	56,162	27,831	26,527
Other kinds.... „	19,695	20,934	15,259	14,990
Leather:—				
Unwrought .... „	11,004	12,569	101,663	102,408
Wrought ..... Value £	..	..	40,931	38,553
Seed oil.......... Tons	5,944	3,512	92,646	60,938
Floorcloth ..... Sq. Yds.	1,651,200	1,638,900	71,323	71,656
Painters' materials Val. £	..	..	128,641	117,865
Paper .... Cwt.	70,941	80,248	112,273	118,382
Rags............ Tons	3,647	4,396	22,153	22,948
Soap .... Cwt.	57,531	59,442	59,407	57,836
Total value	..	..	2,548,560	2,394,179

Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—PLANT, APPARATUS, AND MACHINERY.

Applications.

18,950. P. Boimare. Improvements in smokeless furnaces. Aug. 16.

19,077. W. Astley. Chemical boiling-down pans expansion trough bottoms. Aug. 18.

19,196. E. Stephenson. Improvements in condensers. Aug. 19.

19,249. A. Fidler. Improvements in and relating to precipitating tanks and the like. Aug. 20.

19,344. T. Swift and G. H. Swift. A liquid composition for the removal and prevention of scale and protection from corrosion on inside of boilers. Aug. 21.

19,372. A. J. Boult.—From P. Muratel, France. Improvements in filtering apparatus. Aug. 21.

19,405. A. Cottier and J. Jacquetin. System of hydro-extractor. Aug. 23.

19,468. F. Turnbull. Apparatus for washing coal, coke, and similar materials and minerals. Aug. 24.

19,718. T. R. Berkley. Improvements in refuse-consuming furnaces. Aug. 26.

20,218. J. Weichmann. Improvements in distilling apparatus. Sept. 2.

20,349. H. J. Demuth, E. N. Perkins, and J. T. Dade. Improvements in brine-evaporating apparatus. Sept. 4.

21,285. J. L. Collier. *See* Class XVI.

21,311. G. T. Holloway. A new or improved laboratory gas furnace. Sept. 17.

21,359. A. Barr and W. Stroud. Improvements in apparatus for the production of high vacua. Sept. 17.

COMPLETE SPECIFICATIONS ACCEPTED.*

1896.

19,039. W. Defries. Filtering apparatus. Sept. 1.

22,109. W. H. Strype and J. A. Mason. Improvements in metal hampers or baskets for carboys. Sept. 15.

23,189. A. S. Barham and L. de Wittenbach. Improvements in apparatus for sterilising milk and other liquids. Sept. 8.

23,379. H. F. Stanley and The Farringdon Works, Ltd. Improvements in apparatus for refrigerating air and other gases. Aug. 25.

23,723. T. C. Taylor and J. E. Tolson. Improvements in retorts for carbonising machines. Sept. 8.

24,405. R. M. McDougall. Improvements in the packing of the tubes in the rectifiers of Coffey's distilling apparatus. Sept. 15.

28,728. P. E. Placet. Improvements in the lining of crucibles and furnaces. Sept. 22.

1897.

17,356. W. G. Heys.—From J. Klein, of the Maschinen und Armaturfabrik vormals Klein, Schanzlin, and Becker, Germany. Improvements in apparatus for cooling liquids. Sept. 1.

18,578. H. Hirzel. An improved absorption apparatus for absorbing gases in liquids. Sept. 22.

II.—FUEL, GAS, AND LIGHT.

APPLICATIONS.

18,904. J. W. Scarth and W. A. Thornton. Improvements in apparatus for the generation of acetylene gas, and like purposes. Aug. 16.

18,907. A. Denich. Portable apparatus for producing acetylene gas. Aug. 16.

18,971. J. R. Wigham. Manufacturing acetylene gas from calcium carbide. Aug. 17.

18,978. C. E. Billing. Improvements in the method of and means for the economical use of gas for lighting purposes. Aug. 17.

18,989. H. Maxim and W. H. Graham. Method and apparatus for the production of calcium carbide; applicable also to other purposes. Aug. 17.

19,149. W. E. Kochs. Improvements in coke ovens and in machinery in connection therewith. Complete Specification. Aug. 19.

19,319. E. Barnard. Improved apparatus for generating and supplying acetylene or other gases. Aug. 20.

19,411. H. J. Bell and The Niagara Falls Acetylene Gas Machine Company, Ltd. Apparatus for the production and storage of acetylene gas. Complete Specification. Aug. 23.

19,437. A. A. Flürscheim. Manufacture of "mantles" and the like for incandescent gas lights. Aug. 23.

19,442. J. Y. Johnson.—From C. Bartelt and F. Battelt, Germany. Treatment or preparation of oxygen to bring it into a form suitable for internal application. Aug. 23.

* *See* Note (*) on previous page.

19,590. F. M. Mooney. Treatment of the oxide of iron used for the absorption of sulphuretted hydrogen in the purification of illuminating or other gases or otherwise. Aug. 25.

19,615. O. Münsterberg. Improvements in apparatus for the manufacture of acetylene gas. Aug. 25.

19,765. A. A. Flürscheim. Manufacture of "mantles" or the like for incandescent gas lights. Aug. 27.

19,798. J. H. H. Duncan and G. Bourrelly. Method and apparatus for producing flame and light by the use of hydrocarbons. Aug. 27.

19,823. T. Thorp and T. G. Marsh. Improvements in apparatus for the production of acetylene from calcium carbide and water. Complete Specification. Aug. 28.

19,887. L. G. Harris. Manufacture of gas from garbage and other refuse. Aug. 28.

19,889. F. J. Lothammer. Method of and apparatus for carburising air. Complete Specification. Aug. 28.

19,951. L. Chambault. Method and apparatus for generating and burning acetylene gas, chiefly designed for use on velocipedes, autocars, boats, and the like. Complete Specification. Aug. 30.

20,011. E. J. Dolan. Improvements in acetylene gas generators. Date applied for March 19, 1897, being date of application in United States. Complete Specification. Aug. 31.

20,051. F. Rhind. Improvements in gas generating. Date applied for Feb. 27, 1897, being date of application in United States. Complete Specification. Aug. 31.

20,052. F. Rhind. Improvements in gas generating lamps. Aug. 31.

20,142. F. A. Keiffer. Improvements in apparatus for the automatic supply of carbide of calcium in the production of acetylene gas. Date applied for Feb. 4, 1897, being date of application in France. Sept. 1.

20,184. W. Dancer. Improvements in the production of gas for illuminating and heating purposes and of certain bye-products. Sept. 2.

20,225. L. Freudenthal. An improved solution or fluid for impregnating incandescent bodies. Complete Specification. Sept. 2.

20,319. E. Saizenberg. Improvements in incandescent gas lighting. Complete Specification. Sept. 3.

20,381. J. A. Sinclair and E. R. Powell. Improvements in means or apparatus for use in the production, storing, and cooling of acetylene gas. Sept. 4.

20,464. A. Chauvel and G. Ménétrier. Improvements in the manufacture of fuel cakes or blocks for industrial and domestic purposes. Sept. 6.

20,532. C. Petit and G. Gavrel. Improvements in or relating to apparatus for the manufacture of acetylene gas. Date applied for March 8, 1897, being date of application in France. Sept. 7.

20,537. F. Windham and E. Fry. Portable lamps for producing gas from carbide of calcium. Sept. 7.

20,571. O. Kahns. Process and apparatus for the production of carburetted air for incandescent gas light. Complete Specification. Sept. 7.

20,572. H. H. Leigh.—From H. Abraham and L. Marmier, France. Generating of ozone and apparatus therefor. Sept. 7.

20,574. G. Lebrun and F. Cornaille. A burner for acetylene and other gases rich in carbon. Date applied for March 4, 1897, being date of application in France. Complete Specification. Sept. 7.

20,886. J. Dymond. Apparatus for producing luminous water-gas. Sept. 11.

20,987. F. S. Thorn and C. Hoddle. Acetylene generator for lighting cycle and other vehicle lamps. Sept. 13.

21,094. G. B. Ellis.—From N. Lepage and L. M. Maisonnier, France. Improvements in apparatus for producing acetylene gas. Sept. 14.

21,114. H. R. Bean and H. Ringwood. Improvements in apparatus for the generation of acetylene and other gases. Sept. 14.

21,115. T. C. Palmer, of the firm of W. J. Fraser and Co. See Class XII.

21,150. J. Carter. The Carter incandescent gas light mantle and chimney protector. Sept. 15.

21,346. W. A. Thornton. Improvements in or relating to apparatus for the generation of acetylene gas and like purposes. Sept. 17.

21,347. W. A. Thornton. Improvement in or relating to apparatus for the generation of acetylene gas and like purposes. Sept. 17.

21,372. E. C. Chardin. Improvements in apparatus for producing acetylene gas. Sept. 17.

21,461. J. Windmüller, of the firm of Windmüller and Co. An improved device for producing and burning acetylene gas. Sept. 18. Complete Specification.

Complete Specifications Accepted.

1896.

11,109. C. Weygang. Improvements in application of artificial fuel. Aug. 25.

19,044. A. F. de Villepigue, V. Fournier, and G. Shenton. Apparatus for extracting oxygen from the air. Sept. 8.

19,521 J. S V. Bickford. Apparatus for lighting and heating by means of petroleum vapour. Sept. 8.

20,084. W. W. Hughes and S. Willson. Treatment or utilisation of peat and its products. Sept. 8.

20,090. E. Chesnay and L. Pillion. Apparatus for generating and storing acetylene gas. Sept. 1.

20,156. P. R. de F. d'Humy. Manufacture of fuel. Sept. 15.

20,254. E. Chesnay and L. Pillion. Apparatus for generating and storing acetylene gas. Sept. 8.

20,713. J. L. Waldapfel. Improvements in burning liquid hydrocarbons in conjunction with an incandescent hood or mantle to produce an incandescent oil burner. Sept. 22.

22,194. P. Jebsen. Process for carbonisation of peat by electric heating. Aug. 25.

22,828. C. W. S. Crawley, A. Soames, and C. V. Drysdale. Method of obtaining a continuous supply of gas or vapour under pressure. Sept. 15.

23,006. A. Quentin. Improvements in and relating to incandescence bodies for heating purposes. Sept. 22.

23,433. F. B. Grundy and J. Moeller. Improvements in the manufacture of appliances for effecting self-lighting of gas. Sept. 1.

23,752. W. J. Mackenzie. Improvements in apparatus for generating acetylene gas. Sept. 15.

24,611. A. Kay. Improvements in portable apparatus for generating and storing acetylene gas. Sept. 22.

25,236. E. Chesnay. L. Pillion, and C. Bertolus. Apparatus for automatically producing and storing acetylene. Sept. 15.

25,488. A. Fournier. Apparatus for the production of acetylene gas. Sept. 22.

25,827. E. Skriwan. Improvements in the method of and means for binding mantles for incandescent lights. Sept. 22.

1897.

1777. L. Denayrouze. An improvement in burners for incandescent gas lights. Sept. 1.

9714. U. Kesselring. An improved apparatus for the manufacture of acetylene gas. Sept. 1.

12,021. H. Freise. Process for manufacturing a smokeless inflammable wick. Sept. 8.

14,091. E. Jimeno. Improvements in or connected with gas holders for acetylene and other lighting gases. Sept. 8.

15,963. A. Kiesewalter. Improvements in incandescent mantles. Sept. 15.

16,145. A. George. Improvements in the manufacture of briquette fuel. Sept. 8.

16,195. G. C. Marks.—From A. F. Bowers, France. Improvements in apparatus for the generation of acetylene gas. Sept. 8.

17,021. E. Godin. Improvements in acetylene gas generators. Sept. 1.

18,005. W. P. Thompson.—From A. Julien, Belgium. Improvements in and relating to apparatus for producing heating or lighting gas by the aid of volatile hydrocarbons. Sept. 22.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Complete Specification Accepted.

1896.

23,649. J. Meikle. Improvements in obtaining benzene, toluene, and other products from mineral oils and liquid tars. Sept. 15.

IV.—COLOURING MATTERS and DYES.

Applications.

18,990. T. R. Shillito.—From J. R. Geigy and Co., Switzerland. Production of paradinitrodibenzyldisulpho acid and of new colouring matters or dyes. Aug. 17.

19,198. T. R. Shillito.—From J. R. Geigy and Co., Switzerland. Manufacture of aldehydonaptholsulphonic, aldehydonaptholcarboxylic. and aldehydonaptholsulphocarboxylic acids and of new blue and green colouring matters or dyes therefrom. Aug. 24.

19,622. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture of anthraquinone dyestuffs. Aug. 25.

19,634. O. Imray. — From H. Schneider, Germany. Manufacture of a new amido base and new direct-dyeing polyazo dyestuffs therefrom. Aug. 25.

19,749. P. Cannell-Bunn. Manufacture of colours. Aug. 27.

19,783. R. Webb. Improvements in security inks. Complete Specification. Aug. 27.

19,785. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement Gilliard P. Monnet et Cartier, France. Production of colouring matters and intermediate products. Aug. 27.

19,790. C. E. Anquetel. A new or improved ink. Aug. 27.

19,901. I. Levinstein and Levinstein, Ltd. Production of colouring matters. Aug. 30.

19,994. J. Y. Johnson. — From The Badische Anilin and Soda Fabrik, Germany. Production of new colouring matters related to the rhodamine series. Aug. 31.

20,125. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of dyestuff directly dyeing cotton black. Sept. 1.

20,278. T. R. Shillito.—From J. R. Geigy and Co., Switzerland. Manufacture of new polyazo colouring matters or dyes. Sept. 3.

20,333. W. H. Claus, A. Rée, and L. Marchlewski. Production of new derivatives of alkyl-hydroxy-para-amidobenzenes. Sept. 4.

20,410. E. S Wilson and E. Stewart. Improvements in the production of dyes. Sept. 6.

20,649 H. E. Newton.—From Farbenfabriken vormals Friedrich Bayer and Co., Germany. The manufacture or production of colouring matters derived from anthra quinone. Sept. 8.

20,827. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of new alizarine dyestuffs. Sept. 10.

21,060. I. Levinstein and Levinstein, Ltd. Manufacture or production of colouring matters. Sept. 14.

21,297. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of new black colouring matters derived from naphthazarine. Sept. 16.

21,316. I. Levinstein and Levinstein, Ltd. Manufacture or production of colouring matters. Sept. 17.

21,398. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. *See* Class XIII.

21,399. A. G. Green and A. R. Wahl. Improvements in the preparation of colouring matters for cotton. Sept. 17.

21,451. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of blue to green dyestuffs soluble in water. Sept. 18.

Complete Specifications Accepted.

1896.

21,197. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier. Improvements in the production of colouring matters. Sept. 8.

21,211. W. Clark. From C. H. Boehringer and Sohn. Process of preparing solutions of artificial and natural colouring matters for dyeing and printing purposes. July 7.

24,134. S. Pitt.—From L. Cassella and Co. Introduction of a blue dyestuff suitable for dyeing wool. Sept. 15.

V.—TEXTILES, COTTON, WOOL, SILK, Etc.

Applications.

18,928. F. Vanoutryve. Process and apparatus for treating cotton and other vegetable fibre, either in the woven or filamentous condition, for imparting lustre thereto. Aug. 16.

19,256. W. Baldwin. Improvements in the process of and means for sizing yarn. Aug. 20.

20,222. W. Grandage and H. Grandage. Improvements in or relating to the treatment of cotton and other yarns, and in apparatus therefor. Sept. 2.

Complete Specifications Accepted.

1896.

20,714. R. Thomas and E. Prevost. Improvements in mercerising vegetable fibrous substances. Sept. 8.

20,737. C. O'Brien and J. Shearer. Method for improving the colour of raw jute fibre. Sept. 22.

20,960. O. Junge.—From S. B. Allison. Improvements in machines for separating and cleaning the fibres of plants of every description. Sept. 1.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

Applications.

19,005. H. Seidel. Improvements in the mordanting of fibrous materials preparatory to dyeing such materials. Aug. 17.

19,260. W. Hadfield, J. J. Sumner, and H. Hadfield. Improvements in method and means for bleaching textile fabrics. Aug. 20.

19,269. J. O. Barlow. Improved chemical process for the dyeing of vegetable and animal fibres with reduced indigo. Aug. 20.

19,592. E. Laube and H. Resch. Improvements in method and apparatus for bleaching and treating textile fibres and yarns. Aug. 25.

20,210. B. Willcox.—From Badische Anilin and Soda Fabrik, Germany, Improvements in the production of discharge effects on indigo. Sept. 2.

Complete Specifications Accepted.

1896.

23,635. E. Makin, jun. Improvements in connection with kiers and like vessels for bleaching, dyeing, and similarly treating textile materials, yarns, and fabrics. Sept. 15.

23,718. H. Bentley. Improvements in apparatus for scouring and dyeing hanks of yarn or fibrous material. Sept. 15.

23,741. A. Liebmann and W. Kerr. Improvements in the methods or means for mordanting yarns, threads, fabrics, and other materials composed of fibres having a vegetable origin. Sept 15.

1897.

14,319. V. Floquet and L. Bonnet. Improvements in bleaching and disinfecting fibrous materials, and apparatus for that purpose. Sept. 1.

VII.—ACIDS, ALKALIS, and SALTS.

Applications.

19,107. F. M. Lyte, O. J. Steinhart, and W. H. Wickham. Production of ammonic phosphates. Aug. 18.

19,512. J. W. Keneval, C. A. Spofford, and J. H. Mead. Means for manufacturing carbides. Complete Specification. Aug. 24.

20,346. A. Haaz. Improvements in apparatus for making vinegar. Sept. 4.

20,567. W. Muir. A new alkaline compound, a method of forming it, and a description of some of its uses. Sept. 7.

21,073. J. Reich. Method of transforming silico fluorides and borofluorides into corresponding fluorides or fluorine compounds. Complete Specification. Sept. 14.

21,178. J. Hargreaves. Improvements in and relating to the treatment of solutions of carbonates of soda resulting from electrolysis of chloride of sodium, and in apparatus applicable for use therein. Sept. 15.

Complete Specifications Accepted.

1896.

18,468. I. Jurin. Proceeding for cleaning carbonic acid Aug. 25.

18,589. J. Hargreaves and R. Armstrong. Manufacture and treatment of detergent and bleaching compounds, and apparatus employed therein. Sept. 1.

20,423. P. Jensen.—From H. Cappelen and D. Cappelen. Improvements in pumping apparatus for chlorine gas and the like. Sept. 15.

22,512. M. Schwab. Improved method of and apparatus for concentrating sulphuric acid. Sept 15.

22,792. J. W. Chenhall. Improvements in the manufacture of bleaching compounds. Sept. 15.

23,614. J. W. Weston and T. I. Weston. Improvements in the manufacture of salt from brine, and in apparatus therefor. Sept. 8.

VIII.—GLASS, POTTERY, and ENAMELS.

Applications.

19,003. H. Brooke and J. C. Grout. Apparatus for the manufacture of glass. Complete Specification. Aug. 17.

19,303. W. Preston. Improvements in glass annealing furnaces or leers. Aug. 20.

19,493. C. C. Hartung. Making plate glass. Complete Specification. Aug. 24.

20,388. J. B. Vernay. An automatic machine for the manufacture of bottles and other articles of glass. Date applied for Feb. 5, 1897, being date of application in France. Complete Specification. Sept. 4.

20,399. W. Boulton. Improvements in or applicable to filter presses for treating potter's clay and other substances. Sept. 4.

20,414. W. Thomson. Improved glass tiles. Sept. 6.

Complete Specifications Accepted.

1896.

24,033. J. Jacques., W. E. McCalla, and J. Nicholson. Improvements in the manufacture of articles in glass, tile, pottery, metals, and like substances by a process for producing thereon letters, figures, or designs in permanent colours. Sept. 15.

21,095. J. W. Knights. Improved method of making coloured tiles for floors or building purposes. Sept. 15.

16,523. W. Cliff. Improvements in or connected with the enamelling of fire-clay baths and other articles. Sept. 1.

16,873. R. Good and R. Good, jun. Improvements in machinery for manufacturing glass vessels. Sept. 15.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Applications.

20,670. T. Holden and C. Major. Improvements in the manufacture of cement. Sept. 8.

20,960. E. M. Fox. Improvements relating to the treatment of wood and other substances for rendering the same uninflammable. Sept. 11.

21,141. J. A. Jones. Improvements in the manufacture of plaster for walls, ceilings, and the like. Sept. 15.

21,146. W. J. Pearce. Improvements in and connected with the vitreous decoration of walls, panels, and other surfaces. Sept. 15.

21,456. C. Heap and T. Oddy. Improvements in the manufacture of cement, artificial stone, preservative composition, concrete, or the like. Sept. 18.

Complete Specifications Accepted.

1896.

18,622. G. F. Thomson. Manufacture and treatment of artificial and other stone, roofing and other tiles, and enamel and apparatus therefor. Sept. 1.

23,244. L. A. Smart and H. Robertson. Improvements in cement compositions for application in steel and iron ships and otherwise. Sept. 1.

1897.

15,083. F. D. T. Lehmann and P. N. Kohlsaat. Improvements in fireproof buildings and structures. Sept. 1.

X.—METALLURGY, MINING, Etc.

Applications.

18,900. F. W. Jackson. Method of extracting gold from slimes and other deposits. Aug. 16.

19,338. J. Riley. Improvements in utilising slag or cinder from steel-melting furnaces. Aug. 21.

19,480. E. H. T. Plant and S. Dellow. Amalgamating pan. Complete Specification. Aug. 24.

19,650. Kynoch, Ltd., and A. T. Cocking. Improvements relating to blasting. Aug. 25.

19,764. A. E. Thomas. Thomas's universal convertor for cleansing molten iron from its impurities and converting the same into refined iron or malleable iron or steel, as may be required to produce. Complete Specification. Aug. 27.

19,922. E. van Toll. Manufacturing vanadium and its alloys. Aug. 30

20,312. S. O. Cowper-Coles and G. B. Pearce. Improvements in the treatment of zinc-bearing ores for the obtainment of the zinc and other metals therefrom. Sept. 3.

20,514. A. E. Tucker. Improvements in the manufacture of drawn metals. Sept. 7.

21,006. C. P. Shrewsbury and F. L. Marshall. Improvements in the treatment of pyritic ores to extract the gold and silver contained therein. Sept. 13.

21,034. W. C. Stacey.—From F. H. Keane. Roumania. Improvements in the manufacture of chilled castings. Sept. 14.

21,092. C. W. Ogden, G. Wilkins, and C. F. Morris. Improvements in and relating to the treatment of auriferous ores and deposits. Sept. 14.

21,123. T. J. Heskett and H. Jones. Improvements in the manufacture of steel. Sept. 14.

21,381. G. Attwood. Improvements in the treatment of ores. Sept. 17.

21,448. J. Baxeres and S. de la Precilla. Improvements in or relating to the extraction of gold, silver, and other metals from ores and the like. Sept. 18.

21,483. C. Wetherwax. An improved process of treating gold ores. Sept. 18.

Complete Specifications Accepted.

1896.

14,222. A. E. Tucker and T. V. Hughes. Improvements in ferro-sodium fluxes. Sept. 1.

14,223. A. E. Tucker and T. V. Hughes. Improvements in and connected with the alloying of metals. Sept. 1.

18,537. J. Ducot. Apparatus intended to determine the quantity of metal which a galvanoplastic bath should deposit on the object treated. Aug. 25.

18,760. F. O'C. Prince. Means for introducing, diffusing, and regulating the air blast to cupolas for melting metal. Sept. 1.

20,615. A. J. Boult.—From Koehler and Co. Improvements in or relating to the obtainment of metals or metallic compounds from ore residues or other bodies containing them. Sept. 22.

22,355. W. P. Thompson.—From A. Sinding-Larsen. Improved process for extracting metals. Sept. 1.

22,721. T. Storer. Improvements in the treatment of nickel ores for extraction of the nickel and the production of iron oxide pigment. Sept. 15.

22,923. W. Beardmore and H. V. Holden. Improvements in casting armour plates, and moulds therefor. Aug. 25.

22,976. D. C. Dalzell and A. Fairley. Improvements relating to the annealing of steel or iron. Sept. 8.

24,703. R. A. Hadfield. Improvements in the manufacture of iron alloys. Sept. 8.

25,461. W. P. Thompson. From La Société Civile d'Etudes au Syndicat de l'Acier Gerard. Improvements in the manufacture of steel. Sept. 22.

27,776. P. Marius. Improvements in or connected with the obtainment of metals or alloys by electrolysis or hydro-electro-chemical action. Sept. 8.

1897.

290. W. E. May. Improvements relating to the casting of metals and to apparatus for use in connection therewith. Aug. 25.

3932. M. Hinzelmann. An improved process for applying leaf metal. Sept. 22.

8887. O. Imray.—From The Pennsylvania Salt Manufacturing Co. Improvements in apparatus and appliances for casting metals. Sept. 8.

13,672. W. E. Harris. Method of treating and annealing sheet metal. Sept. 15.

15,569. T. G. Bowick. Improvements in and relating to the recovery of gold and other precious metals. Aug. 25.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

Applications.

19,035. H. S. Jones, of the firm of W. P. Thompson and Co.—From E. Balbach, jun., United States. Improvements in electrolytic apparatus. Complete Specification. Aug. 17.

19,037. J. S. Morrison. Anodes and vats for use in the electro-deposition of nickel. Aug. 17.

19,614. T. Scott and H. Hankins. Improvements in electric storage batteries or accumulators. Aug. 25.

19,638. R. Kennedy. Improvements in electrical storage batteries and plates therefor. Aug. 25.

19,846. L. Gnelzow. Manufacture of plates for electric accumulators. Complete Specification. Aug. 28.

19,959. H. Maxim. Process and apparatus for electro-thermally treating materials. Aug. 31.

20,145. A. Lehmann and A. Mann. Improvements in accumulator plates. Sept. 1.

20,428. A. E. McKechnie. Improvements in cells for batteries and connections therefor. Sept. 6.

20,486. W. E. Heys. From The Electrical Copper Company, Ltd., France. Improvements in and connected with the electro-deposition of copper and other metals on rotary cathodes. Complete Specification. Sept. 7.

20,596. A. F. Harris. Improvements in or connected with anodes for apparatus for the electro-deposition of metals. Sept. 8.

20,703. B. E. F. Rhodin. Improved metallic diaphragm for electrolysis. Sept. 9.

20,845. A. Müller. Improvements in accumulators or secondary batteries and in means for transporting the same. Sept. 10.

21,186. F. H. Perry. Improvements in primary batteries. Sept. 15.

Complete Specifications Accepted.

1896.

18,570. F. Dannert. Improvements in electric batteries. Sept. 1.

21,826. T. Froggatt. Improved grids for plates in secondary electrical batteries. Sept. 22.

23,258. B. E. Preston and C. Simkins. Improvements in primary electric batteries. Aug. 25.

25,601. J. M. Moffat. Improvements in the cells or boxes of electric batteries. Sept. 22.

26,491. J. Lelestre. Improvements in and relating to electric accumulators. Sept. 1.

28,238. T. R. Canning. Improvements in anodes for the electro-deposition of nickel. Sept. 8.

1897.

1,572. H. Leitner. Improvements in electrolytes for the deposition of zinc, and for use in secondary batteries. Aug. 25.

14,508. J. Kitsée. Improvements in primary batteries or cells. Sept. 1.

16,942. C. Bertolus. An electric furnace for the application of polyphase currents in processes of fusion or decomposition by means of the voltaic arc. Aug. 25.

17,026. C. D. Abel.—From C. N. Seoneff, Russia. A new galvanic element. Sept. 1.

17,471. C. D. Abel.—From Siemens and Halske, Germany. Improvements in electric furnaces. Sept. 1.

XII.—FATS, OILS, and SOAP.

Applications.

19,429. F. Arledter. Manufacture of resinous soap and apparatus therefor. Complete Specification. Aug. 23.

19,494. A. Cawood. Preparation or composition for scouring or washing wool, woollen textiles, or any other suitable animal or vegetable fibre. Aug. 24.

20,082. J. McKee. An improved method of and compound for treating lighting oils. Sept. 1.

20,294. W. H. Whiteway-Wilkinson. An improved lubricant. Sept. 3.

20,478. W. Ambler and J. A. Gardner. Improvements in the method of and apparatus for extracting fatty matters from soap suds and like trade refuse. Sept. 7.

20,675. W. Schuh. An improved soap and process of manufacturing the same. Sept. 8.

20,840. F. J. Lothammer. An improved process for treating petroleum and its homologues. Sept. 10.

21,115. T. C. Palmer, of the firm of W. J. Fraser and Co. Improvements in the distillation of petroleum and other oil, and in apparatus therefor. Sept. 14.

Complete Specifications Accepted.

20,438. T. Coad. Improved means and apparatus for treating fat. Sept. 15.

23,025. F. W. Wright and The United Alkali Co., Ltd. Improvements in the manufacture and production of "dry soap" or soap-powder. Sept. 1.

26,285. E. G. Scott. Improvements in the manufacture of soap. Sept. 8.

1897.

17,738. R. Gesell. Improved process for making soap. Sept. 1.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

19,030. A. H. Eyles. Apparatus for use in the manufacture of white lead. Aug. 17.

19,179. H. C. Woltereck. Process of manufacturing white lead by electrolysis. Complete Specification. Aug. 19.

19,182. F. Crane. Improvements in compounds used for the production of films, coatings, or analogous purposes. Aug. 19.

19,617. P. C. Carré. Improved material for paint and processes for manufacturing the same. Aug. 25.

19,727. R. J. Friswell and Brooke, Simpson, and Spiller, Ltd. Improvements in the obtaining of gutta percha. Aug. 26.

19,728. R. J. Friswell and Brooke, Simpson, and Spiller, Ltd. Improvements in the obtaining of gutta percha. Aug. 26.

19,729. R. J. Friswell and Brooke, Simpson, and Spiller, Ltd. Improvements in obtaining gutta percha and in the the utilisation of the bye-products arising therefrom. Aug. 26.

19,736. J. E. Bedford and C. S. Bedford. Manufacture of varnish. Complete Specification. Aug. 27.

20,303. W. Ramsay. Improvements in the purification of crude commercial gutta percha. Sept. 3.

20,531. T. Thompson. A new or improved waterproofing compound or composition. Sept. 7.

20,902. W. Walters and A. Webb. Improvements in patent leather harness enamel. Complete Specification. Sept. 11.

21,328. H. Oldnall. A new or improved composition for blacking. Sept. 17.

21,398. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Improvements in the manufacture of alizarine lumps. Sept. 17.

Complete Specifications Accepted.

1896.

22,721. T. Storer. *See* Class X.

1897.

8,811. R. Hingston and J. C. Rankin. An improved anti-fouling and anti-corrosive paint. Sept. 15.

17,936. W. Ramsay. Improvements in extracting and purifying gutta percha. Sept 8.

18,519. J. Goldbleum. Improvements in or connected with the manufacture of varnishes, paints, and the like. Sept. 15.

XIV.—TANNING, LEATHER, GLUE, and SIZE.

Applications.

18,930. C. D. Abel. — From the firm of Vereinigte Köln-Rottweiler Pulverfabriken, Germany. Manufacture of hard, dense, horny, cellulose material. August 16.

19,216. R. Condy and A. Christen.—From J. Nussbaumer, Austria. Improved process of tanning. August 19.

19,356. A. E. E. Cantley and E. Springborn. Artificial imitation leather. August 21.

19,661. H. Bolder. Improvements in liquid casein glue. Aug. 26.

19,874. E. Du Bois. Apparatus for tanning. Complete Specification. Aug. 28.

20,125. J. Mossop, jun., and E. B. Garland. An improved process for tanning hides and skins. Complete Specification. Sept. 1.

20,134. H. Sefton-Jones, of the firm of W. P. Thompson and Co. From E. Bardon, France. Improved tanning apparatus. Sept. 1.

20,273. J. Hall. Improvements in machinery for treating skins, hides, leather, and other like substances. Sept 3.

20,354. E. J. Thibaut. Improved means to be used in the tanning of hides and skins. Complete Specification. Sept 4.

Complete Specifications Accepted.

1896.

22,965. F. Billing and A. Letalle. A new material to be employed as a substitute for leather, paper, and other analogous substances, and a process of manufacturing the same. Sept. 1.

24,812. A. M. Clark.—From Les Fils de F. P. Heinrich. An improved tanning process. Sept. 8.

24,885. L. de Banville, J. T. Roulleau, and L. V. Rancelaut. The manufacture of a substitute for leather and the like, and apparatus for use therein. Sept. 22.

1897.

15,404. A. J. Boult.—From G. Brigalant, France. Improved manufacture of artificial leather or leather substitute. Sept. 22.

16,477. J. L. Garein. Improvements in or relating to the manufacture of washable leather or skins. Aug. 25.

18,930. C. D. Abel. — From the firm of Vereinigte Köln-Rottweiler Pulverfabriken, Germany. Manufacture of hard, dense, horny, cellulose material. Sept. 22.

XV.—AGRICULTURE and MANURES, Etc.

Applications.

19,033. H. S. Jones.—From J. Pioger, France. Improvements in the quick removal to a distance of town refuse and its conversion into manure. Aug. 17.

20,660. J. E. J. Johnson. New or improved manufacture or production of fertilising material. Sept. 8.

21,175. W. Walters. Improvements in the manufacture of artificial manures. Sept. 15.

Complete Specifications Accepted.

1896.

20,509. A. Schlepitzky. A new or improved artificial manure or fertiliser. Aug. 25.

1897.

17,926. W. E. Rowlands. Manufacture of manure from waste leather and phosphate of lime. Sept. 15.

XVI.—SUGARS, STARCHES, GUMS, Etc.

Applications.

19,497. E. R. Savigny. Improvements in sweetening compounds. Aug. 24.

20,152. C. J. Crossfield and S. Stein. Improvements in or connected with the manufacture of sugar. Sept. 2.

21,285. J. L. Collier. Improvements in steam pans for boiling sugar and other substances. Sept. 16.

Complete Specifications Accepted.

1896.

23,318. J. Grosse. Improvements in the granulation of crystallisable liquids and especially of the by-products of sugar manufacture. Sept. 22.

1897.

586. R. Ansorge. An improved starch preparation and process of making the same. Sept. 22.

17,144. J. Keil Improved process and apparatus for the production of starch and gluten out of cerealia and legumines. Sept. 15.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

19,161. C. O'Sullivan. Improvements in the treatment of the bye-products brewers' and distillers' yeast, or either of them, whereby a substance is obtained that can be used as a food by man, and for other purposes. Aug. 19.

19,245. A. B. Lennox. Method of and apparatus for removing impurities from alcohols and other liquids. Aug. 20.

19,759. A. Schulze. Brewing beer without the husks of the barley or malt. Aug. 27.

19,858. A. Collette, jun. Process for the extraction of alcohol by saccharification and fermentation by mucedineæ, and apparatus therefor. Complete Specification. Aug. 28.

20,103. T. Hyatt. Improvements in malt liquors and process for manufacturing the same. Sept. 1.

20,107. R. S. Kaufmann. Plant malt for ale and beer brewery purposes instead and to replace partly the barley. Sept. 1.

20,218. J. Weichmann. *See* Class I.

20,671. T. Skurray. Improved process for the production of fermented liquors and ferments for use in the same. Sept. 8.

20,955. J. E. J. Johnson. Improved treatment of alcoholic or fermented liquids. Sept. 11.

Complete Specifications Accepted.

1896.

25,852. J. Schneible. Improvements in the manufacture of fermented liquors. Sept. 22.

1897.

18,112. H. H. Lake.—From L. S. Langville and H. Tauszky, United States. Improvements in and relating to the manufacture of yeast. Sept. 22.

XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

Applications.

A.—*Foods.*

19,161. C. O'Sullivan. *See* Class XVII.

19,215. W. Majert. Manufacture or treatment of butter for the removal of bacilli and germs. Aug. 19.

19,357. H. Higgins. Proteid breads, biscuits, and cakes. Aug. 21.

19,446. H. Grono and H. Pateson. Process of chilling and freezing meat and other alimentary substances, and means therefor. Complete Specification. Aug. 25.

19,979. C. Estcourt. Production of a valuable food for animals. Aug. 31.

19,980. C. Estcourt. Production of a new food for human consumption. Aug. 31.

20,013. E. G. N. Salenius. Method and apparatus for sterilising milk and other liquids. Complete Specification. Aug. 31.

20,527. A. W. Rothwell.—From T. T. Rothwell, Victoria. Elimination of tannin from tea in infusion. Sept. 7.

20,898. W. G. Coventry. Improvements in the application of chemical preservatives to meat. Sept. 11.

21,387. F. W. H. Graeff. Preservation of organic substances. Complete Specification. Sept. 17.

Complete Specifications Accepted.

1896.

23,785. W. Luplan. Improved apparatus for use in the aëration of milk, and for attachment to milk cans and the like receptacles. Sept. 22.

1897.

16,088. W. Horlick. Improvements in edible and soluble tablets, and method of producing the same. Sept. 15.

Applications.

B.—Sanitation.

19,284. W. H. Bowers. Retorts for the drying or carbonisation of sewage matter and generally of vegetable or other substances. Aug. 20.

19,426. J. Desmaroux. Apparatus for sterilising water. Complete Specification. Aug. 23.

19,747. W. Noton. Method of and means for purifying and filtering sewage and other foul and impure water. Aug. 27.

20,832. S. Platt. Improvements in apparatus for treating sewage and other waste liquors. Sept. 10.

21,402. L. Devaux. System of purification of water. Sept. 17.

C.—Disinfectants.

19,204. H. E. Newton. — From The Farbenfabriken vormals Friedrich Bayer and Co., Germany. *See* Class XX.

Complete Specifications Accepted.

1897.

17,464. W. Loebinger. Improvements in or relating to disinfection by means of formaldehyde. Sept. 1.

18,250. H. Oppermann. Process for the preparation of easily soluble compounds of formic aldehyde for preserving, disinfecting, and therapeutic purposes. Sept. 22.

XIX.—PAPER, PASTEBOARD, Etc.

Application.

21,193. C. Beadle. Treatment of waste paper or the like for removal of sizing and gumming ingredients. Sept. 15.

Complete Specifications Accepted.

1896.

21,978. A. Masson. Improved method and means of drying and treating coated paper. Sept. 22.

22,965. F. Billing and A. Letalle. *See* Class XIV.

24,010. D. C. Simpson. A new or improved composition for treating paper, cardboard, wood, or other material of a similar nature, to render such material waterproof, and the markings with writing inks thereon indelible. Sept. 22.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

Applications.

19,014. A. J. Boult.—From E. Vial, Belgium. Improvements in the extraction of suintate (suintine). Aug. 17.

19,204. H. E. Newton. — From The Farbenfabriken vormals Friedrich Bayer and Co., Germany. Process for producing aromatic aldehydes. Aug. 19.

19,809. E. C. C. Stanford. Improvements in pharmaceutical preparations. Aug. 28.

Complete Specifications Accepted.

1896.

21,031. J. G. McIntosh. Improvements in the manufacture and application of terebenthene mono hydrochloride. Sept. 22.

1897.

8387. H. Mack. Method of producing highly concentrated fat solutions of fragrant substances. Sept. 1.

9827. F. Valentiner. A process for the production of aromatic fluoro hydrocarbons in aqueous solution. Sept. 8.

17,409. O. Imray. — From The Society of Chemical Industry in Basle, Switzerland. Manufacture of new therapeutical compounds. Sept. 8.

17,692. L. Lederer. Improvements in the process for preparing the halogen derivatives of acetone. Sept. 22.

XXI.—PHOTOGRAPHY.

Applications.

18,973. E. J. Browne and F. J. Abbott, jun. Process for reproducing photographs in natural colours. Aug. 17.

19,168. C. T. Jago. Method of obtaining permanent coloured photographs. Aug. 19.

19,208. A. E. Wade. Photographic solutions as used for toning and fixing prints. Aug. 19.

29,975. H. Soar. Improved means of preparing photo mechanical surfaces analogous to collotype and the like. Sept. 13.

Complete Specification Accepted.

1896.

19,726. W. Grunow. A new or improved apparatus for developing, fixing, and toning photographs. Sept. 15.

XXII.—EXPLOSIVES, MATCHES, Etc.

Applications.

18,929. C. D. Abel.—From the Firm of Gesellschaft für Lindes Eismaschinen, Germany. New explosive compound. Aug. 16.

19,259. L. Davies. Explosive cartridges. Aug. 20.

19,418. S. Neuburg and I. Neuburg. Manufacture of matches. Aug. 23.

20,090. A. E. Blondel. Improvements in fog signals. Sept. 1.

20,223. E. S. Clark. Improvements in or relating to safety tamping for explosives used for blasting operations in coal and other mines, and in apparatus for use in connection therewith. Sept. 2.

20,763. W. Hope. Improvements in sporting and other ammunition. Sept. 9.

Complete Specifications Accepted.

1896.

22,540. R. W. Strehlenert. A new or improved solvent or solvents for nitrocellulose whereby improved solutions are obtained. Sept. 15.

1897.

18,563. J. Caveri. Improvements in the manufacture of matches. Sept. 22.

PATENT UNCLASSIFIABLE.

Application.

20,775. F. J. Lothammer. Improvements in the treatment of asbestos. Sept. 9.

Printed and Published by Eyre and Spottiswoode, East Harding Street, London, E.C., for the Society of Chemical Industry

THE JOURNAL

OF THE

Society of Chemical Industry.

A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

No. 10.—Vol. XVI.] **OCTOBER 30, 1897.** [Non-Members 30/- per annum; Members 21/- per Set of extra or back numbers; Single Copies (Members only) 2/6.

NOTICES.

Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, part of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows:—

To Members (see Rules 25 and 27) and Past Members (see Rule 30); Libraries, Corporations, and Exchanges on the Society's List	Each copy 10s.
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Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed. The circulation of the Journal is now more than 3,000 per month.

Foreign and Colonial Members are reminded that the subscription of 25s. for 1898, payable on January 1st next, should be sent in good time to the Treasurer, in order to ensure continuity in the receipt of the Society's Journal. Any changes of address to appear in the new List of Members now in course of preparation, should reach the General Secretary not later than January 15th, 1898.

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May, Geo. H., Hohokus P.O., Bergen Co., N.J., U.S.A., Assistant Chemist (American Pegamoid Co.).

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Peter, Dr. A. H., 205, Third Avenue, New York, U.S.A., Chemist.

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Wardle, Bernard, Leek, Staffordshire, Silk and Cotton Printer.

Wense, Dr. Wilhelm, Westeregeln, Prov Sachsen, Germany.

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MEMBER OMITTED FROM LIST.

Aikman, C. M., 128, Wellington Street, Glasgow, Analytical Chemist.

Death.

Jackson, Thos., Beechmount, Clayton, Manchester. October 3rd.

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SESSION 1897–98.

Nov. 1st:—

W. Harry Stanger and Bertram Blount. "The Adulteration of Portland Cement."

Dr. W. P. Evans. "An Improved Adjustable Drip-Proof Bunsen."

Dec. 6th.—H. L. Sulman and Dr. F. L. Teed. "The Sulman-Teed Process of Gold Extraction."

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F. Hurter.	

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SESSION 1897–98.

Oct. 27th.—Chairman's Address. "The Manufacture and Properties of Carborundum."

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E. Knecht.	D. Watson.
I. Levinstein.	

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Bank House, The Cliff, Higher Broughton, Manchester.

SESSION 1897–98.

Nov. 5th.—Chairman's Address. "Recent Developments in the Chlorine Industry."

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Vice-Chairman: G. T. France.

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H. Forth.	

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SESSION 1897–98.

Oct. 27th.—F. J. R. Carulla. "Nitrate of Soda in Iron Metallurgy."

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Vice-Chairman: Christopher Rawson.

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J. Cohen.	H. R. Procter.
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Hon. Secretary and Treasurer:

Thomas Gray, Technical College, 204, George Street, Glasgow.

SESSION 1897–98.

A special meeting for the Oil Industry will be held at Broxburn on Monday, Nov. 29th, subject:—"Recent Improvements in Retorting."

Any members who desire to contribute to the proceedings of this meeting should at once communicate with the Hon. Local Secretary as above, from whom all particulars may be obtained.

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Vice-Chairman: T. J. Parker.
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Hon. Treasurer: R. C. Woodcock.
Hon. Local Secretary:
Dr. H. Schweitzer, 77, William Street, New York, U.S.A.

SESSION 1897-98.

The Session was opened on October 22nd by an address from the Chairman. Five papers were also read.

Meetings will be held on the third Friday after the first Monday in every month. The meeting on Nov. 19th will be held at Columbia College, on Washington Heights.

Obituary.

VICTOR MEYER.

Geheimrath and Professor of Chemistry in Heidelberg University.

Chemical science has lost one of her most distinguished men, and Germany one of her most enthusiastic teachers, by the sudden death of Victor Meyer. In that loss it may with truth be said that all the civilised world participates, for students came from far and near to enjoy the advantages of his direction of their studies in the laboratory, and of listening to his masterly expositions in the lecture room.

Victor Meyer was born on Sept. 8, 1848, in Berlin. He was the son of Jacques Meyer, a cotton manufacturer of that city, well known for his large-hearted benevolence. As a youth of 16 years of age he had passed through the Werder Gymnasium, and been entered as a student of Berlin University. After one semester's study here, he went to Heidelberg to study under Bunsen, and later on, having obtained his degree of Doctor of Philosophy with the highest distinction, he became Bunsen's assistant. After considerable promotion in Heidelberg, he went to continue his studies under A. von Baeyer in Berlin, and from thence he went, in 1871, to Stuttgart Polytechnikum to be the first assistant of Fehling, and also as Professor of Organic and Theoretical Chemistry. A year later he obtained the post of Professor of Chemistry in the Polytechnikum of Zürich in place of Wislicenus, and this post he filled with brilliant success for the space of 12 years. In 1885, he went to Göttingen, and in 1889 to Heidelberg as successor to Bunsen. Bunsen had proposed him as "the most gifted of my pupils." In 1890, it became necessary to commence the work of erecting new laboratories, and in 1892 the new building was completed, and very soon afterwards it was found that the accommodation still failed to suffice for the increased and ever-increasing influx of students. In the last year or so, on the average, 50 to 60 applications for places in the laboratory per semester, had to be refused for sheer lack of space.

The reason for this is easily given. Victor Meyer worked not in one department of his science, but in all departments, and in each he had won distinction. We owe to him the discovery of the aliphatic nitro-compounds. He enriched the branch of chemical physics with numerous improved methods and instruments. The older methods of vapour-density determination, so troublesome in detail, he materially simplified, and introduced his now well-known method by displacement of air, for the determination of the vapour densities of substances boiling or vaporising at exceedingly high temperatures.

Victor Meyer was the discoverer of the aldoximes and ketoximes—two new classes of substances important for the characterising of the aldehydes and ketones. But his discovery of greatest importance to technologists was that of Thiophen and its homologues. At the time great surprise was caused by the fact that a substance so much investigated as the so-called "pure benzol" should still be found to contain an extraneous body—a sulphur compound, too. Having isolated this substance in a state of purity, Victor Meyer and his pupils worked upon it and its derivatives, and at length published a book devoted to their researches thereon, and entitled "Die Thiophengruppe" (Vieweg. Brunswick, 1888).

Victor Meyer was, in 1882, elected a corresponding member of the Bavarian Academy of Sciences, and in 1883 a foreign member of the Chemical Society of London.—W. S.

Journal and Patent* Literature.

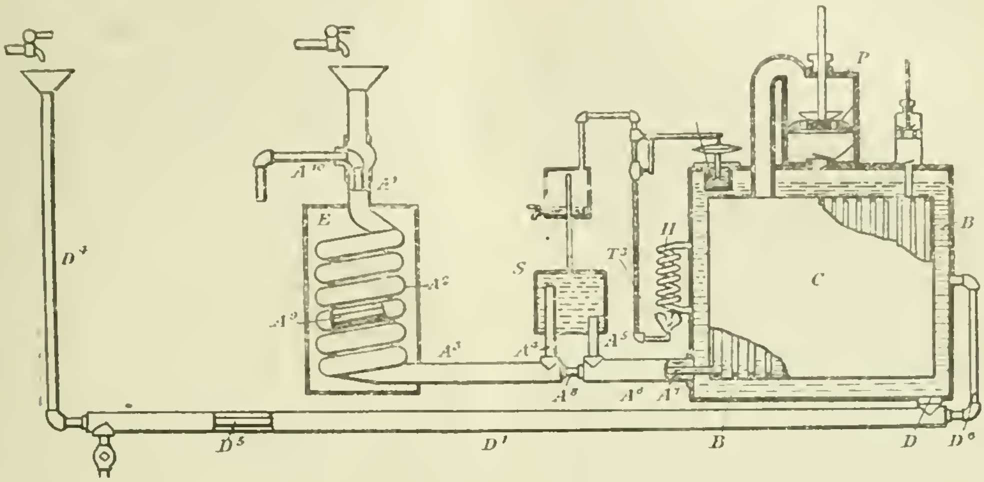
I.—PLANT, APPARATUS, AND MACHINERY.

Heating Fluids and Distilling [*Water, &c.*], *Impts. in or relating to.* A. J. Boult, 111, Hatton Garden, London. From A. G. Waterhouse, Hartford, Connecticut, U.S.A. Eng. Pat. 16,035, July 6, 1897.

The invention, when applied to the process of distilling liquids, consists of a method of and means for transferring the heat given off by the condensation of the steam or vapours, and by the condensate, to the liquid to be distilled while on its way to the still. When used merely for the purpose of heating liquids, the heat is transferred from the liquid passing from the heating apparatus to that passing to the same. The figure illustrates one typical form of the apparatus employed, several modifications, including "multiple-effect" arrangements, being also described in the specification. The liquid is fed through the pipe A^1, coil A^2 (which encloses the return coil A^9), pipes A^3, A^4, heating tank S, and pipes A^5, A^6 to the still B, the condensed steam or vapours from the still being returned through the chamber C, pipes A^7, A^8, coil A^9, and pipe A^{10}.

* Any of these specifications may be obtained by post by remitting 8*d.*—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

In starting, the still B is heated by the heater H, which may be heated by the burner T^3, fed by gas evolved from the liquid in the tank S, but, afterwards, heat is supplied only so far as is necessary to replace that lost by radiation, &c. In the apparatus shown, the process is supplemented and maintained by the pump P, which withdraws the steam or vapour from the still B and forces it back through the chamber C, the steam or vapour being condensed at a higher



temperature than that at which it was formed. When the liquid in the still B deposits a sediment, &c., it is drawn off, at intervals, through the pipes D, D^1, the heat being abstracted from this liquor by feed-liquor supplied through the pipes D^4, D^5, D^6. The "heat exchange," A^2, A^3, is enclosed in a casing E, this and other parts of the apparatus being coated with non-conducting material. When required for heating purposes only, the still B is omitted. In a modification, the pump P is worked by an electromotor, and the heater H is replaced by an electric heater. The current through the heater is controlled by an adjustable resistance, which is automatically regulated by the pressure within the still. "The method is claimed, of distilling liquids by transferring the heat given off by the condensation of steam or vapour, to the liquid to be distilled, and transferring the heat from the condensed liquid to liquid to be distilled while on its way to the still." The apparatus is also claimed.—R. A.

II.—FUEL, GAS, AND LIGHT.

Illuminating Petroleums used in Italy. A. Volpi and R. Ruggeri. Annali del Laborat. Chim. Centr. delle Gabelle, III., 119—137.

This paper contains an account of the examination of 39 samples of American and Russian petroleums obtained from various cities in Italy. The question of the flashing points adopted by various countries is discussed, and allusion is made to the recent writings of Lobry de Bruyn, Kissling, Dennstedt, and others. In Italy the limit fixed by law until 1895 was 35° C. (Abel); since then it has been lowered by the High Council of Health to 21° C. (Abel). The conclusions drawn from the samples examined are as follows:—(i.) All samples except one had a flashing point below 35° C. (Abel)—the former standard, but above 21° (Abel)—the present standard; (ii.) petroleums declared to be of Russian origin had generally a higher flashing point than those of American origin; (iii.) one-fourth of the samples, on distillation at 310°, gave a residue above 10 per cent.; (iv.) the illuminating power varied from 4·47 to 10·22 candles (German Union standard), and the consumption from 2·6 to 5 grms. per candle-hour. As to the former, three-fourths of the samples, and as to the latter, one fourth, should be regarded as of inferior quality.—V. H. V.

Acetylene for Lighting Purposes, Application of. L. M. Bullier. Bull. Soc. Chim. 1897, 646—654.

The author has carried out a series of experiments with a view of testing various types of burners for acetylene, either pure or in admixture with other gases, such as nitrogen, carbon mon- and dioxide, hydrogen, illuminating gas, and air, and mixtures of these gases. He arrives at the following conclusions:—The amount of pure acetylene consumed per Carcel (1 Carcel = 9·6 sperm candles), in the case of special burners or in those used for oil gas, is 7—8 litres, and, as in the case of other illuminants, the intensity increases proportionately with the amount. Mixtures give the same yield of luminosity as pure acetylene, providing that they contain at least 50 per cent. of the acetylene gas. For the same intensity, the consumption of the burners diminishes proportionately as the amount of acetylene increases in the mixture burnt. Mixtures containing a large proportion of carbonic acid are of no advantage, whilst mixtures of acetylene with pure nitrogen show diminution of at least 10 per cent. of that of acetylene alone. The addition of a small proportion of oxygen to either of these two mixtures is of considerable advantage, whilst hydrogen containing relatively small proportions of acetylene gives good results. It is thus possible to employ mixtures of acetylene and water-gas, or of nitrogen and acetylene containing very small quantities of oxygen (air). With regard to the former, it possesses the grave disadvantage of containing a not inconsiderable proportion of carbonic oxide, whereas the latter, since it only contains a quantity of oxygen below that required by theory, will not have any explosive properties.—T. A. L.

Solutions of Acetylene, and their Explosive Properties. Berthelot and Vieille. Comptes Rend. 1897, **124**, [19], 988—996.

A study, under three heads, of the properties of solutions of acetylene in acetone, which have recently been brought before the public as a means of reducing the risk attending the use of acetylene as an illuminating agent.

I. *Pressure of Dissolved Acetylene.*—Details are given of the pressures, over a range of about 60° C., of solutions containing respectively 69 grms. of acetylene and 301 grms. of acetone, 118 grms. of acetylene and 315 grms. of acetone, and 203 grms. of acetylene and 315 grms. of acetone. The most important results are that the observed pressures follow the same general law as the pressures of the saturated vapour of a homogeneous liquid, and are attributable almost entirely to the acetylene; the pressure attributable to the acetone forms only a small fraction (a few hundredths) of the total pressure.

II. *Liability of Dissolved Acetylene to Detonate.*—The explosion of 1·5 grm. of fulminate in a metallic bottle of 700 c.c. capacity, containing 320 grms. of acetone and 132 grms. of acetylene, caused a dull noise, with an escape of gas. The bottle was cracked, but there was no explosion nor inflammation. In an identical experiment made by the

authors with liquefied acetylene, the bottle was smashed to small fragments.

III. *Liability of Dissolved Acetylene (and of the Vapour) to Inflame.*—A 50 c.c. steel tube, provided with crusher manometers, was charged with 14 c.c. of acetone in a first set of experiments and 8·2 c.c. in a second set. The acetone was saturated with acetylene at the ordinary temperature, and at pressures varying from 10 to 20 kilos. per sq. cm. When the initial pressure did not exceed 10 kilos., and the inflammation was produced by an incandescent platinum wire in the gas, the observed pressures did not differ from those corresponding to the combustion of pure acetylene under the same pressure. From this it may be concluded that the dissolved acetylene is not decomposed, and that it takes no part in the combustion. The maximum pressures observed are only one-tenth of those which would correspond to the explosive decomposition of the whole of the contained acetylene (gaseous and dissolved). But when the initial pressure exceeds 10 kilos., the effect produced becomes analogous to the explosion of pure liquid acetylene. Not only is the acetylene decomposed; the acetone which holds it in solution is also destroyed simultaneously. The explosive decomposition produces a compact mass of carbon, which takes the form of the tube. The gases formed consist of hydrogen and carbon monoxide, mixed with carbonic acid. The experiments have been repeated with a larger receiver (13·5 litres) of the kind commercially used. It is found that the bottles ordinarily used (tested to 250 atmospheres) can support without rupture the pressure resulting from an accidental inflammation of the gaseous atmosphere contained in the bottles (the acetone having been saturated with acetylene at pressures of 6 to 8 kilos. and temperatures of 10°—15° C.). The pressure developed does not exceed 155 kilos. But these bottles are no longer safe if the initial pressure exceed 10 kilos., or if they are exposed to temperatures exceeding 35° C. With an initial pressure of 20 kilos., when the inflammation is produced in the gaseous atmosphere, it can develop a pressure of 568 kilos., and when it is produced in the liquid itself, the pressure rises to 5,100 kilos. No commercial receiver is safe under such conditions.

Finally, acetylene is less dangerous when dissolved in acetone, for it cannot be exploded by internal inflammation when the pressure is below 10 kilos. and the temperature below 15° C. Acetylene *per se* can be exploded by internal inflammation when a receiver of a litre capacity contains 2·5 grms. or more of it. Under the conditions stated the same receiver could contain without risk nearly 50 times as much acetylene (100—120 grms.) dissolved in acetone.

—D. E. J.

Solutions of Acetylene, Remarks on their Explosive Decomposition. Berthelot and Vieille. Comptes Rend. 1897, **124**, [19], 996—1000.

When acetylene is heated in contact with its solution in acetone (in a closed receiver), under certain conditions the dissolved acetylene suffers no decomposition, whilst under other conditions it suffers explosive decomposition. In the latter case the solvent (acetone) is decomposed, and in the main decomposed into its elements, carbon and hydrogen; the oxygen appears in the form of carbon monoxide and dioxide and water—

$$2C_3H_6O = \begin{cases} 5C + 12H + CO_2 \\ 5C + 10H + CO + H_2O. \end{cases}$$

This total decomposition of the solvent is produced by the explosive shock resulting from the destruction of the acetylene at constant volume. It is exceptionally interesting as an example of the sudden and total destruction of a substance which is formed (as acetone is) with evolution of heat. The determining factor in the conditions, is the pressure. The authors proceed to explain, from thermochemical considerations, why acetylene dissolved in acetone is stable up to a certain pressure (about 10 kilos. per sq. cm.). The decomposition of 26 grms. of gaseous acetylene into its elements, evolves + 51·4 calories; allowing for the heat of solution of dissolved acetylene, this would be reduced to + 46·3 calories. The heat of vaporisation of a molecule of acetone is 7·5 cal., so that the decomposition of a molecule of acetylene would suffice to vaporise 6 mols. (or 13 times its weight) of acetone. Such would be the effect produced in a solution containing 77 grms. of acetylene per kilo. of acetone. To this must be added the heat required to heat to the same temperature the carbon and hydrogen produced by the decomposition of the acetylene. Clearly, the high temperature required for the total destruction of the acetylene cannot be attained under these conditions: a much larger percentage of the endothermic compound is required. At a pressure of 10 kilos. per sq. cm., 1 kilo. of acetone dissolves 350 grms. of acetylene; the latter by its own decomposition would evolve 623·3 cal. This, at constant volume, would raise the mixture of acetone (supposed unaltered), carbon, and hydrogen to 730° at most. Now, this temperature is not high enough to decompose acetylene into its elements. A similar calculation for acetone saturated with acetylene under a pressure of 20 kilos. (and containing 700 grms. of acetylene per kilo. of acetone) gives a temperature of 1,300°, which is about the actual temperature of decomposition of acetylene. The above calculations are only approximate, and reference must be made to the original for further developments; *e.g.*, the heat absorbed in the decomposition of the acetone into carbon, hydrogen, and carbonic acid would reduce the above temperature to 1,160° C. But this same decomposition would determine an increase of volume from 1 to $3\frac{1}{2}$; or, at constant volume, a corresponding increase of pressure. Taking both into account, the decomposition of the acetone would result in a doubling of the final pressure, as compared with that due to the decomposition of the acetylene alone. Under high initial pressures the acetone may become a source of danger rather than of safety.—D. E. J.

Acetylene, Conditions of the Propagation of the Decomposition of. Berthelot and Vieille. Comptes Rend. 1897, **124**, [19], 1000.

The authors have already shown that, under the normal pressure, a decomposition excited at any point in an atmosphere of pure acetylene gas does not spread throughout the mass; but that at higher pressures (about double the normal) the gas exhibits the properties of an explosive mixture. At a given pressure the tendency to propagation is affected by other conditions (mode of excitation and external cooling effects). They have now studied the intermediate state and conditions (under which an explosion sometimes occurs, at other times not) using various methods of exciting decomposition, both in large receivers (4 to 25 litres) and in metallic tubes 3 m. long and 22 mm. in diameter. The results (details of which are given) show that for a given mode of excitation it is not possible to specify an absolutely fixed critical pressure, such that below it propagation would be impossible, whereas above it propagation would be certain. The change takes place gradually, following a scale of pressures to which correspond increasing probabilities of explosion. This behaviour is not peculiar to acetylene. For all explosive substances the phenomena of propagation present the same character. The conditions of certain explosion are always separated by a large interval, from those of assured safety. Between these there exist zones of danger, and it is impossible to define anything beyond the probability of an explosion occurring.—D. E. J.

Acetylene: Solution in Acetone. C. H. Bothamley. Photography, Sept. 1897. (See this Journal, 1896, 793; 1897, 318, 319, and 524, and the foregoing three abstracts.)

The practical points that the author deduces from the foregoing researches of Berthelot and Vieille, are summed up in the following conclusions for those who use acetylene generators:—(1) Avoid the use of copper or brass in the construction of the apparatus, whether for generating or storing, and (2) take care that the pressure in the apparatus as a whole, or in any part of it, never exceeds half an atmosphere above the ordinary atmospheric pressure. If these points are strictly attended to, the danger of any explosion of the acetylene is very small, provided, of course, that it does not become mixed with air.

Incandescent Gas Light, The Theory of. C. Hohmann. J. für Gasbeleucht. 1897, **40**, 456.

Killing (this Journal, 1896, 794) has advanced the theory that mantles composed of thoria with a small proportion of ceria, platinum, &c., owe their light-emissive power to the ceria, &c., acting catalytically as oxygen-carriers, which present oxygen to the gas flame in the nascent state, thus producing a higher temperature in the region of the mantle. Geelmuyden found that ordinary slit- and Argand burners gave no imperfectly burnt gaseous products, whereas Welsbach burners frequently yielded small quantities of these; and Krebs has utilised these results in attacking Killing's theory, which he considers to imply that a more perfect combustion should take place in a Welsbach burner than in an ordinary gas flame.

The author points out that with burners having badly arranged air-inlets, imperfectly burnt gas may readily escape, but that Bunte has found that the gaseous products from Welsbach burners, acting under normal conditions, only contain mere traces of carbon monoxide; this he also found was the case with an ordinary slit-burner. Krebs' experimental data are thus controverted. But it does not follow that the emission of incompletely burnt gas would be evidence against Killing's theory. The oxygen-carrying action of the ceria does not necessarily lead to a more complete combustion, but merely to an increased *velocity* of reaction at the point of contact. It might even be that the greatly increased temperature would be a condition unfavourable to the complete combustion of the rest of the gas.—H. B.

Acetylene, Method of Estimating, Applicable to Acetylides of the Formula R.C ⋮ C.H. Chavastelon. Comptes Rend. **125**, [4], 245.

See under XXIII., *page* 828.

PATENTS.

Peat, A Process for Carbonisation of, by Electric Heating. P. Jebsen, Bruvik, Norway. Eng. Pat. 22,194, Oct. 6, 1896. (Date claimed under Internat. Conv., March 13, 1896.)

Peat is carbonised in retorts lined with asbestos, &c., against which are coiled iron wires for carrying the current. Other iron wires may be coiled around an insulating covering fixed on a central hollow stem. The drawings attached to the specification show three retorts carried upon central bearings, so that the peat and charcoal may be charged into and discharged from the retorts through the same lid, the retort being turned, with the end covered by the lid, upwards or downwards as required.—R. S.

Peat and its Products, Impts. in or relating to the Treatment or Utilisation of. W. W. Hughes, Queen Victoria Street, and S. Wilison, Wembley, Middlesex. Eng. Pat. 20,084, Sept. 10, 1896.

Calcium carbide is prepared from peat by carbonising the latter in a vertical retort, in which it is supported upon inclined partitions, which may be slid out so as to allow it to pass gradually downwards to the bottom and hottest part of the furnace. The carbonised peat is then pulverised, mixed with finely divided lime, and then preliminarily heated, and afterwards intensely heated in an electric furnace to form the carbide.—R. S.

Charring Peat, Wood, or the like: Impts. relating to the Method of, and to Apparatus therefor. S. G. Laurenius, New York. Eng. Pat. 11,851, May 12, 1897.

The method consists of discharging the heavier gases formed during the carbonisation, from the lower part of the retort, and of introducing steam or water into the retort when the charring should be stopped or the temperature reduced. The former is effected by one or more openings covered by a hood, in the bottom of the retort, through which the gases enter a small chamber, provided above with two valves—a lighter one leading to an outlet pipe having a valve, and a heavier one, through which the products pass when the valve on the first is closed, leading to the furnace, where they are consumed.—R. S.

Smokeless Inflammable Wick, Process for Manufacturing. A. H. Freise, Hamme-Bothum, Prussia. Eng. Pat. 12,021, May 14, 1897.

The wick is formed of a loose coarse felt or similar material, or of paper or paper pulp, and is covered with three coatings, namely: (1) of inflammable paste, (2) of powdered sulphur with the addition of a compound of oxygen, (3) of powdered sulphur mixed with stearine, paraffin, or the like. The layers are fixed to the wick with suitable adhesive substances, as glue, gum, albumins, or resins, or by adding to the pulpy material when such is used to form the wick, or (when the coating contains sulphur) by means of hot rollers.—R. S.

Liquid Fuel Burners, Impts. in. La Société des Générateurs à Vaporisation Instantanée Système. L. Serpollet and L. Serpollet, Paris. Eng. Pat. 12,438, May 19, 1897.

Petroleum or other liquid hydrocarbon is forced, in larger or smaller quantities, by pressure from a compressed air reservoir, into a burner having a central body serving as a boiler, wherein the hydrocarbon is vaporised, and from which it passes as vapour to a set of nozzles directed upwards, downwards, or horizontally, and arranged in the axes of Bunsen passages, into which the jet draws the surrounding air. The air and vapour, heated by contact with and radiation from the walls, are ignited, and the jets impinge against plates of refractory material, which deflect them. The preliminary heating of the petroleum for starting the apparatus may be effected in various ways.—R. S.

Electrical Furnaces, Impts. in. [*Wedge-shaped Electrodes.*] C. D. Abel, London. From Siemens and Halske, Berlin, Germany. Eng. Pat. 17,471, July 24, 1897.

To ensure an automatic downward feed of the material to be treated, the upper electrode in this electric furnace is wedge-shaped or conical while the lower is hollow or funnel-shaped. The cylindrical portion of the upper electrode is surrounded with a casing and provided with flanges, which the regulating motion of the arc causes to loosen the material, and counteracts the tendency to cake, while the gas generated, escapes through the interspace between the casing and the electrode. The pulverulent material to be treated is formed into lumps or briquettes with some suitable binding substance so as to facilitate the escape of the gases, and provision is made for withdrawing the product during working, by means of a hole in the bottom of the funnel, below which, at a suitable distance, is placed a laterally extending closing-piece, or by a tapping device consisting of a lateral opening in the lower part of the funnel, opposite which is placed a boring tool for periodically making a hole in the crust formed, and withdrawing the fluid material.—G. H. R.

Illuminating Gas, Impts. in the Manufacture of. W. Young, Peebles; S. Glover, St. Helen's; and T. Glover, West Bromwich. Eng. Pat. 20,125, Sept. 11, 1896.

To remove or prevent the deposition in the solid form of the naphthalene present in large quantity in gas resulting from the carbonisation of coal at high temperatures, the inventors propose to wash the gas with liquid hydrocarbons capable of dissolving out the naphthalene, and having at the same time a sufficiently high vapour-tension to prevent the absorption of the illuminating constituents. The illuminating power of the gas may be increased by carburetting with the products obtained by carbonising or cracking cannel coal, shale, blast-furnace petroleum, paraffin oils, &c. at low temperatures, in such a manner that the said products are nearly free from naphthalene, and are consequently able to dissolve this substance, whilst they contain volatile hydrocarbons, which they yield up to the gas under treatment.

The operation of carburetting is performed after the gas has been cooled and freed from tarry matters and naphthalene as far as possible, by condensation; and the naphthalene is separated from solution by cooling, the solvents being afterwards used over again—after cracking, once more if they contain a sufficiency of paraffins and olefines to yield a further supply of volatile hydrocarbons.

Another method proposed is the use of the vapours of hydrocarbons capable of dissolving naphthalene, and having the same vapour-tension as the latter, so that if the naphthalene be deposited, as a result of cooling, the said hydrocarbons will be simultaneously liquefied, and carry it into solution.—C. S.

Incandescent Lamps, Impts. in Hydrocarbon. R. Haddon, London. From J. W. Allison, New York. Eng. Pat. 3447, Feb. 9, 1897.

The oil is fed to a vertical vaporising tube, situated close to the burner or burners, and conveying the vapours into an upper vertical tube leading to the mixing chamber, air being drawn in at the point of junction between the two tubes, through a transverse tube mounted in a bell-shaped hood below the mixing chamber, against which it directs the hot air and gases from the burners. From the mixing chamber, pipes convey the vapour and air down to the burners. The vaporising tube is surmounted by a cap forming a needle valve, the needle of which is enclosed, for protection, in a small tube, and passes down through a stuffing box at the lower extremity of the vaporising tube. The valve is closed and opened by moving a slotted sleeve engaging with a lug on the stuffing box, and thus regulates the supply of oil vapour to the mixing chamber.

To start the lamp, a cup situated on the base of the burner and surrounding the vaporising tube, is filled with oil or alcohol, which, when lighted, heats the said tube and generates sufficient vapour to supply the burners at the outset, the heat from the latter afterwards sufficing to maintain the supply.—C. S.

Calcium Carbide and the like, Impts. in the Manufacture of, and in the Method of using the same in Lamps or Generators. F. H. Smith, Dunblane, N.B. Eng. Pat. 13,103, May 27, 1897.

The calcium carbide or other compound is powdered and compressed to form tablets, or is mixed with oil or the like to the consistency of dough, and is then formed into pills, tablets, &c. The tablets, &c. are coated with wax, varnish, or other waterproof substance. In use, they are mechanically fed from an upright tube, and means are provided for abrading the varnish at one place on each tablet, so as to allow the water to act thereon.—R. S.

Gas Generators [Acetylene], Impts. in. C. H. Campbell, Philadelphia, U.S.A. Eng. Pat. 12,120, May 15, 1897.

The supply of calcium carbide to the generator is automatically regulated by the movement of the gasometer, by means of a rack mounted on the latter and actuating a pinion which, by intermediate sprocket gearing, works a spiral conveyor in the bottom of the carbide chamber. This motion is produced only during the descent of the gasometer cover, a ratchet and pawl on one of the sprocket wheels preventing a reversed movement during the ascent of the cover. A valve in the neck of the generator is so attached, by a lever and swinging yoke, to the cover of the carbide chamber, that when the latter is opened, the valve closes and prevents the escape of gas.—C. S.

Acetylene Gas, Impts. in Apparatus for Generating and Storing. E. Chesnay and L. Pillion, both of Dijon, France. Eng. Pat. 20,090, Sept. 10, 1896. (Under Internat. Convention.)

Improvements in the apparatus claimed under Eng. Pat. 18,992 of 1896 (this Journal, 1897, 665) are described. They comprise a composite support for the carbide, a cylindrical bell over the container, and a layer of oil above the water in the generator.—J. A. B.

Acetylene Gas, Impts. in Apparatus for Generating and Storing. E. Chesnay and L. Pillion, both of Dijon, France. Eng. Pat. 20,254, Sept. 12, 1896. (Under Internat. Convention.)

This is supplementary to Eng. Pats. 18,992 and 20,090 of 1896 (see foregoing). The supply of water to the carbide is controlled by the displacement caused by a float attached to the gasholder, or by an oscillatory movement of the apparatus.—J. A. B.

Acetylene Gas Generators and Gasometers, Impts. in. E. H. Maddock and W. Jones, both of Liverpool. Eng. Pat. 22,359, Oct. 8, 1896.

An inverted vessel has an annular space divided into compartments, each of which contains a generator, from which the gas passes into the central space of the vessel. Conical caps seal the tops of the generators or the vacant compartments. Water condensed from the gas is removed by a drip-tap at the lower end of an inclined exit pipe.
—J. A. B.

Acetylene Gas, Impts. in and connected with the Production of. L. F. FitzGibbon, London. Eng. Pat. 22,526, Oct. 10, 1896.

A sheet-metal vessel, containing water, has either a side shoot below the water-level for the admission of carbide, or a vertical feed-tube closed by a conical float. Ammonia is removed from the gas by the use of dilute hydrochloric acid either as the decomposing liquid or in a washer. The decomposition is moderated by agglutination of the carbide with 8 to 10 per cent. of paraffin.—J. A. B.

Acetylene Gas, Impts. in or relating to Apparatus for the Manufacture of. C. F. J. B. Bécherel, Paris. Eng. Pat. 23,289, Oct. 20, 1896. (Under Internat. Convention.)

A gasholder acts as or actuates a float which, by displacement of water within a reservoir, causes an overflow therefrom to an acetylene generator, which discharges into the gasholder.—J. A. B.

Acetylene Gas, Impts. in or relating to Apparatus for the Manufacture of. C. F. J. B. Bécherel, Paris. Eng. Pat. 23,290, Oct. 20, 1896.

Is supplementary to the preceding specification. Two or more generators, arranged stepwise, or in other manner, are put in operation in turn by the float or floats actuated by the gasholder.—J. A. B.

Acetylene Gas Generator, An Improved Automatic. A. J. Smith and A. G. Smith, both of Aberdeen. Eng. Pat. 24,414, Nov. 2, 1896.

A fixed gasholder, open at the bottom to its containing tank, communicates by a U-tube with the side of a carbide chamber provided with a gas-exit to the gasholder. Water passes to the chamber until the gas in the holder depresses its level below the mouth of the U-tube. By means of two U-tubes and a four-way cock, two carbide chambers may be used in turn. Partitions to prevent splashing on board ship are inserted in the holders.—J. A. B.

Acetylene, Impts. in Apparatus for the Manufacture of. P. de Resener and H. L. A. Luchaire, both of Paris. Eng. Pat. 24,440, Nov. 2, 1896. (Under Internat. Convention.)

Two closed receivers for carbide of calcium are connected with the bottom of the tank of a bell gasholder; and also, by inverted U-tubes passing over its rim, with a point a little above its bottom. A pipe, entering the top of the bend of each of these tubes, communicates with the interior of the bell, and permits the flow of water to the receivers only when the bell is low. The receivers are used alternately. Superposed boxes for carbide, and translucent receivers are claimed.—J. A. B.

Acetylene Gas, An Improved Apparatus for the Manufacture of. U. Kesselring, St. Imier, Switzerland. Eng. Pat. 9714, April 15, 1897.

A distributing drum, divided into sectors, is charged with calcium carbide. A valve, operated by means of levers by the rise and fall of a gasholder bell, discharges the contents of each sector in succession into a water-box, whence the gas formed passes through a purifier containing moss to the gasholder.—J. A. B.

Carbide of Calcium, An Improved Furnace for the Production of. R. Haddan, London. From H. Colberg, Adrien Serret, and F. Amigo, Ibero. Eng. Pat. 16,398, July 10, 1897.

Acetylene gas, alone or mixed with a supporter of combustion, is injected into the fuel-burning chamber of a

furnace for the preparation of calcium carbide, in order to add to the temperature produced by the ordinary combustible in the furnace.—J. A. B.

Acetylene Gas, Impts. in Apparatus for the Generation of. G. C. Marks, London. From A. F. Bowers, Paris. Eng. Pat. 16,975, July 17, 1897.

Water is supplied to acetylene generators by the regulating device claimed in Eng. Pat. 13,511 of 1896 (this Journal, 1897, 721). The gas, water, and lime pass together from the generators to a separator, where the lime is retained, the water discharged, and the gas allowed to pass to the burners. A modification, which is also described separately as applied to a household lamp, has a distinct gas outlet to the place of consumption, with a side arm in communication with the device which regulates the supply of water to the generator.—J. A. B.

Acetylene Gas Generators, Impts. in. E. Godin, Three-Rivers, Quebec, Canada. Eng. Pat. 17,021, July 19, 1897.

A bell-shaped vessel supported in a tank of water communicates by two pipes—one below, one above the level of the water in the tank—with a vessel containing carbide. Water flows through the lower pipe on to the latter and the gas generated passing through the upper pipe depresses the water level within the bell. The gas exit is from an arm of the upper pipe.—J. A. B.

Oil Vapour Burners, Impts. in. G. C. Marks, London. From J. Gilbert, Paris. Eng. Pat. 25,801, Oct. 26, 1896.

The oil to be vaporised, for the production of a non-luminous flame for heating an incandescent mantle, is fed to the vaporising chamber through one or more tubes filled with thin metal wires regulating the delivery by capillary attraction. The gas passes down from the said chamber through tubes leading to the nozzle of the burner, where it becomes mixed with air, and burns, the flame heating the vaporising chamber and the superimposed mantle. The initial heating of the vaporising chamber is effected by any suitable means, such as the flame of a spirit lamp, but when once started the flame is self-maintaining.—C. S.

Benzine Lamp, Improved. L. d'Haemars, Brussels. Eng. Pat. 21,659, Sept. 30, 1896.

The benzine receiver is surmounted by an annular receiver containing alcohol and fitted at one side with a small burner and wick. The burner of the main lamp rests on the alcohol lamp, through the central tube of which a tube from the main burner passes down into the benzine receiver. The wick contained in this tube allows the benzine to ascend into a blind lateral duct, or retort, at the top, directly over the alcohol flame, whereby the benzine is vaporised, and the resulting gas ascends, mixed with air, into the main burner, where it is ignited, and burns. It is claimed that in this manner a perfectly regular flame is produced with a very small consumption of alcohol.—C. S.

Night-Lights, Impts. in. J. D. and H. Palmer and H. E. Trestrail, London. Eng. Pat. 21,374, Sept. 26, 1896.

For the more effectual diffusion of disinfectants and perfumes by the heat of burning night-lights, the former, instead of being mixed with the fat, are disposed in compartments so placed as to be heated by the flame. Thus the more volatile substances are placed in an outer jacket attached to the night-light or forming part of the glass vessel in which it is placed. Less volatile matters are contained in tubes of small bore inserted into holes in the fat near to the wick. Or, the volatile substance may be absorbed by blotting paper, which is wrapped round the fat and protected by a coating of wax outside.—L. A.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Mineral Oils, Solubility in Absolute Alcohol. S. Aisinmann. Chem. Rev. Fett- u. Harz-Ind. **4**, [12], 161—163; [13], 176—180.

Continuing his researches (see this Journal, 1895, 812) for a simple method of determining the local origin of mineral oils from their behaviour in presence of this solvent, the author finds that the crude oils are unsuitable for this purpose, as are also the fractions boiling below 250° C., the latter being readily miscible in absolute alcohol. He therefore proposes the distillation (by the Engler fractional method) of 100 c.c. of crude oil and the collection of the fraction coming over between 250° and 270° C., 5 c.c. of which are then well shaken up with successive additions of alcohol (sp. gr. 0·705) until the initial turbidity gives place to a clear solution. The following results were obtained for the oils indicated :—

Solubility of Petroleum Fractions of Various Crude Oils in Absolute Alcohol (Sp. Gr. 0·705).

Crude Oil from	Specific Gravity of Fractions.						
	Up to 0·830.	0·835.	0·840.	0·845.	0·850.	0·855.	0·860.
Roumania I.	In all proportions.	In all proportions.	In 2·8 vols.	..	..	In 5·8 vols.	..
„ II. (Bustenari)	„	„	In all proportions.	In all proportions.	In all proportions.	In all proportions.	In all proportions.
Galicia I. (Krosno)	„	„	„	„	„	In 1·7 vol.	..
„ II. (Potok)	„	In 1·5 vol.	In 1·8 vol.	In 2·4 vols.	..	In 3·2 vols.	In 4·2 vols.
Russia ("Kunstöl") (90 per cent. petroleum and 8 per cent. masut).	„	In all proportions.	In 1·6 vol.	..	..	In 3 vols.	In 4·5 vols.

Solubility of Lubricating Oil Fractions from Various Crude Oils in Absolute Alcohol (Sp. Gr. 0·705).

Crude Oil from	Specific Gravity of Fractions.								
	0·865.	0·875.	0·885.	0·890.	0·895.	0·900.	0·905.	0·910.	0·915.
Galicia (Krosno)	..	..	..	..	In 7 vols.	In 9 vols.	In 9·5 vols.	In 12·5 vols.	In 14 vols.
„ (Ropieza-Ruska)	In 9 vols.	In 10 vols.	..	..	In 10 vols.	In 11 vols.	..	In 14·5 vols.	..
„ (Harklova)	..	..	..	In 6 vols.	In 6·5 vols.	In 8 vols.	In 12 vols.	In 13·5 vols.	..
Roumania (Bustenari).	..	In all proportions.	In 2 vols.	In 2·5 vols.	In 3 vols.	In 3·1 vols.	In 4·5 vols.	..	..

The subjoined table shows the relative proportion of the hydrocarbons boiling below and above 250° C. in the various petroleum fractions of some of the above-named crude oils :—

Crude Oil from	Vol. per Cent.	Specific Gravity of Fractions.									
		0·765	0·770	0·775	0·780	0·785	0·790	0·795	0·800	0·805	0·810
Roumania II. (Bustenari)	Up to... 250° C.	..	..	..	..	..	..	..	..	..	..
	Above .. 250° C.	..	..	..	..	..	..	..	..	..	..
„ I.	Up to... 250° C.	100	100	..	97·5	..	..	93	93	..	87·5
	Above .. 250° C.	0	0	..	2	..	..	6	7	..	12
Galicia I. (Krosno)	Up to... 250° C.	100	100	100	100	100	100	100	100	92	91
	Above .. 250° C.	0	0	0	0	0	0	0	0	8	9
„ II. (Potok)	Up to... 250° C.	100	100	100	100	100	100	..	92	..	86
	Above .. 250° C.	0	0	0	0	0	0	..	8	..	14
Russia ("Kunstol")	Up to... 250° C.	100	100	100	100	100	100	100	100	..	95
	Above .. 250° C.	0	0	0	0	0	0	0	0	..	5

Crude Oil from	Vol. per Cent.	Specific Gravity of Fractions.									
		0·815	0·820	0·825	0·830	0·835	0·840	0·845	0·850	0·855	0·860
Roumania II. (Bustenari)	Up to... 250° C.	..	..	..	..	..	..	..	..	..	65
	Above .. 250° C.	..	..	..	..	..	..	..	..	..	35
„ I.	Up to... 250° C.	..	..	78	..	63	30	..	..	0	0
	Above .. 250° C.	..	..	22	..	37	70	..	..	100	100
Galicia I. (Krosno)	Up to... 250° C.	89·5	87·5	..	77	74	73	..	65	54	36
	Above .. 250° C.	10·5	12·5	..	23	25	27	..	35	46	64
„ II. (Potok)	Up to... 250° C.	85	..	78	..	60·5	60	46·5	..	22·5	0
	Above .. 250° C.	15	..	22	..	39·5	40	53·5	..	77·5	100
Russia ("Kunstol")	Up to... 250° C.	..	90	..	81	..	50	..	0	0	0
	Above .. 250° C.	..	10	..	19	..	50	..	100	100	100

The author considers that the relative solubility of the fractions affords support to the views of Engler and Jeziaransky (this Journal, 1896, 191) on the nature of the hydrocarbons predominating in the fractions below and above 200° C., the former being saturated hydrocarbons miscible with alcohol in all proportions, and the latter (above 250° C.) more or less readily soluble according as they belong to the aliphatic or naphthene series.—C. S.

Petroleum Trade, Russian. Eng. and Mining J. **64**, [4], 95—96.

The United States Consul at Batoum reports that the increasing depth to which wells in the Baku district have to be sunk has considerably heightened their cost. At Balakhani, paying oil is still met with at between 500 and 700 ft., but at Sabountchi the oil layer is about 1,100 to 1,400 ft., and at Romani and Bibi-Eibat the latter depth is exceeded. The average cost of wells at Baku—based on a surface diameter of 26 ins., lined with 175-ft. of 26-in. pipe ($\frac{3}{16}$ to $\frac{1}{4}$-in. iron), 313 ft. of 24-in., 455 ft. of 22-in., 735 ft. of 20-in., and 1,025 ft. of 18-in. pipe, the well being finished with 1,148 ft. of 16-in. pipe—is about 4,000*l.*

Two large flowing wells were struck at Bibi-Eibat in 1896, one producing over three million barrels of oil in 36 days, and the other an even larger quantity. Prospecting has been carried on briskly, but with unsatisfactory results, one well having been sunk 861 ft. at Poota, and two others, 784 and 973 ft. respectively, at Hindar-Sindi (35 miles N. of Baku), without reaching paying oil, though surface indications and shallow trial drillings were favourable. Another well at Sonpsa reached a depth of 400 ft., and yielded a quantity of gas, but no oil, and at Koodakoo, on the Kuban river, six or eight wells have been sunk without any return.

The projected 8-in. pipe line (for refined oil) from Michailova to Batoum was to have been completed by October of the present year, but as the plant for making some of the pipe is only just erected at Mariopol, the line is not expected to be ready soon enough to influence materially the trade of 1897.

The Grosni field does not seem to be fulfilling expectations, as in January there were only 12 wells, yielding some 7,500 barrels per diem, seven new ones sinking, two being deepened, and eight new derricks. The largest of the two refineries near the railway station has only turned out one cargo of refined oil, and the other one has not yet begun working.

Probably the Grosni oil, which gives only 15—18 per cent. of burning oil, and leaves a residuum too heavy for fuel, cannot compete with Baku oil; and possibly, Grosni being nearer the home markets, it is more profitable to sell the crude oil for fuel than to refine it.—C. S.

IV.—COLOURING MATTERS AND DYES.

PATENTS.

Benzene and Monatomic Phenols, Action of Acid Chlorides on, in Presence of Ferric Chloride. M. Nencki and E. Stoeber. Ber. **30**, 1768—1772.

By mixing 5 parts by weight of benzene, 7 parts of benzoyl chloride, and gradually adding 7 parts of sublimed ferric chloride, a vigorous evolution of hydrochloric acid takes place, and finally the liquid solidifies to a mass of crystals. After washing with water and dilute hydrochloric acid, extracting with ether, and distilling off the latter, a fraction was obtained which was benzophenone. In a similar manner 7 parts of benzene, 8 parts of acetyl chloride, and 8 parts of ferric chloride give acetophenone. The phenols react like the hydrocarbons, and by mixing 5 parts of phenol with the same weight of carbon bisulphide, 6 parts of acetyl chloride, and slowly adding 7 parts of ferric chloride, *p*-oxyacetophenone is obtained, 100 grms. of phenol giving about 30 grms. of the ketone. The three isomeric cresols react in the same way. Thus *o*-cresol gives methyloxyacetophenone having the constitution—

$$CH_3 : OH : CH_3CO = 1:2:5.$$

The product crystallises in flat prisms melting at 104° C. The *m*-cresol also gives an aceto-ketone sparingly soluble in water, which melts at 126° C. It is probable from analogy that the constitution is $1.3.6.C_6H_3.CH_3(OH)COCH_3$. It is noteworthy that phenol and benzoyl chloride in presence of ferric chloride only give benzoic acid phenol ether. By the action of ferric chloride (8 parts) on a mixture of *p*-chlorophenol (6 parts) and acetyl chloride (8 parts), the authors obtained *p*-chloroxyacetophenone melting at 55° C. The product obtained from *o*-chlorophenol and acetyl chloride crystallised in needles melting at 96° C., and gave a hydrazone crystallising in yellow needles. By acting with ferric chloride (6 parts) on o-chlorophenol (5 parts)

and benzoyl chloride (6 parts), chloroxybenzophenone is obtained, which crystallises from alcohol in yellow needles melting at 176° C. In applying the same reaction to aromatic aldehydes, generally tarry, non-crystallisable products were obtained, but salicylic aldehyde yielded a dyestuff on slowly adding 1 part of ferric chloride to 4 parts of salicylic aldehyde. The product has probably the formula—

$$HO—C_6H_4—\overset{\diagup O \diagdown}{C = C}—C_6H_4—OH,$$

and was obtained by Bourquin (Ber. 17, 502), by the action of zinc chloride on salicylic aldehyde.—T. A. L.

Dimethyl-α naphthylamine, Some Derivatives of. P. Friedländer and K. Lagodzinski. Mitt. des k.k. Tech. Gew. Museums, 1897, 7, 125—132.

Dimethyl-α-naphthylamine was first obtained in 1870 by Landshof, by the action of methyl iodide on naphthylamine. Hantzsch, later, obtained a quantitative yield of it by heating the dry hydrochloride of the latter base with methyl alcohol. Monnet, Reverdin, and Nölting studied the action of mild oxidising agents upon it, with the object of preparing a compound analogous to Methyl Violet, but found that the base was in every instance decomposed without the formation of a dyestuff. Dobner, and Friedländer and Welmans jointly, attempted, also unsuccessfully, to produce the naphthalene analogue of Malachite Green—the first-named by treating the base with benzotrichloride, the latter two chemists by oxidising tetramethyldiamidodinaphthylphenylmethane, which results from the action of benzaldehyde on the base. The latter, moreover, observed that hexamethyltriamidotrinaphthylmethane, like the tetramethyldiamido compound mentioned, is decomposed when oxidised.

1.4-Amidodimethylnaphthylamine was obtained by Friedländer and Welmans by the reduction of benzene-azodimethylnaphthylamine sulphonic acid It may be also prepared by reducing the nitroso compound, which is formed by the action of nitrous acid on dimethyl-α-naphthylamine, and again (P. Cohn, Monatsh. f. Chem. 16, 798), by reducing tetramethyldiamido-azonaphthalene. It is an oil which rapidly oxidises on exposure to air.

Carbonyl chloride acts upon dimethyl-α-naphthylamine at the ordinary temperature, yielding a carboxylic acid, which, when treated with diazotised sulphanilic acid, loses carbon dioxide and gives an azo compound. This is also formed from dimethyl-α-naphthylamine and the same diazo compound. It would therefore appear that the carboxyl group occupies in the carboxylic acid the position para relatively to the dimethylamido group.

Bromine acts upon the base in acetic acid solution, giving a monobromo derivative. This does not combine with diazo salts, and hence would appear to be a para-substituted compound. It is an oil decomposing at 260°.

1.4-*Dimethylamidonaphthol* is obtained by alkali-fusion from 1.4-dimethylamidonaphthalene sulphonic acid. It forms long, colourless prisms (m. pt. 113°), which turn brown on exposure to air. It is sparingly soluble in water and ether, but dissolves readily in alcohol. It forms soluble compounds with acids and alkalis. Diazobenzene chloride acts upon it in an alkaline solution, forming a red-orange compound. Chromic acid colours its aqueous solution cherry-red, while ferric chloride is without characteristic action.

1.4-Dimethylamidonaphthol is not produced by the action of nitrous acid on the above-mentioned amidodimethylnaphthylamine, but α-naphthoquinone instead. The latter is also formed by the action of nitrous acid on an acidified solution of dimethylamidonaphthol.

Formaldehyde condenses with dimethyl-α-naphthylamine, giving *tetramethyldiamido-α-dinaphthylmethane*, a compound which crystallises in flat, colourless prisms (m. pt. 173°—174°, uncorr.). It dissolves with difficulty in alcohol and ether, but with ease in benzene and in aqueous solutions of acids, and it does not react with diazo salts.—E. B.

1.5-*Amidonaphthol.* P. Friedländer and K. Lagodzinski. Mitt. des k.k. Tech. Gew. Museums, 1897, 7, 130—131.

This compound is prepared, by fusion with caustic potash at 230°—235°, from the corresponding naphthylamine sulphonic acid, which is obtained by sulphonating α-naphthylamine in the cold. It forms nearly colourless needles, melting with decomposition at 193° (uncorr.); does not yield a platino-chloride double salt or a precipitate with stannous chloride and is coloured green by ferric chloride and bluish-red by chromic acid.—E. B.

Aldehydes, Synthesis of Aromatic. L. Gattermann and J. A Koch. Ber. 30, 1622.

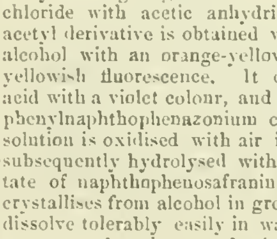
See under XXIV., *page* 832.

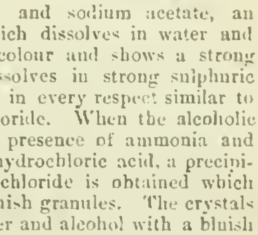
Phenylphenazonium into Phenosafranine, and Nietzki and Ott's Isorosinduline into Naphthophenosafranine; Conversion of. F. Kehrmann and W. Schaposchinkoff. Ber. 30, 1565—1572.

When the orange yellow alcoholic solution of acetylaposafranine chloride is agitated for some time with aqueous ammonia in the air, the deep bluish violet colour of the solution finally turns magenta red and the solution contains acetylphenosafranine. On acidulation with hydrochloric acid and boiling, the acetyl group is split off, and on evaporating the alcohol, phenosafranine chloride crystallises out identical in every respect with that obtained from aniline and *p*-phenylenediamine. It has already been shown that the phenazonium salts are converted by ammonia in an alcoholic solution into aposafranine salts, and it is thus possible to convert, on the one hand, phenosafranine through aposafranine into phenazonium, and, conversely, the latter through aposafranine and its acetyl derivative into phenosafranine.

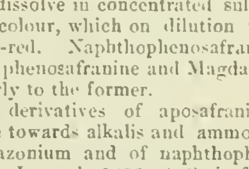
By heating together on the water-bath isorosinduline chloride with acetic anhydride and sodium acetate, an acetyl derivative is obtained which dissolves in water and alcohol with an orange-yellow colour and shows a strong yellowish fluorescence. It dissolves in strong sulphuric acid with a violet colour, and is in every respect similar to phenylnaphthophenazonium chloride. When the alcoholic solution is oxidised with air in presence of ammonia and subsequently hydrolysed with hydrochloric acid, a precipitate of naphthophenosafranine chloride is obtained which crystallises from alcohol in greenish granules. The crystals dissolve tolerably easily in water and alcohol with a bluish ponceau-red colour and show a strong orange-yellow fluorescence. They dissolve in concentrated sulphuric acid with a bluish-green colour, which on dilution turns dirty blue and finally rose-red. Naphthophenosafranine stands intermediate between phenosafranine and Magdala Red, but approaches more nearly to the former.

Since the acetyl derivatives of aposafranine and of isorosinduline behave towards alkalis and ammonia exactly like the salts of phenazonium and of naphthophenazonium (Ber. 29, 2316; this Journal, 1896, 802) it follows that these acetyl derivatives are azonium compounds, and have a constitution corresponding to the non-substituted azonium compounds, whilst acetyl-rosinduline cannot be directly amidised. This behaviour is at once evident if acetyl isorosinduline and acetyl rosinduline be regarded as derivatives of β-naphthoquinone, according to the following formulæ:—



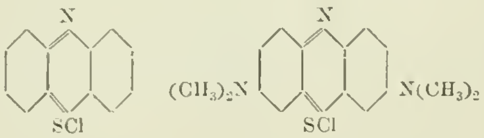


Similarly, acetylaposafranine has the following formula:—



which alone explains the conversion by ammonia into phenosafranine.

Criticising Fischer and Hepp's paper (Ber. **30**, 391; this Journal, 1897, 435), it is pointed out that the conclusion of the former as to the formula of methylphenosafranine is incorrect; and, further, from information to be published later, there is reason to believe that the indones are, as Nietzki assumes, inner anhydrides of *o*-quinonoid azonium compounds. With regard to the oxazine and thiazine dyestuffs, only those of the former which contain ammonium groups are strong bases. Thus the simplest Meldola Blue obtained from *β*-naphthol and quinone dichlorimide is less basic than the corresponding eurhodine, whilst the isomeric phenonaphthazime with the imido group in the naphthalene ring is so feeble a base that it will not even give an acetate; but neither substance is to be compared at all, as regards basicity, either with rosinduline or with isorosinduline. The thiazine dyestuffs are tolerably strong bases, but for another reason. It has been found that the simplest thiazine corresponding to aposafranine, and having, according to Bernthsen, a *p*-quinonoid formula, can, like aposafranine, be diazotised, and gives azo compounds with phenols. Hence the thiazine dyestuffs may exist in a form corresponding to the azonium compounds, according to the following scheme:—

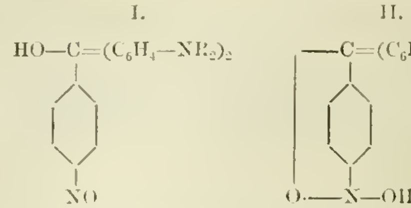


Thiazine chloride. Methylene blue chloride.

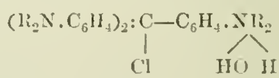
the pentavalent nitrogen being replaced by tetravalent sulphur. The bases free from oxygen, of the thiazines and the thiazones, however, still have most probably a *p*-quinonoid constitution, and in the oxazone series an *o*-quinonoid formula would require the assumption of tetravalent oxygen. It is pointed out finally that Fischer and Hepp subsequently confirmed (Ber. **29**, 2760; this Journal, 1897, 132) Kehrmann's observation (Ber. **29**, 2316, *loc. cit.*) that naphthophenazonium is converted by alcoholic ammonia into rosinduline.—T. A. L.

Rosanilines or Bases of the Corresponding Rosanilines, New Methods for Converting p-Nitrodiamidotriphenylmethane into. M. Prud'homme. Bull. Soc. Chim. 1897, 654—659.

By heating together 1 grm. of *p*-nitrodiamidotriphenylmethane, 10 grms. of caustic soda (38°), 80 c.c. of water, and 160 grms. of alcohol (95° Tr.) in a flask furnished with a reflux condenser, a product is obtained which, by the action of reducing agents, is converted into *p*-rosaniline or into a corresponding base. The reduction may be effected in an acid or alkaline solution: according to the former method, by dissolving the product in dilute acetic acid, heating to boiling, and adding sodium sulphide, stannous chloride, or zinc dust. The shade passes from dirty violet to the characteristic red of *p*-rosaniline. If, instead of employing acetic acid, the product be dissolved in dilute hydrochloric or sulphuric acid and reduced in the cold with zinc dust, a colourless solution is obtained which contains the *p*-hydroxylamine of diamidotriphenylmethane. This, on boiling, is converted into triamidotriphenyl carbinol. If the reduction be carried out in an alkaline solution, the time of boiling varies with the nature of the reducing agent. The best yield is obtained by heating together, for about 12 hours, 1 grm. of the *p*-nitroleuco base, 8 grms. of crystallised sodium sulphide, 80 c.c. of water, and 160 grms. of alcohol. Only about 60 per cent. of the theoretical yield is, however, obtained. By employing the tetramethylated base it is possible to isolate two substances which are formed in the reaction, and the constitution of which may be expressed by the formulæ given at the top of the next column. A mixture of 1 grm. of the tetramethylated leuco base, 10 grms. of caustic soda (38°), 80 c.c. of water, and 160 grms. of alcohol is heated together on the water-bath for about $1\frac{1}{2}$ hours, or until the leuco base is completely dissolved. After filtering and standing 24 hours, the filtrate deposits



red crystals, whilst the mother-liquor, on standing for several days, yields yellow crystals. The red substance changes colour at 85° C., and is completely melted with partial decomposition at 100°—105° C., whilst the yellow crystals melt at 142°—143° C. The two compounds, when acted on by reducing agents, yield Tetramethyl-rosaniline Violet, and the same dyestuffs are produced from both by their action on phenols and amines. They differ, however, in respect to their behaviour to acids. The red substance dissolved in dilute mineral acids or concentrated acetic acid, more especially in presence of alcohol, gives a blue solution which dyes cotton mordanted with tannin or silk a greyish-blue. On heating or standing, this solution turns yellowish-green. The yellow substance on the other hand, dissolves to an almost colourless solution, which only becomes coloured on heating, yielding the same green as the red substance. The author considers that formula I. corresponds to the yellow substance, whilst the red crystals are represented by formula II., and that the intermediate blue compound has the formula—



This subsequently loses water, yielding $Cl—C=(C_6H_4.NR_2)_3$, and is thus a confirmation of Rosenstiehl's formula for rosaniline. That the yellow substance is not at once coloured by dilute acids is probably due to the fact that the hydroxyl of diamidotriphenyl carbinol is less easily attacked than that of triamidotriphenyl carbinol, and it is known that the triphenyl carbinol is no longer capable of etherification.

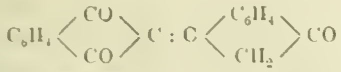
—T. A. L.

Cochineal Dyestuffs.—II. C. Liebermann and H. Voswinckel. Ber. **30**, 1731—1744.

The authors confirm their formulæ, $C_{10}H_8O_7$ and $C_9H_8O_5$, already given for cochenillic and coccic acid (Ber. **30**, 688; this Journal, 1897, 474). They have further examined the tribromocresotic acid, which is obtained by treating cochenillic acid with bromine water. When acted on with sodium amalgam this acid is slowly converted into cresotic acid, melting at 210° C. This 1.3.5 cresotic acid is also obtained directly from cochenillic acid by heating for 2—3 hours with water to 200°—210° C. Since, moreover coccic acid has the composition of a cresol dicarboxylic acid, and is generally formed in the preparation of cochenillic acid, it appeared probable that this acid was an intermediate product of the decomposition of cochenillic acid, which was found to be actually the case. By heating cochenillic acid for 1 hour with water to 170° C., carbonic acid, cresotic acid, and coccic acid are obtained. The latter acid is identical with oxyuvitic acid, and has the constitution $1.3.4.6.C_6H_2.CH_3.OH.(CO_2H)_2$. This acid is termed *α*-coccic acid, since an isomeric acid termed *β* has been obtained by repeated sublimation of cochenillic acid. Water and carbonic acid are given off and the anhydride of a dicarboxylic acid is formed, which on boiling with water, yields *β*-coccic acid, melting at 164° C. It has the constitution $1.3.5.6.C_6H_2.CH_3.OH.(CO_2H)_2$, whilst cochenillic acid is homohydroxyhemimellithic acid of the formula $1.2.4.5.6.C_6H.CH_3.OH.(CO_2H)_3$. With regard to the constitution of Carmine itself, $C_{11}H_{12}O_6$, the authors consider that it is probably a hydrindene derivative, and that its parent substance is diketohydrindene—

$$C_6H_4\left\langle\begin{matrix}CO\\CO\end{matrix}\right\rangle CH_2$$

(from phthalic ester and acetic ester). This, as Wislicenus and Kotzle (Annalen, **246**, 347, 252, 72) have pointed out, is easily converted into a condensation product, probably—



which has the remarkable property of dissolving in cold dilute alkali with a red colour, and of dyeing wool red from a slightly acid bath, although it can in no sense be considered as a mordant dyestuff.—T. A. L.

PATENTS.

Colouring Matters [Saccharofluoresceïn], Impts. in and relating to the Production of. G. B. Ellis, London. From "La Société Chimique des Usines du Rhône," and G. P. Monnet et Cartier, Lyons, France. Eng. Pat. 21,197, Sept. 24, 1896.

In a similar manner to that already described in Eng. Pat. 21,196 of 1896 (this Journal, 1897, 735), dyestuffs of the same class can be obtained by condensing saccharine with *m*-amidophenol or cresol, or with the mono- or dialkylated derivatives of these substances. For instance, 1 kilo. of diethyl-*m*-amidophenol is heated with twice its weight of saccharine (anhydrosulphamidobenzoic acid) for 36 hours to 165° C. The time of reaction may be considerably shortened by the use of condensing agents. When the reaction is complete, the melt is cooled, ground, and suspended in water containing at least 1 kilo. of sodium carbonate. After boiling up and filtering from dissolved saccharine (about half the weight of that taken), the insoluble residue is digested for several hours with caustic soda-lye. The insoluble powder is finally dissolved in water made strongly acid with hydrochloric acid. The boiling solution, after filtering, deposits on cooling small green crystals with a metallic reflex. The product in this condition is slightly soluble in water, giving a violet-red solution with a yellow fluorescence. When this solution is boiled, it becomes decolorised and deposits a violet flocculent precipitate. The colour reappears on the addition of an acid. A similar decomposition takes place on the addition of an alkali. The base, after recrystallisation from benzene, melts at 240°—245° C. The products are known as Sulpho-rhodamines.
—T. A. L.

Colouring Matters [Anisolines], Impts. in and relating to the Manufacture of. G. B. Ellis, London. From "La Société Chimique des Usines du Rhône," and G. P. Monnet et Cartier, Lyons, France. Eng. Pat. 21,198, Sept. 24, 1896.

The specification describes a novel method for alkylating Rhodamines by converting these latter into their acid chlorides, which react with alcohols, forming ethers or esters. The method consists in suspending the Rhodamine salt or its base in phosphorus oxychloride, chloroform, carbon bisulphide, or other indifferent solvent or diluent, and adding one molecular equivalent of phosphorus pentachloride. A brisk reaction takes place, and hydrochloric acid is evolved. The melt is finally boiled for some time under an inverted condenser and the chlorides of phosphorus are subsequently removed by distillation. The rhodamine chloride remains as a resinous mass with a brownish-red to greenish reflex. A purer product may be obtained by carrying out the reaction in the cold and precipitating the product with toluene. In order to convert it into an Anisoline the product is powdered and boiled with absolute alcohol when it gradually dissolves. After driving off the alcohol the crystalline product remaining may be purified by dissolving in water, adding sufficient hydrochloric acid to form the salt, and precipitating with salt.—T. A. L.

Ortho- and Paranitrobenzylaniline and its Homologues, Manufacture of. O. Imray, London. From "The Farbwerke vorm. Meister, Lucius, and Brüning," Hoechst a M., Germany. Eng. Pat. 22,041, Oct. 5, 1896.

The action of chlorine or bromine on *o*- or *p*-nitrotoluene, with or without a halogen "carrier," gives under the best conditions more than 50 per cent. of the corresponding nitrobenzyl chloride or bromide, but hitherto no satisfactory method has been known for obtaining the nitrobenzyl haloid in a pure state. By reacting with aniline or its homologues on the nitrobenzyl haloids, they are converted into bases of the general formula $C_6H_5.NH.CH_2.C_6H_4.NO_2$. These on oxidation yield the corresponding nitrobenzylidene compounds $C_6H_5.N:CH.C_6H_4.NO_2$, which by the action of dilute mineral acids are converted into amido bases and nitrobenzaldehyde. The separation of the pure nitrobenzyl haloid is thus of great technical importance, and is carried out by heating to 85°—90° C. with wet steam, in a copper or cast-iron boiler provided with a rapid agitator, about 300 kilos. of an *o*- or *p*-nitrotoluene chlorinated to about 50 per cent., corresponding to about 170 kilos. of *o*- or *p*-nitrobenzyl chloride, together with 190 kilos. of aniline, or the equivalent amount of toluidine or xylidine. When the reaction has commenced, the temperature rises spontaneously to 120°—180° C., according to whether aniline or one of its homologues has been employed. When the temperature falls, the reaction is complete, and after cooling to about 90° C., 300 litres of water are added, and the whole is boiled up, when the aniline hydrochloride dissolves and may be drawn off. The insoluble residue, consisting of nitrobenzylaniline and nitrotoluene, is distilled with steam, when the latter passes over, leaving nitrobenzylaniline, which may be purified by crystallisation from alcohol or by dissolving in dilute acids and reprecipitating. Nitrobenzyl bromide may be used in place of the chloride, and caustic alkali, alkaline carbonate or acetate, or an alkaline earth or ammonia may be substituted for the aniline; but in this case the addition of 50—100 litres of water is advisable.—T. A. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

Tendering of Cotton Fibres by Acid during Sulphur Stoving. Mitt. aus den k.t. Versuchsanst. zu Berlin, 1897, **15**, [2], 134—136.

In a sample of flannel, in which the warp consisted of cotton and the weft of wool, it was noticed that the cotton had become tender, the wool not being affected. The tender parts had a yellow appearance, and crumbled on being touched. According to the report of the works, the webs were milled, washed twice, and bleached in the sulphur stove.

The destruction of the cotton was due to free sulphuric acid, of which there was a considerable quantity in the flannel. As the material itself could not contain any sulphuric acid, its presence must have been due to the sulphurous acid used in the bleaching, having become oxidised to sulphuric acid, the latter not having been washed out. On making a close search at the mill, an aperture was found in the sulphur stove, which admitted the air. The flannel made after closing this aperture was not tendered.—S. P. E.

PATENTS.

Fibres and Fibrous or Textile Materials, Impts. in the Treatment of, to Improve the Appearance or Finish and Facilitate the Operation of Dyeing. J. Schneider, Ashton-on-Mersey. Eng. Pat. 19,428, Sept. 2, 1896.

An improvement in the mercerising process is described. The mercerising agent recommended is sodium or potassium sulphide, but caustic soda may be used. In either case an addition is made to the bath of a solvent for fatty matters; this may be alcohol, benzene, petroleum spirit, aniline, or turpentine. The fibre is stretched either during mercerising or immediately afterwards.—R. B. B.

Silky or Similar Lustrous Effect, Impts. in the Production of, on Yarns, Threads, and Fabrics having a Vegetable Origin. A. Liebmann, Manchester. Eng. Pat. 19,633, Sept. 5, 1896.

To obtain the desired effect, yarns or fabrics made from Egyptian or Sea Island cotton, slightly twisted and loosely doubled, are mercerised in caustic soda at 52° Tw. The

liquid is partially removed by squeezing, and the material subjected to stretching for 5—10 minutes, well washed with water, and released from tension. The appearance and softness of the fibre are enhanced by a subsequent treatment with soap or acetic acid.—R. B. B.

Cotton, A Process for Giving a Silk-like Appearance to. O. Seyfert, Glauchau, Saxony. Eng. Pat. 28,870, Dec. 16, 1896.

Cotton, mercerised with caustic soda in the usual manner, is dried at a low temperature (30°—40° C.), in a tightly stretched condition, and finally washed without tension. —R. B. B.

Mercerising Vegetable Fibrous Substances, Impts. in or relating to, and in the Manufacture of Fabrics. R. Thomas and E. Prevost, Crefeld, Germany. Eng. Pat. 20,714, Sept. 18, 1896.

This patent describes several modifications and extensions in Thomas and Prevost's previous patent (18,040 of 1895; this Journal, 1896, 804), relating to the mercerising of fibres and fabrics under tension. The extensions claimed are: (1) the treatment in a similar manner of vegetable fibres on cops, reels, or bobbins; (2) the fibrous substance is stretched beyond its original length during or immediately after the mercerising process, and is then washed without tension; (3) tightly twisted yarns and dense fabrics are washed with dilute acid or alkali, to neutralise the alkali or acid used for mercerising, instead of washing with water; (4) mercerised and non-mercerised yarns are woven together and the mixed fabric is dyed; (5) more dilute caustic soda may be employed, *viz.*, at 10°—20° B., if the temperature during mercerising is reduced to 0° C., the stretching being continued until the normal temperature is regained.—R. B. B.

Textile and other Fabrics, Impts. in or relating to. J. and H. Jelley, London. Eng. Pat. 23,588, Oct. 23, 1896.

It is claimed that fabrics are toughened and made less easily penetrable by sharp objects when impregnated or coated with a mixture of turpentine and glycerin in naphtha or other solvent. The material thus obtained is stated to be specially suitable for pneumatic tyres.—R. B. B.

Jute Bast, China Grass, Rhea, and like Plants; Impts. in the Treatment of, for Use in Textile Industries. A. H. Prinz, Vienna, Austria. Eng. Pat. 11,804, May 12, 1897.

The crude jute bast, &c., is loosened and freed from tips and root ends by well-known mechanical methods. A special chemical process then follows, consisting of four operations: (1) The fibres are macerated in the cold with caustic soda, sp. gr. 1·035. The liquors are used for three successive quantities of fibre, being made up to the original volume for this purpose. (2) Without washing, the fibres are exposed for 8—12 hours to the action of chlorine gas, and it is claimed that the incrustations are thereby converted into chlorine compounds, which are easily removed, after washing with water, by (3) a second treatment with caustic soda similar to the first. After again washing, the fibres are (4) bleached by means of hypochlorite of soda at 5° B. for 4—6 hours, and are allowed to remain in the bleaching vessel for 2—4 hours after drawing off the liquor. A final washing to remove chlorine completes the process.—R. B. B.

Absorbent Material for Use in Making Boot Socks, Dress Shields, and other Articles of Clothing. F. Bossbardt, Manchester. From Rufle-Sulzer and Co., Glarus, Switzerland. Eng. Pat. 13,965, June 8, 1897.

Sponge is reduced to fine particles, freed from sand and other foreign matter, washed in a dilute soda solution, and further torn up by mechanical means. A little hemp is added to the paste obtained, and the whole pressed into moulds, squeezed, and dried. The material is called "sponge wadding," and, when enclosed between layers of textile material, may be used for boot socks, &c. for the purpose of absorbing moisture and perspiration.—R. B. B.

Bleaching and Disinfecting Fibrous Materials, Impts. in, and Apparatus for that Purpose. V. Floquet and L. Bonnet, Rheims, France. Eng. Pat. 14,319, June 12, 1897.

In order to obviate certain defects of the sulphur bleaching process due to contact with air, bleaching is carried on in a vessel from which the air has been exhausted. Sulphurous acid in liquid form is admitted, and acts—it is claimed—with great rapidity. When the action is complete the acid is pumped out, and water for washing is admitted. —R. B. B.

Asbestos Fabric, Improved, and Process for the Production of the same. R. Klinger, Gumpoldskirchen, Austria. Eng. Pat. 16,379, July 10, 1897.

Asbestos is divided into very fine fibres, of as great a length as possible, and immersed in an india-rubber solution, the whole being thoroughly mixed until each fibre appears coated with the solution; the solvent, *e.g.*, petroleum spirit, is then evaporated. The asbestos fibres will have become very adhesive, and the mass may be compressed or rolled into any desired form. The name given to the resulting material is "asbestos leather," and it is said to resemble leather in its properties and structure.—R. B. B.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Aniline Black on [Cotton] Knitting Yarn. H. Weise. Leipziger Färber Zeit. **46**, [7], 285—286.

A black on cotton yarn, not turning greenish, and which does not rub off, is obtained as follows:—

1 kilo. of glue is softened, and subsequently dissolved in 10 litres of water, and then 3,600 grms. of "aniline salt" (aniline hydrochloride) are dissolved in it. In a second vessel, 600 grms. of sodium chlorate and 300 grms. of ammonium chloride are dissolved in 10 litres of hot water. In a third vessel, 150 grms. of copper sulphate are dissolved in 8 litres of water. When cold, these three solutions are united, and 2 litres of aluminium acetate solution (prepared as stated below) are added to the mixture.

The previously well-cleansed and dried yarn is twice impregnated with this solution in lots of 1 kilo., and aged at a temperature not exceeding 30° C. For developing 25 kilos. of yarn, 1,200 grms. of potassium bichromate are dissolved in a sufficiency of boiling water, and 100 grms. of "aniline salt" are added to the bath. The yarns are passed through this bath four times, and, after adding ¼ litre of sulphuric acid, six times more, well rinsed, and finally soaped.—I. S.

Woollen Cloth after Carbonising, Uneven Dyeing of. K. Schimke. Färber Zeit. **8**, [15], 233—234.

The author calls attention to the difficulty of obtaining level shades with the dye-woods on woollen cloth from which every trace of acid has not been removed after carbonising. By careless work during the carbonising process, wool may be permanently injured for dyeing purposes, as such "acid stains" do not dye to the same depth of shade as the other parts.—I. S.

PATENT.

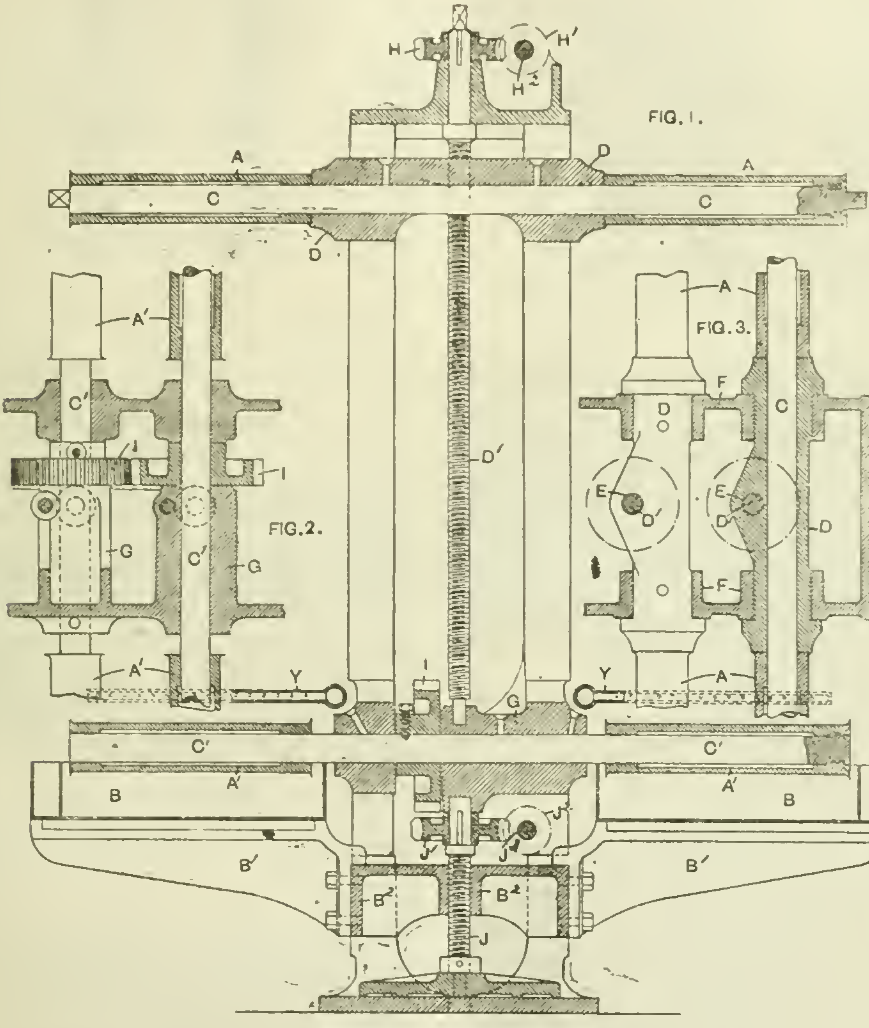
Dyeing, Finishing, Sizing, Washing, or similarly Treating Yarn; A New or Improved Machine for. F. Davies, Blackburn, and A. Liebmann, Manchester. Eng. Pat. 21,492, Sept. 29, 1896.

This machine has primarily been designed for the imparting of a silky lustre on cotton yarn by mercerisation whilst in a stretched condition. Its essential features are a pair (or a number of pairs) of parallel rollers, to receive the hanks of yarn.

The bearings of the rollers are mounted in a slide block traversed by a screw, by actuating which, the distance between the rollers may be increased or diminished, so as to stretch the yarn.

Fig. 1 shows a transverse section; Fig. 2 a partial horizontal section of two contiguous lower rollers; and Fig. 3 a similar section of two upper rollers.

The yarn is arranged upon and around the rollers A, A[1], each lower roller rotating within a cistern B, which can be raised or lowered by the screw J, on which slides the block B[2] carrying the brackets B[1] on which the cisterns rest. The rollers are mounted upon the through-shafts C, passing through a sliding block D, upon one side of which there is a projecting part having a vertical screw-threaded hole E, through which is passing the vertical screw D[1]. On the upper end of each of the screws there is fixed a worm wheel



H, with which gears a worm H[1], fixed upon the shaft H[2]. By rotating this shaft in one or other direction, the upper rollers are raised or lowered, as the case may be.

The lower rollers A[1] are similarly mounted on the shafts C[1], passing through the stationary block G, and are geared together by spur wheels I, by which means they are made to revolve, the upper rollers being driven by the hanks of yarn, which also serve as belts.

The hanks of yarn having been arranged upon the rollers, the latter are set in motion, and the tanks are filled with the caustic soda "or other mercerising solution." When the mercerisation is complete, the lye is allowed to run through valves into a reservoir, and the yarn is washed with water supplied in a spray by the perforated pipes Y, the water flowing into the tanks B, whence it escapes through an outlet.—I. S.

VII.—ACIDS, ALKALIS, AND SALTS.

Solubility of Certain Easily Soluble Salts in Water at 18° C. F. Mylius and R. Funk. Ber. 1897, **30**, [12], 1716—1725.

The powdered salts were shaken for at least an hour with water at 18° C.; excess of salt was allowed to settle, the clear liquid drawn off with a pipette, weighed, and analysed.

In the table the solubility is expressed (A) by percentage of anhydrous salt in the solution, (B) by the weight of anhydrous salt dissolved by 100 c.c. of water, and (C) by the number of water molecules to one molecule of anhydrous salt.

For purposes of regularity in the last column, the formulæ of salts of two monovalent constituents are doubled.

Magnesium iodate is known anhydrous and crystallised with 4 mols. of water. If the saturated solution be cooled to 0° and crystallisation effected by rubbing with a glass rod, the hydrate with $10H_2O$ separates; this melts at 50°, and slowly decomposes at the ordinary temperature. The transition point is 13° C. Modifications with less than $4H_2O$ are obtained by heating saturated solutions; these and the anhydrous salt are "insoluble" at the ordinary temperature, but dissolve in time after hydration.

Calcium iodate exists in the forms $Ca(IO_3)_2 + 6H_2O$ and $Ca(IO_3)_2 + H_2O$; the solubility curves cut at 32°. The existence of the hydrate with $4H_2O$ was not confirmed.

Salt.	Stable Form in Solution at 18°.	Specific Gravity of Saturated Solution.	A.	B.	C.
Lithium chlorate	$(LiClO_3)_2$	1·815	75·80	313·50	3·2
Calcium „	$Ca(ClO_3)_2 + 2H_2O$	1·729	64·00	177·80	6·5
Lithium chromate	$Li_2CrO_4 + 2H_2O$	1·574	52·60	110·90	6·7
Zinc chlorate	$Zn(ClO_3)_2 + 6H_2O$	1·914	65·00	186·20	7·0
Potassium fluoride	$(KF)_2 + 4H_2O$	1·502	48·00	99·30	7·0
Calcium nitrate	$Ca(NO_3)_2 + 4H_2O$	1·548	54·80	121·20	7·4
Magnesium chlorate	$Mg(NO_3)_2 + 6H_2O$	1·594	56·30	128·60	8·2
Zinc nitrate	$Zn(NO_3)_2 + 6H_2O$	1·664	53·90	116·90	9·0
Strontium chlorate	$Sr(ClO_3)_2$	1·839	63·60	174·90	9·3
Lithium bromate	$(LiBrO_3)_2$	1·833	60·40	153·70	9·8
Magnesium bromide	$MgBr_2 + 6H_2O$	1·655	50·80	103·40	9·9
„ iodide	$MgI_2 + 8H_2O$	1·909	59·70	148·00	10·4
„ nitrate	$Mg(NO^3)_2 + 6H_2O$	1·384	43·10	75·70	10·8
„ chromate	$MgCrO_4 + 7H_2O$	1·422	42·00	72·30	11·0
Lead chlorate	$Pb(ClO^3)_2 + H_2O$	1·947	60·20	151·30	13·7
Sodium chromate	$Na_2CrO_4 + 10H_2O$	1·409	38·10	61·40	14·6
Lithium iodate	$(LiIO_3)_2$	1·568	44·60	80·30	25·2
Sodium fluoride	$(NaF)_2$	1·044	4·30	4·40	104·8
Magnesium iodate	$Mg(IO_3)_2 + 4H_2O$	1·078	6·44	6·88	292·0
Calcium iodate	$Ca(IO^3)_2 + 6H_2O$	1·000	0·25	0·25	868·0
Lithium fluoride	$(LiF)_2$	1·033	0·27	0·27	1,038·0

—A. C. W.

Salt-Cake Furnaces, Measuring the Temperature of. G. Schüler. Zeits. f. angew. Chem. 1897, **12**, 361—364.

At the present time the correctness of the temperature at which a salt-cake furnace is worked is judged either by the appearance of the muffle or, where the pot is heated by the gases leaving the muffle, by the length of time in which the material in the pot becomes stiff. Neither of these methods is sufficiently satisfactory, especially when the close connection between the life of the muffle and the temperature to which it is heated, are taken into account. The author has therefore attempted to measure the temperature pyrometrically. He recommends the use of the Seger standard cones, the fusion of which is not a physical but a pyrochemical process (this Journal, 1894, 762).

The method of using is simple. A firebrick is covered with powdered fireclay, and the cones—say Nos. 1, 2, and 3—are pressed into this. The whole is then placed in that part of the furnace, the temperature measurement of which is desired, and is allowed to remain there for any desired time (24 hours).

The author chooses as point of observation that angle of the lower flue into which the hot gases fall from above. This place is easily reached through the cleaning holes; it is the hottest spot beneath the hearth, and, on account of the mixing of the gases caused by their change of direction, a mean heat is fairly certain to be attained. The cones must not be in any way protected from the direct flames, these also playing upon the firebrick. The flue dust has no influence on the test. The observation consists in noting which is the cone fusible with most difficulty, that melts during the period of observation. Each cone, the apex of which bending over touches the supporting plate, is to be considered melted. The results obtained are very sharp. From his observations the author concludes that the temperature to be maintained at the above point of observation should lie between the fusion points of cones 1 and 2; it should certainly not fall below 02 or rise above 3; that is, it should be 1,150°, with a range of from 1,100° to 1,200°. New hearths may be heated somewhat higher; the temperature will also vary according to the salt-cake production.

Regarding the kind of fireclay best suited for muffles, the author points out that the fire-resisting qualities are not the only ones to be considered. Not even the worst fireclay fuses at so low a temperature as cones 4 and 5, and yet many hearths are ruined at temperatures far below this point—in fact, below 900° C., since this temperature is above the fusing point of sodium sulphate. The burning of the hearth is thus not merely a question of the fusibility of the fireclay employed, but is to be ascribed to the greater fusibility produced in the mass by the gradual diffusion of the sulphate through the pores of the clay; and the use is therefore recommended of firebricks as non-porous as possible without injury to fire-resisting properties.—J. T. C.

Waste Acid from Metal Works. Public Health Engineer, Oct. 2. 1897, 415.

See under X., *page* 801.

PATENTS.

Carbonic Acid, Process and Apparatus for Purifying. I. Qurin, Cologne, Prussia. Eng. Pat. 18,468, Aug. 20, 1896.

Impure carbonic acid is strongly compressed in contact with water containing an oxidiser, such as potassium permanganate, in solution; dissolved gases, other than carbonic acid, are expelled either by passage of pure carbonic acid or by diminution of pressure; and, finally, the dissolved carbonic acid is released "by pressure diminution or by use of vacuum." "The water makes a continuous circuit through the whole apparatus employed, being saturated in the absorber with the carbonic acid, and freed in the purifier from foreign gases, in order to separate in the gas-remover the purified carbonic acid."—E. S.

Detergent and Bleaching Compounds, Impts. in the Manufacture and Treatment of, and in Apparatus employed therein. J. Hargreaves, Widnes, and R. Armstrong, Preston, Lancaster. Eng. Pat. 18,589, Aug. 22, 1896.

The sodium carbonate required for the preparation of detergent compounds is derived either from "vat liquor" of the Le Blanc process; from solutions obtained by the electrolytic decomposition of sodium chloride; or from mother-liquors occurring in the manufacture of sodium carbonates, processes being described adapted to each case. When caustic soda is present, it may be carbonated, according to one method, by addition of excess of sodium bicarbonate. A soluble detergent compound is obtained by incorporating monohydrated sodium carbonate with a strong solution of sodium carbonate at about 80° F., and then adding suitable oils, fats, fatty acids, resin, or the "niger" occurring in the manufacture of soap; but when "niger" is used any caustic alkali present is neutralised with acid fat or with sodium bicarbonate. The following is given as the approximate composition of an efficient compound:—Sodium carbonate, 50 parts; water, 35 parts; soap (estimated as fatty acids), 15 parts. To give bleaching properties, a small proportion of sodium hypochlorite is added, combined with oil of camphor, during or subsequent to the pulverising of the compound in the production of detergent powders.—E. S.

Chlorine Gas, Improved Means for Effecting the Absorption of; applicable in the Manufacture of Chlorates and Bleaching Liquor. W. R. Clarke and The United Alkali Company, Ltd., Liverpool. Eng. Pat. 20,068, Sept. 10, 1896.

Dilute chlorine, such as that produced in the Deacon process, is led up a high tower fitted with baffle plates,

down which milk of lime or magnesia is caused to flow. The gases taken from the top are led up a second similar tower, in which they meet fresh milk of lime, which usually suffices to absorb practically all the chlorine; but additional towers may be used if necessary. The liquid flows from the bottom of the towers into troughs having plug holes, whence it flows into tanks provided with agitators, each tower having its own system of pumps and vessels. From the tank belonging to the first tower, the liquid is passed through a coil of piping immersed in a cold-water tank, and is thence forced to the top of the tower, the process being repeated as often as necessary. When the liquid passing through the first tower has absorbed sufficient chlorine, it is replaced by the partially saturated liquid from the second tower, which, in turn, is supplied with fresh liquid. For the manufacture of chlorates, the milk of lime or of magnesia may be of such concentration as to give a finished liquor of 50° to 60° Tw.; but if bleaching liquor is to be made the milk of lime must not be fully saturated with chlorine, and should test about 7° Tw.—E. S.

Tartaric Acid and Tartrates, Process for Obtaining. A. Schmitz and E. C. W. Toenges, Düsseldorf, Prussia. Eng. Pat. 21,178, Sept. 24, 1896.

Argols, or other crude tartars, are boiled with an alkali carbonate solution in slight excess, and the warmed lye is treated with sodium hypochlorite until the brown colour of the liquid changes to a light vinous yellow; hydrochloric or acetic acid is then added until the reaction is neutral. The solution is precipitated by calcium chloride, the calcium tartrate separated, is boiled for some hours with sodium carbonate solution in slight excess, and the solution is treated with hypochlorite and neutralised as before. Pure calcium tartrate is then thrown down by calcium chloride, and may be decomposed in the usual manner to obtain tartaric acid. When it is desired to obtain cream of tartar, the calcium tartrate, first obtained as described, is boiled with potassium carbonate solution, and after separating the calcium carbonate, the liquid is treated with a little hypochlorite, and acid potassium tartrate is thrown down by addition of a suitable acid. The claim is for the obtaining of "tartrate of lime from raw products, partly manufactured substances, by-products, and refuse containing tartaric acid and tartrates" by the process described.—E. S.

Salt, Impts. in the Manufacture of, from Brine, and in Apparatus therefor. J. W. Weston, Thurcaston, Surbiton, and T. I. Weston, Wickham Bishops, Essex. Eng. Pat. 23,614, Oct. 23, 1896.

In order to obtain small bright crystals of salt, brine is spread in thin films, which are not allowed to evaporate to dryness, the crystals being separated from the mother-liquor by mechanical means. The boiler shown, is open at the top, has vertical sides and ends, and a bottom inclined towards two pockets for removing deposited salt. A hollow cylinder, partly immersed in the liquid, takes up as it rotates a film of the brine, the crystals formed during rotation being removed by a scraper into pockets, whence they are withdrawn by suitable apparatus. A fireplace below the boiler communicates with surrounding flues and with a flue traversing the brine, part of the fire-gases being led through the revolving cylinder. The crystals obtained are centrifugally drained and then dried.—E. S.

VIII.—GLASS, POTTERY, ENAMELS.

China, Manufacture of Limoges. U.S. Consular Reports, 1897, **54**, [202], 426—430.

Kaolin and Clays.—The body of the china consists principally of felspar and kaolin, a certain proportion of ground china being sometimes added. One of the features of Limoges glazes is the entire absence of lead. The kaolin used is that obtained from the very pure deposits which occur in the quarries around St. Yrieix, Haute Vienne; to this kaolin the formula $Al_2O_3.2SiO_2 + 2H_2O$ may be ascribed. Most of the deposits occurring in other parts of France and in Great Britain contain more or less water, iron, mica, magnesium, and often sulphur; and an excess of the first-named is extremely injurious in making the delicate paste used by Limoges potters. The inferior grades of china are frequently prepared by mixing the cheaper and impure clays with the pure kaolin. The kaolin is washed and ground, and all impurities removed before delivery at the works, where it is again washed. The liquid paste is passed between two electro-magnets, to remove all traces of iron, as the latter discolours the china, frequently by the formation of black spots. The superfluous water is then forced out of the clay, and the latter is twice subjected to the action of heavy zinc-covered cast-iron cylinders running over a zinc-covered table; this operation gives the requisite plasticity and evenness to the paste.

Machinery.—The machines are so arranged as to form several kinds of moulds. For plates, saucers, &c., three machines are used—one for reducing the paste to the desired thickness, the second for throwing the paste on to the moulds, and the third for finishing the article. Two machines are used for hollow ware, and the gauge, which has a side- as well as a down-motion, is arranged so as to fit both hollow and curved ware. For oblong platters, &c., the jigger has one long and one short motion of the eccentric. An improved cup-making machine is now used, which has not only the down-motion, but also the gradual side-motion corresponding to the movement of the human hand.

Firing.—The method of firing is different from that used by American and British potters, as the latter make a very hard bisque and a very soft glaze, whilst in the manufacture of Limoges china the reverse is the case. Up to the year 1895, the down-draught furnace system, with kilns built in two storeys, was used; but within the past two years, smaller, single-storey kilns have been employed. The latter are fired in a continuous manner by a system of flues connecting each kiln with the others at the top and bottom, and the waste heat, after passing through the whole series of kilns, escapes from the last one into the chimney. With this system, only half the amount of fuel is required to produce the same degree of heat, and the china is better fired in less than half the time required by the down-draught system.

Decoration.—The design is drawn upon stone in the same manner as a chromo-lithograph, each colour having a separate stone. The powdered colour is sprinkled on the paper after it has received the impression from the stone. Only that portion which should receive the given colour is impressed, so that after the different paints are passed upon the sheets called for by the design, the sheet is ready for transferring. To prevent lead-poisoning, the powdering is done under cover. The tops of the tables are in glass, the backs and sides are made of wood, but the front is of cloth, in which are armholes fitted with elastic bands, so that the hands can be thrust through and the powdering done without the danger of inhaling the dry colours. The work is seen through the glass top. The muffles are of two kinds. In the first, the firing is done with wood, and there is a space between the outside and the inside lining to protect the decorated ware from the smoke and impurities during combustion.—A. S.

Brick, Vitrified, as a Paving Material. J. W. Sibley. Eng. and Mining J. 1897, **64**, [7], 187.

See under IX., *page* 800.

PATENTS.

Permanent Photographs on China and other Vitreous Surfaces, A New or Improved Method or Process of Producing. J. Slater, Burslem. Eng. Pat. 21,081, Sept. 23, 1896.

See under XXI., *page* 825.

Glass, Porcelain, Enamel, or Metal: Impts. in or appertaining to a Process for the Production of White, Black, or Coloured Pictures, Writing, or the like on. F. Albrecht, Liverpool. Eng. Pat. 8890, Aug. 14, 1897.

This is a modification of the inventor's Eng. Pats. 13,838 of 1893 and 24,283 of 1895, for ornamenting glass, &c. The

invention consists in uniting gold leaf, powdered metals, or other substances to the surface of glass by means of a menstruum composed of 60 grms. of precipitated silica, or the equivalent amount of SiO_2 in the form of hydrate, in 1 pint of commercial silicate of soda solution. The use of other alkaline silicates in place of silicate of soda is, however, also claimed, and particularly the double silicate of soda and potash, though, in this case, a smaller proportion of silica is used. The material with which it is desired to decorate, is either mixed with the menstruum and so applied, or the menstruum is first applied and the colour afterwards dusted over. After pressing, the articles are stoved at a temperature of about 1,500° F.—H. H. B. S.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Cement Admixtures, Report of the Cement Trade Section of the London Chamber of Commerce on, with Reports and Evidence of Experts. 1897, 1—197.

An investigation into the admixture of foreign substances with Portland cement has recently been carried out under the auspices of the Cement Trade Section of the London Chamber of Commerce, who now issue a very complete report upon the subject. The Section record their belief that the practice of mixing materials with Portland cement after calcination does not prevail extensively on either the Thames or the Medway, and that, where it is practised, such admixture is, with the exception of gypsum, confined almost entirely to Kentish ragstone. They believe, however, that a considerable quantity of iron furnace slag is, at the present time, being mixed with cement of British manufacture, though not by either Thames or Medway makers.

The possibility of the interaction of ragstone and cement resolves itself into the question as to whether such mixtures show any tendency to lose carbonic anhydride on keeping. The experiments show that, instead of this being the case, such mixtures actually show a slight gain, due to absorption of carbonic acid from the air. Other conclusions arrived at are as follows:—Mixtures of ragstone and cement have a specific gravity lower than that of unmixed cement, and indeed correspond closely with that calculated from the respective specific gravities of the two materials. With fresh cement, admixture of ragstone tends to cause a prolongation of the setting-time, but this effect is not necessarily permanent, and the alteration of setting-time on keeping is more irregular than that taking place in unmixed cement. Mixtures of cement with 10 or 20 per cent. of ragstone show the same slight expansion when the test-bar is kept in water as does unmixed cement; 50 per cent. mixture expands somewhat less. Mixtures of cement with 10, 20, and 50 per cent. of ragstone, contract to the same slight extent as unmixed cement when the test-bar is kept in air. Cement which is not overlimed and is well matured is not improved by the addition of ragstone. None of the mixtures of ragstone and well-matured cement which have been examined, has proved equal in strength to the unmixed cement. Mixtures of ragstone and cement, when exposed to a red heat and subsequently slaked with water, disintegrate more readily than does unmixed cement.

A definition of Portland cement has been proposed, and a rule adopted, by the Cement Trade Section; to these all makers in Great Britain and Ireland are invited to subscribe, in the following terms:—

Resolved,—That Portland cement be defined as a mixture of two or more suitable materials, intimately and artificially mixed in the requisite proportions, and afterwards properly calcined and ground, to which nothing has been added during or after calcination, excepting that an addition, not exceeding 2 per cent. of gypsum, is permissible for the purpose of regulating the setting. That if any material whatever, excepting an amount not exceeding 2 per cent. of gypsum for the purpose of regulating the setting, be added to the Portland cement clinker during or after calcination, the article so produced shall not be sold as Portland cement, but under some other distinctive name.

—H. H. B. S.

Brick, Vitrified, as a Paving Material. J. W. Sibley. Eng. and Mining J. 1897, **64**, [7], 187.

It is stated that, by the use of vitrified brick, a pavement is obtained which will successfully resist the wear and tear of the horse-shoes, is non-absorbent, easily repaired, may be cleaned by washing or sweeping, and will present a smooth surface, even when the brick is worn half through.—A. S.

PATENTS.

Wood and other Cellular or Fibrous Substances, Improved [Electro-capillary] Process and Apparatus for Treating or Saturating with Liquids. L. A. Bretonneau and A. L. C. Nodon, Paris, France. Eng. Pat. 13,242, May 28, 1897.

See under XI. A., *page* 807.

Artificial Stone, A New or Improved Manufacture of. P. Kleber, Saarbrücken, Germany. Eng. Pat. 20,612, Aug. 11, 1897.

This invention consists in the production of an artificial stone which is not affected by atmospheric conditions. A dry mixture is made of sifted ashes, potash, and other alkaline carbonates, gypsum, and slaked lime. This is further mixed with dilute sulphuric acid and hydrated sulphate of lime, with the result that potassium sulphate is formed, which dissolves, and a plastic, pliable mass of gypsum is left, which is moulded by compression into the desired forms. With the view of getting rid of any alkali that may remain in the stone, it is finally immersed in a solution of alum acidified with sulphuric acid. The alum is decomposed and alumina formed, and this still further hardens the stone.—H. H. B. S.

Artificial and other Stone Roofing, and other Tiles, and Enamel; Impts. in the Manufacture and Treatment of, and in Apparatus therefor. G. F. Thomson, Wellingborough. Eng. Pat. 18,622, Aug. 21, 1897.

Blast-furnace slag, ashes, clay, sand, or other waste or cheap material is mixed with dilute sulphuric acid, and the mixture allowed to stand for some time. Solution of sodium hydrate, carbonate, or silicate is then added, and the whole stirred from time to time; or, in some cases, it is boiled with soda. The solution is then drained off, and the residue run into moulds to dry. The stone thus prepared is surface-hardened and whitened by immersion, first in dilute sulphuric acid, and afterwards in solution of silicate of soda or sodium carbonate, after which it may be polished. For imitating marbles, suitable colours are added to separate portions, which are then poured simultaneously into the moulds. The construction of a mould for casting slabs, &c. is also claimed. This consists of a frame with removable sides, and divided into partitions by plates fitting into grooves.—H. H. B. S.

Enamelling of Fireclay Baths and other Articles, Impts. in or connected with the. W. Cliff, Leeds. Eng. Pat. 16,528, Aug. 28, 1897.

Fireclay baths or other articles are enamelled on the inside with a hard glaze, and, after firing, are coated on the outside with a softer glaze or spirit varnish, which is then hardened by stoving at a much lower temperature. The advantages claimed are, first, that the glaze and the bath are less liable to crack, and secondly, that by glazing in this way, baths can be decorated in any desired tint, which is not always possible when a hard glaze is used on the outside.—H. H. B. S.

Cement, Slurry, Liquid Clay, and other Substances in like Condition, Improved Means applicable for Use in Drying. G. G. M. Hardingham, London. From F. D. Cummer, Cleveland, Ohio, U.S. Eng. Pat. 20,603, Sept. 17, 1896.

The process of drying slurry is apt to be troublesome, owing to clogging of the apparatus. The inventor surmounts this difficulty by adding a dry dust or powder (consisting of material of a character similar to that of the material of the slurry) in such quantity that the mixture does not adhere to the drying apparatus.—V. C.

Artificial Stone for Grinding or Abrading Purposes, Impts. in. C. C. Schirm and O. Lessing, Berlin, Germany. Eng. Pat. 11,630, Sept. 29, 1897.

Sand, pulverised quartz, emery, or similar substance, according to the purpose in view, is mixed with a flux of suitable hardness, such as glass-flux or fluorspar, and the whole heated to the fusing point of the flux. According to the quantity of flux used, the grains of the grinding agent can either be united at their points of contact only, or they can be entirely embedded; thus, the degree of porosity can be suited to the grinding requirements.—H. H. B. S.

X.—METALLURGY.

Steel, The Diffusion of Sulphides through. E. D. Campbell. Iron and Steel Institute, Autumn Meeting, 1897.

The author describes his recent experiments (this Journal, 1896, 906) on the diffusion of sulphides of iron, copper, &c., through solid steel, from which he concluded that the diffusion was dependent upon the presence of oxysulphide of iron, which by its great mobility was able to penetrate the pores of the metal. In the oxysulphide prepared for those experiments the ratio of FeS to FeO was practically 4:3, and the diffusion was complete. An oxysulphide having a crystalline structure has now been prepared, having the same ratio of FeS to FeO, by mixing ordinary sulphide of iron containing 26·3 per cent. of sulphur with 40 per cent. of its weight of precipitated magnetic oxide of iron, melting, and heating for 10 minutes in a porcelain crucible. On placing some of the crystalline pieces in the hole in the steel and heating as in the previous experiments, it was found that practically no diffusion took place, the oxysulphide simply having melted. Other attempts to make a pure diffusing oxysulphide resulted in the production of over 40 different specimens, none of which diffused completely as the original oxysulphide of the early experiments did, although some of them partly diffused. The conditions, whether chemical or physical, which govern the diffusibility or otherwise of the oxysulphide are not determined.—A. W.

Carbon and Iron. E. H. Saniter. Iron and Steel Institute, Autumn Meeting, 1897.

The experiments are considered under four headings:—

(1.) *The thermal treatment of Fe_3C.*—When Fe_3C is heated to 800° C. and slowly cooled, or to 1,000° and chilled in water, the molecular ratio of combined carbon to iron remains practically unaltered, and very little graphite is formed. When heated to 1,000° and slowly cooled much graphite is formed, whilst when fused in magnesia in an atmosphere of carbon monoxide to about 1,100°, and slowly cooled, there is a loss of total carbon, and the greater portion of what remains is graphitic.

(2.) *The saturation point of iron with carbon by fusion in contact with excess of carbon.*—The presence of silicon increases the formation of graphitic carbon in saturated carbon-iron compounds, and endeavours were therefore made to obtain a carbide free from silicon. The saturation point obtained was 4·81 per cent. of carbon in an iron containing 0·04 per cent. of silicon, which latter can hardly reduce the saturation point to any great extent.

(3.) *The saturation point of iron with carbon by heating without fusion in contact with excess of carbon.*—Pure iron wire was heated to about 900° C. in a porcelain tube full of charcoal for three periods of seven hours each. The percentages of carbon absorbed in each period were 1·64, 1·15, and 0·16, and the saturation point is therefore put down at 2·95 per cent., of which amount 0·53 per cent. was graphitic.

(4.) *The etching of pure carbon-iron alloys at a red heat, in order to ascertain their structure at that temperature by means of the microscope.*—The steel was polished as usual and etched by dropping it into fused calcium chloride kept at a bright red heat in a platinum crucible. After the steel had attained this temperature the whole was heated for a further 15 seconds, and then cooled by dipping into cold water. The steel was washed with water and dried in alcohol. It would appear that the cementite is first dissolved bodily until a certain amount of oxide of iron is floating in the fused chloride. The carbon then begins to be removed without further solution of iron, probably by the action of the floating oxide. When the carbon is nearly all removed the iron is again rapidly dissolved. The etching was stopped before the end of the first period, *i.e.*, while both carbon and iron were being dissolved. The appearance of acicular crystals in some of the plates examined, is considered an indication of a definite carbide of iron existing in steel at a bright red heat, and if the carbon is retained by chilling in the form in which it exists at this temperature, these crystals would appear to be the hardening carbide. In its bearing upon Osmond's theory of β-iron, the author thinks that the crystals do not support it while at the same time they do not disprove it. If the acicular structure were an indication of allotropic hard iron it would exist with 0·1 per cent. carbon-steel as with 1·8 per cent., but it is entirely absent. Iron containing 1·0 per cent. of carbon has a totally different structure at a bright red heat to iron containing 0·1 per cent. of carbon, and the same difference exists in corresponding chilled steels, which differences must be due to carbon, and which may be considered as evidence in favour of the hardening of steel being due to a carbide of iron of great hardness.—A. W.

Waste Acid from Metal Works: Report to the Sanitary Committee of the Worcestershire County Council, by Dr. G. H. Fosbroke, Medical Officer of Health. Public Health Engineer, Oct. 1897, 415—417.

1. Introduction.

2. Explains that the "acid" above referred to is a trade effluent from galvanising works, and is used in the process of covering iron with a coating of zinc. It is applied to sheet-iron, iron wire and netting, iron pails, tubes, &c. At large works the sheets are rolled, and the wire is drawn as well as galvanised.

3. The preparation of the iron consists in cleaning the surface by dipping the articles into an acid bath. This is called "pickling," and the acid bath is technically called "pickle."

4. The pickle usually consists of muriatic (hydrochloric) acid properly diluted, and is generally used cold. At some works, dilute, hot sulphuric acid is employed. On being removed from the acid the articles are allowed to drain, and then washed with water, sometimes containing a little lime; this water is technically known as "wash water" as distinct from "pickle."

5. The object of thus pickling iron is to remove the oxide on its surface, and to cause the metal to alloy with the commercial zinc, known as "spelter."

6. Thus the pickling process may give rise to a waste pickle containing either ferrous chloride or ferrous sulphate.

7. Hydrochloric acid is preferred by the great majority of Worcestershire manufacturers, not only because it is cheaper than sulphuric, bulk for bulk, but also because the latter attacks the metal too quickly.

8. After use the waste pickle often contains about one-third of the acid added at the commencement of the process.

9. Many works have been visited in various counties and many medical officers of health and analytical chemists have been communicated with, in order to ascertain "the extent to which acid turned into streams is injurious to health"; and it was found that there is a general consensus of opinion among such officials that there is no evidence to support the view that "acid turned into streams is injurious to health."

10. Through the courtesy of the 17 manufacturers in the galvanising trade in Worcestershire, it is ascertained that at the present time they employ about 3,500 men, and utilise 1,053 carboys (cwt.) of acid (corresponding to some 3,000 cwt. or 27,000 galls. of waste pickle) on an average every week.

11. Comparatively few manufacturers in this country treat their "waste pickle"; some, however, tip it on to cinder heaps, or throw it down disused pit shafts, while others discharge it directly into the River Stour, which from time to time consequently manifests a characteristic ochreous hue.

12. With regard to the effect of pickle in its crude state upon sewers and sewage, it is found that brick sewers at Birmingham have fallen in at two points where such waste liquor has been discharged for a length of time, owing to the action of the acid upon the mortar, and that the Wolverhampton sewers have also been injured.

14. Mr. Jones, county analyst for Staffordshire, states that at Wolverhampton the enormous quantity of iron in the pickle which reached the sewers at a time when the sewage was dealt with by irrigation alone, greatly interfered with the sewage treatment; coming, as it did, with intermittent rushes, the sewage was at times so loaded with iron in solution as to be quite poisonous to vegetation. To counteract this Mr. Jones advocated lime treatment of the sewage at the farm, but it having been found difficult to adjust, owing to the intermittent rushes of acid, the Corporation took steps to prevent the manufacturers putting untreated pickle into the sewers. On the other hand, Mr. Jones is of opinion that, provided muriatic pickle be properly treated before being added to the sewage, the sewers cannot be injured, and the sewage itself is improved.

16. Where sulphuric acid is used, the pickle is not entirely a waste product, as manufacturers get some return by recovering the sulphate of iron. The process consists in passing the hot pickle into wooden troughs (lined with sheet lead) containing pieces of board, upon which the sulphate of iron crystallises as the pickle cools; the crystals are collected for sale, and the effluent is then run off.

17. Mr. Ernest P. Peyton, in his pamphlet on "Pickling or cleaning Iron, with recovery of Sulphate of Iron," writes:

The hot pickling solution passes slowly along the (crystallising) channel, cools on its way, and a large part of the sulphate of iron it contains is there deposited as crystals.

The cold mother-liquor containing the unexhausted sulphuric acid overflows from the crystallising channel, and is conducted and returned to the pickling vat.

18. The value of such crystals (ferrous sulphate) varies, according to quality and local demands, from about 12*s.* to 20*s.* per ton. Commercial sulphuric acid (common brown) is at the present time about 4*l.* per ton; and as 22 carboys (cwt.) of sulphuric acid should yield about 5 tons of crystals, it is obvious that the recovery of crystals would (at the rate of 15*s.* per ton) give a return of 3*l.* 15*s.* as compared with an outlay of 1*l.* 8*s.* for acid. For this reason, and on account of the facility with which sulphuric acid waste can be dealt with, it has been proposed to substitute sulphuric acid for hydrochloric in galvanising.

19. But in some cases it is merely necessary to cleanse the surface of the metal, whilst in others a scale of oxide of iron, formed by exposure to very high temperature, has to be removed, and in this latter case, the use of hydrochloric acid is imperative.

20. Where hydrochloric acid is used, in but one process is any claim made that treatment of the pickle is attended with financial success.

21. This is "Turner's process." Turner patented it, and subsequently disposed of the patent rights to Messrs. Walker Bros., Walsall, who have worked it for the past seven years, and are the only manufacturers who now carry it out.

22. In "Turner's process" the acid liquor is made to flow slowly on the heated bed of a reverberatory furnace with access to air, when it is decomposed, with liberation of hydrochloric acid and formation of ferric oxide, the latter of which can be used as a colour, or as "fettling" in furnaces. The hydrochloric acid is condensed in towers, collected, and used over again in the pickling vats. The district medical officer where this process is being tried states that some effluvium nuisance results therefrom.

23. The working account for 1896, issued by Messrs. Walker, shows a favourable balance; in this statement, however, the "oxide recovered" is quoted at 4*s.* per ton, whereas it is understood that in other localities this product is of little or no value.

27. It appears that Messrs. Walker's two furnaces evaporate about 25 tons per week. It is found, however, that of the 17 manufacturers in Worcestershire only two use more than 3 tons per week; consequently, as very small furnaces would have to be put down, there is little doubt that these would scarcely pay. Furthermore, if the pickle were collected from the various works and conveyed to a central furnace, the cost of carriage would no doubt preclude financial success.

28. Although "Turner's process" has been known for some years past, and many manufacturers in the country have been required to deal with their "pickle," still it has not been generally adopted.

29. The simplest and most effectual means available for preventing the pollution of streams, or injury to sewers, appears to be to neutralise the spent pickle at the works with an alkali, which disposes of the free acid and precipitates the oxide of iron. Lime is generally preferred for this purpose, owing to the facility with which it can be obtained at moderate cost. One firm in Yorkshire contemplates neutralising the waste pickle with soda, and another with ammonia, but in neither instance is such treatment yet in operation, although it will probably be so in a few weeks.

30. The lime method consists in adding best lime (in the form of "milk") to the waste pickle (collected in underground tanks) until there is a slight alkaline reaction, and then thoroughly mixing. This mixture is then allowed to settle, and subsequently the supernatant liquor is run off, and the sludge pumped up and passed through filter presses.

31. If the neutralisation be properly managed, the oxide of iron sludge will be deposited, and the effluent liquor will neither contain acid nor alkali in excess, and be fit to discharge into streams or sewers.

32. The pressed sludge has then to be removed, and this is a matter of no small cost to manufacturers who carry on large works in populous localities.

33. In attempting to ascertain particulars of what the cost of such treatment is, it was found that, with one exception, those who use it keep no such records.

34. Messrs. Johnson (Ambergate, Derbyshire) at one time sold their pressed sludge at 4*s.* per ton, for mixing with sawdust to purify gas; but the demand is limited and fast decreasing. In fact, there is no doubt the lime process cannot be made to pay, although it enables manufacturers, often at considerable cost, to render their waste pickle fit to be discharged into streams or sewers, without detriment to anybody or anything.

35. Mr. Adolphe Crosbie (Wolverhampton) states that the question of cost of treatment is one very much dependent upon the particular conditions existent, and upon the quantity that may require to be dealt with. The writer has personally dealt at one time with as large a quantity as 50,000 galls. per week, and found no difficulty in turning out an effluent free from iron or acidity. He would much prefer to advise definitely on any particular case submitted, but, generally, he estimates the cost of treating a quantity of waste hydrochloric acid amounting to not less than 20 tons (say, 3,600 galls.) weekly, at 3*s.* 4*d.* (three shillings and fourpence) per ton. He would estimate the cost of a suitable plant for this work, exclusive of any buildings, steam pressure, gas, water, or drains, all of which would probably be available without much extra cost, at 350*l.* 3*s.* If the quantity treated be greater, the cost per ton would be less, whilst the cost of the plant would not increase in anything like equal proportion to the quantity. In regard to smaller uses of hydrochloric acid, producing, say, 4 tons (720 galls.) weekly, the cost of treatment would be about 5*s.* 6*d.* (five shillings and sixpence) per ton, and the cost of a somewhat crude plant would be about 60*l.* In either of the above cases there would be a solid residue amounting to about 7½ cwt. for each ton of liquor treated.

36. No allowance seems to be made in this statement for carting away the sludge, which, as previously stated, in some instances would be a matter of no small cost.

37. Similar conclusions as to cost of treatment were arrived at by Mr. Jones, of Wolverhampton, in 1889.

38. Some idea of what such expense would mean to local manufacturers can be formed when it is stated that on the basis of Mr. Crosbie's figures, and from information in the writer's possession, it would cost one local manufacturer 728*l.*

per annum to treat his pickle, exclusive of first cost and wear and tear of plant, in addition to which 1,560 tons of sludge would have been carted away every year.

39. In connection with facilities for treating pickle, it may be stated that at the meeting held on the 25th July 1896, the following opinion was expressed, and was subsequently approved by the council, *viz.*:

"That a local authority should, as far as possible, afford facilities to manufacturers for dealing with pickle without polluting the rivers and watercourses, inasmuch as it is of importance that local trade should not be unnecessarily hampered.

40. Manufacturers as a rule are not only large ratepayers, but also large employers of labour; therefore there can be no doubt that local authorities should, while protecting the general interest of the inhabitants of their districts, afford such traders every facility for the development of local industries.

41. In the case of "Peebles *v.* The Oswaldtwistle Urban District Council," Mr. Justice Charles gave judgment to the effect that a local authority could be compelled, at the instance of a manufacturer, to make such sewers as are necessary for carrying off effluents from factories within their district. This decision was, however, reversed "on appeal," when it was decided that the proper remedy for any failure of duty on the part of the local authority would be by complaint, under section 299 of the Public Health Act, 1875, that the authority were in default in such respect. This "case" has since been taken to the House of Lords, whose decision is anxiously awaited.

42. Bearing in mind the present unsettled state of the law, and the advisability of the district authorities in the Stour Valley affording facilities for the development of local manufactories, it seems to me desirable that the authorities should offer the manufacturers terms for passing their pickle into their sewers. If the pickle be neutralised in the manner suggested, and stored in duplicate tanks until it could be discharged into the sewers by a duly authorised person, the local authorities could easily satisfy themselves that neither damage to sewers nor to sewage disposal works would result.

43. On the other hand, as it would be more costly, if not impossible, for some manufacturers in that locality to put down the plant necessary for effective neutralisation, in my opinion the district authorities should also take into consideration how far they can assist such manufacturers in their difficulty, and arrange for untreated pickle to be received into the sewers, provided of course that no objectionable gases were thereby evolved, and suitable payments for such accommodation were made.

44. Whereas in Wolverhampton the corporation took steps to prevent the local manufacturers from passing untreated pickle into their sewers, in Birmingham, where very large volumes of waste acid are produced, no such action is taken, and the only recognised damage to the sewers which has resulted has been that on two occasions some brickwork fell in.

45. It is not suggested that the discharges of the pickle should be haphazard, for the writer thinks it should be so controlled that rushes of acid may be prevented: and as to this there should not be much difficulty, for the time when pickle requires to be got rid of can be anticipated by the manufacturers, and consequently arranged for by the district officials.

46. In order to obviate difficulty as to sewage disposal, if such procedure were countenanced, the discharge of the pickle should be under the control of some duly authorised person, who should make arrangements for the pickle to reach the sewers when the volume of sewage is largest (in the morning, for instance), and in something like equal quantities from day to day.

47. Grouping of the various works for the purposes of pickle discharge would effect this latter desideratum.

48. Should the sewers in the Stour Valley district be damaged, the terms arranged between the manufacturers who care to make use of the sewers and the local authorities could be such that the ratepayers would not suffer.

Cowper-Coles Gold Recovery Process. Engineer, **84**, 1897, 273.

The principal feature of the Cowper-Coles gold recovery process is an aluminium cathode, by which, it is said, one of the chief difficulties appertaining to the economical recovery of gold from weak cyanide solutions may be overcome, by electrolysis. In the Siemens Halske process lead foil or lead strips are used, the cutting up of which is a tedious matter. If several sheets are cut superimposed, the strips cling together, and have to be carefully spread out one by one, so that the surface may be exposed to the solution. A clean-up is made every seven to eight weeks, when the wood frames carrying the lead strips are withdrawn, the lead strips removed, and new substituted, the auriferous lead being ultimately melted down and conveyed to a central works to be cupelled. This method is both crude and expensive, the labour of fitting the strips into the frames is considerable, and the consumption of lead is a large item, having been computed by Mr. von Gernet, at the Worcester mine, at 1·10 of a penny per ton of ore treated. The Cowper-Coles process overcomes these difficulties, aluminium plates being substituted for the lead foil, advantage being taken of the fact that a loose oxide is formed on the surface of the aluminium, which enables the deposited gold to be readily stripped or wiped from the cathodes as pure gold. Gold by this process has been successfully extracted from cyanide solutions containing only 0·01 per cent. of cyanide of potassium, $2\frac{1}{3}$ dwt. of gold to the ton of solution. The substitution of aluminium for lead foil, or strip, enables the gold to be obtained pure, and also daily returns to be made of the amount of gold recovered; it also has the additional advantage of reducing the cost of labour, and economising the amount of cyanide of potassium used, as the solution is not contaminated by any base metals, such as zinc, as in the other processes.

Roasting previous to Cyanide Treatment. W. Macgregor. Eng. and Mining J. 1897, **64**, [7], 187.

The majority of ores are rendered more amenable to cyanide treatment, and in many cases with a smaller consumption of cyanide, by first roasting them. In this way, the cyanide solution is kept freer from soluble salts than when raw ores are treated; a very high percentage of gold may be extracted from sulphide ores by the cyanide solution; a greater extraction of gold, with an equal or less consumption of cyanide, is obtained than with raw ore; low-grade silicious ores, carrying the gold in iron pyrites, are rendered amenable to cyanide treatment without previous concentration; and clayey ores lose their water of hydration and become porous, and the subsequent filtration becomes much more rapid. The most important condition for good extractions and low consumption of cyanide on a large scale is to have dead-roasted ore. Sulphide ores, after being only partially roasted, contain sulphates of iron, and these consume a large quantity of cyanide with formation of double salts. Sulphide ores, containing from 2—10 per cent. of pyrites, which will not give more than 20 per cent. extraction of gold in the raw state, will often give 85—98 per cent. after a dead roast and treatment with cyanide. Mixed oxidised and sulphide ores often have an acid reaction, but on roasting, the acidity is destroyed, and the gold in the sulphide ores is rendered more amenable to the action of the cyanide, whilst a higher percentage of gold from the part that was originally oxidised, is frequently saved; the destruction of the acid quality of the ores does away with subsequent lime or soda-ash treatment, which causes trouble in percolation and in the zinc boxes. Very low grade ores and tailings do not pay for roasting, and there are some heavy sulphuretted ores and concentrates where roasting interferes with the extraction of the gold, and causes a very heavy consumption of cyanide. The advisability of previous roasting should be carefully determined in each case, both by laboratory experiments and by small plant tests. The following method is suggested for determining whether the ore is "dead-roasted".—100—250 grms. of the roasted ore are taken from the discharge end of the furnace; the ore is cooled, shaken in a beaker with about 200 c.c. of water, filtered, and to the filtrate is slowly added a small quantity of cyanide solution made up

to the same strength as that used in the regular work of the plant. If no cloudiness at all appear, the ore is dead-roasted, or at least well fitted for treatment by cyanide solution, and the consumption of cyanide will be normal. If a brown coloration be formed, there are some soluble salts of iron left in the ore, which will cause a greater consumption of cyanide, and may lead to a precipitation of compounds of ferrocyanide in the zinc boxes. If there is formed a blue colour changing to a greenish-blue precipitate, the ore is very badly roasted, and there will be a high consumption of cyanide, and the circulating solution will be made foul.—A. S.

Cyanide Process [Gold]; Sump Solutions, Extractor-Box Work, and Cleaning up in; Notes on. A. James. Inst. Mining and Met., 1897, 8 pp.

Experiments were made to test the relative gold-extraction efficiency of fresh and of used cyanide solutions, in which foreign matter had gradually accumulated. It was found invariably that the sump (or old) solution was inferior, giving an extraction of 90 to 95 per cent. of that obtained with the fresh cyanide, with ores ranging from 4 oz. to 21 oz. per ton. The use of lime brought the efficiency up to the normal with pure quartz ores or tailings, but not with those containing any appreciable proportion of sulphides. The addition of sodium sulphide, followed by an excess of a soluble lead salt (acetate or chloride), was successfully tried, time being given for the subsidence of any precipitated sulphides. In this way, the necessity of running foul sump solutions to waste may be avoided when water is scarce, or the cyanide or gold contents are high. It has been thought by some that, in practice, the double cyanide of zinc in the presence of potassium hydroxide would break up into KCy, K_2ZnO_2, and water; but in reality the reverse takes place, with the result that, when free zinc oxide is present, some of the cyanide added to bring up the solution to the working strength is taken up, without any indication of this being given by the silver test, so that the solution becomes less effective for the solution of gold than the cyanide alone would have been. This is a point in favour of the electrical deposition process, for which, however, insoluble anodes are much needed. Accumulations of zinc in the solutions from the old process may be avoided by the use of sodium sulphide solution, as above described.

Losses in Cleaning up.—The methods adopted vary greatly, and it was hoped that it would be possible to find out the causes of loss in each case, and to discover the best method of working. By shaking the gold slimes off the zinc, drying and mixing well, it was found that the losses varied from 0·5 to 6 per cent.; the less the handling of the slimes, the less commonly being the loss. The heaviest losses were incurred in roasting (in proportion to the stirring and handling), the least loss observed on this account being 0·23 per cent. in addition to those resulting from the rest of the treatment. With the nitre method of roasting (this Journal, 1897, 3), the loss was 2·57, which might, however, be reduced with practice. With acid treatment, the loss varied with the nature of the acid, nitric acid causing a greater loss than sulphuric or hydrochloric acid. With the ordinary impure zinc, however, the resulting bullion was purest when nitric acid was employed. With zinc free from lead, the best bullion and the least loss followed the use of sulphuric acid. When the slimes had been passed through a 40-mesh sieve, the bullion obtained by direct melting was fairly pure, and the results were as good as with acid treatment; whilst with scrap and coarse zinc included, the latter method was more advantageous. The lowest total loss obtained was 0·75 per cent., and this was with the sulphuric acid treatment prior to melting. One experiment with lead-free zinc gave a total loss of 0·52 by direct fusion of the sieved slimes, the coarse zinc being treated with sulphuric acid, and the residue being added to the slimes. Pure zinc always gave a loss (averaging) 0·2 per cent. less than that observed with ordinary zinc. Treatment of the slimes with strong solutions of alkali or cyanide (with or without acid treatment subsequently) gave higher losses. Considering the loss in simple melting with borax as x, it was found that the losses were as follows:—

	Per Cent.
Roasting, followed by melting	$x + 0·23$
Sulphuric acid, followed by melting	$x + 0·17$
Sulphuric acid, roasting, then melting	$x + 0·40$
Nitric acid, followed by melting	$x + 0·29$

With pure lead-free zinc, x was 0·43 per cent.; with ordinary zinc it exceeded 0·63. With the former, the roasted bullion was more base than that melted direct with borax. With ordinary zinc the reverse was the case, while the loss by acid treatment was greater, and that by roasting less, than with lead-free zinc. Possibly, the presence of lead, producing fluid lead oxide during roasting, may account for this smaller loss.

From these experiments, it is suggested that roasting be avoided, that the material should be handled as little as possible, and that the number of vessels should be limited. The zinc boxes should discharge their slimes by steel launders through a 40-mesh into small steel vessels with perforated bottoms covered with a filter-cloth. Each of these vessels fits into a small vat, and is so arranged that the overflow of the slimes passes into a large vat, which, with the aid of a filter with a vacuum receiver, collects all the overflowed light slimes from the steel vessels. The slimes are then at once sent to the drying and mixing plate; or the cake of slimes from the steel vessel is emptied into the acid vat, which may be of lead-lined wood, of specially varnished steel, or of aluminium, the latter not being attacked by sulphuric or even nitric acid. The slimes, after acid treatment, are washed with hot water and filter-pressed, run into a detachable steel-lined filter vat with vacuum connection, washed, vacuum-dried, fluxed, and melted. The latter process is preferred to filter-pressing, as being cleaner, simpler, and less liable to cause loss.

Purity of Gold Bullion.—At first it was thought that the lead was necessary to cause the precipitation of the gold with sufficient energy, but it is now found that the presence of a minute amount of iron is enough to produce the desired effect. By adopting a special lead-free zinc for this purpose a purer bullion is obtained; the refiners' fine, ranging from 3*d.* to 1*s.* 8*d.* (and averaging 9*d.*) per oz. of fine gold when lead is present in the bullion, may thus be obviated, whilst the bulk of slimes to be treated will be less, and the loss as above during clean-up will also be reduced by 0·2 per cent. On the other hand, the cost of the zinc will be higher; but, remembering that about 1 lb. of zinc is consumed per oz. of fine gold, there may be a difference of 11*d.* in the prices of the two varieties of zinc, with still a margin in favour of the better quality.—W. G. M.

Gold, Solution of, in Accumulated and Other Slimes. W. A. Caldecott. Proc. Chem. and Metall. Soc. of S. Africa, July 17, 1897, 18—22.

Pyritous ores which have been settled in dams or pits are liable to contain ferrous sulphide, and this imparts a grey or black colour to the material, 0·25 per cent. of FeS being often found in the more pyritous layers, with practically no ferrous salts or free acids. This and ferrous oxide both absorb the free oxygen from the solutions, and it is necessary either to pump in air or to use an oxidising agent, $\frac{1}{8}$ to $\frac{1}{2}$ lb. of potassium permanganate per ton of dry slimes being a suitable addition. All plants now under construction for the treatment of accumulated slimes will be equipped with air-compressors capable of delivering 1,800 cb. ft. of air per min. Under these conditions the vats should be deep and the bubbles of air as small as possible, in order to secure the maximum aëration; it is also advisable to supply the air as rapidly as possible, because a given volume of it is required for any given proportion of deoxidising agent, independently of the time occupied in applying it. The addition of the lime to old and acid slimes, precipitates ferrous oxide, which absorbs oxygen and adds to the necessity for preliminary aëration. By such treatment the consumption of cyanide has been reduced by one-half (to $\frac{1}{3}$ lb. of KCy per ton of dry slimes). The aëration is effected by means of perforated pipes at the bottom of the tanks, and lasts (with agitation) for from 2 to 20 hours, according to the proportion of ferrous sulphide. After complete oxidation of sulphides, the treat-

ment is continued for two hours, the cyanide solution is added, and aeration proceeds again for from 2 to 5 hours. Lime is added during the charging and preliminary aeration, in sufficient quantity to yield an alkali strength of liquid corresponding to from 0·006 to 0·01 per cent.

In the laboratory, the percentage of dry slimes, or the ratio of slimes to solution, in any pulp may be determined with fair accuracy from its sp. gr. x, by applying the following formulæ:—

$$\text{Percentage of dry slimes in pulp} = \frac{158\cdot8\,(x-1)}{x}$$

$$\text{Slimes : Solution} = (x-1) : (1-0\cdot37\,x).$$

All tests to determine gold extraction must be made on the moist material, as the process of drying, causes an oxidation of the reducing substances present. Alkali tests are made by Clennell's method:—100 c.c. of solution are taken, the cyanide strength is determined by standard silver nitrate solution (1 c.c. of $AgNO_3$ sol. = 0·01 per cent. of KCy), one or two excess drops of $AgNO_3$ solution are then added, a few drops of a 0·5 per cent. solution of phenolphthalein are added, and the titration is effected with decinormal sulphuric or oxalic acid.—W. G. M.

Bullion, Low-grade: Plant for Parting. F. Gutzkow. Eng. and Min. J. 1897, 218.

Two flat-bottomed cast-iron pans 4 ft. square are used, one of which is 2 ft., the other only 15 in. deep; the larger kettle is mounted over a fireplace, whilst the smaller is heated by the waste heat from the other. Both are covered with wooden chambers provided with vertically sliding doors, the former being lead-lined and having a leaden flue with a good draught. The pans might be round-bottomed, but square and flat bottoms are more economical of space and are more easily manipulated. The bullion is cast in slabs 12 in. square with 2-in. holes in them; 20 or more of these are suspended by S-shaped iron hooks from lead-sheathed iron rods, which rest on a lead-covered iron frame 3 ft. square, supported by four vertical iron rods (also enclosed in leaden pipes) from the roof of the chamber. These are secured by screws to a wooden frame resting upon the roof. The whole arrangement can be lifted by means of a windlass on the floor of the room. The acid is not allowed to touch the lead-covered iron rods: the suspending hooks dissolve rapidly, but are not costly to replace.

In this apparatus about 800 lb. of low-grade bullion (containing 25 per cent. of Ag and 75 per cent. of Cu) may be treated every 24 hours in about 20 slabs weighing from 40 to 50 lb. each. About 2,000 lb. of strong sulphuric acid will be consumed in the operation, but 6,000 lb. will be required in all, 2,000 lb. being placed in each pan, and the remainder in a sheet-iron reservoir (afterwards used as a crystallising pan), where it is diluted to 60° B. and forms the mother-liquor for the crystallisation of the copper sulphate. As the bullion dissolves in the first pan under the action of heat, the acid consumed, is replaced by the diluted acid from the reservoir, in order that the concentration of the liquid may not cause the crystallisation of the copper sulphate. After three hours, 100 lb. of the bullion should be dissolved, and one-eighth of the diluted acid should have been spent; the frame with the suspended plates is then raised sufficiently to allow of the deposited powder of copper sulphate crystals (about 1 cb. ft. in bulk) being raked to the corners nearest the second pan. A slide, which forms part of the partition between the two pans, is raised, and the powder is raked into the second pan. The partition is then lowered again, diluted acid is introduced, and the operation is continued for another period of three hours, and so on, until after eight of such periods the parting should be complete. The copper sulphate in the second pan is stirred in with the strong sulphuric acid, in order to free it from silver sulphate, and is then taken out for further treatment. The silver is ultimately concentrated in the first solution, which is then siphoned into the crystallising pan (provided with a lead-lined flue for the fume), and the first pan, being left empty, is filled up with acid siphoned from the second pan. When all fume from the first pan has ceased, the sliding doors are opened, pieces of sheet-iron are placed on the pan, the remnants of the bullion are removed, and the frame is charged with a new batch of bullion for a fresh run. The crystallising pan, which is still being used to work off the first batch, would then be required; it is therefore advisable to have two of these latter pans, which may be used alternately. Scraps of bullion too small to be suspended again are remelted. The hot silver solution in the crystallising pan is cooled by means of a jet of steam until it may be safely diluted down to 60 B., by means of about 500 lb. of water added in a small stream. The mixture is then allowed to cool, and the silver sulphate is gradually crystallised out in the ordinary way.—W. G. M.

Tin, Analysis of. E. Prost and A. Van de Casteele. Bull. Assoc. Belge des Chim. **11**, [3], 103.

See under XXIII., *page* 827.

Silver in Silver-plating Solutions, Estimation of. T. J. Baker. Chem. News, 1897, **76**, 167.

See under XXIII., *page* 828.

PATENTS.

Ferro-Sodium Fluxes, Impts. in; more particularly those adapted for use in the Manufacture of Iron and Steel. A. E. Tucker and T. V. Hughes, both of Birmingham. Eng. Pat. 14,222, June 26, 1896.

Oxide of iron, dry sodium carbonate, and dry powdered carbon, generally in the respective proportions of 100, 100, and 44 parts, are finely powdered, mixed, and made into blocks with a suitable binding material, such as 5 per cent. of tar. The blocks are dried at a low temperature and then heated sufficiently to reduce the oxides, but not to vaporise the sodium. The blocks of ferro-sodium so obtained are cooled, coated with paraffin wax to preserve them from the air, and are used chiefly to add to the bath of molten iron, after blowing, to remove the oxides therefrom.—A. W.

Metals, Impts. in and connected with the Alloying of. A. E. Tucker and T. V. Hughes, both of Birmingham. Eng. Pat. 14,223, June 26, 1896.

To prepare the alloying metal in a convenient form for admixture with the mother-metal, the oxide is mixed with carbon, and the two are ground together and made into blocks with tar or other binding material. These are slowly dried, and then heated until the oxide is reduced. The blocks of metal so obtained are protected from the air by coating with wax, and are added, when convenient, to the molten bath.
—A. W.

Gold Separating from other Materials, Impts. in Apparatus for. W. H. Hyatt, Herne Hill. Eng. Pat. 16,032, July 20, 1896.

This invention relates to improvements in detail in the apparatus described in specification, Eng. Pat. 24,782 of 1894; this Journal, 1896, 119.—J. H. C.

Metals [Gold, Copper], Improved Process for Extracting. W. P. Thompson, Liverpool. From A. Sinding-Larsen, Christiania, Norway. Eng. Pat. 22,355, Oct. 8, 1896.

To extract the metals from ores containing sulphur, the raw material is pulverised, washed if necessary, and exposed to and agitated in a current of chlorine, or other halogen gas, free from air. If the subsequent treatment is dry the ore is heated while the chlorine is passing, and the volatile chlorides are passed into a series of receivers heated to temperatures corresponding to the condensation points of the various chlorides, so that the latter become separated one from the other. The chlorides are then dissolved separately in water and electrolysed. By the wet process the chlorinated ore is leached with water, the solution electrolysed, and the chlorine recovered for further use. Metallic iron is used to precipitate the copper, if present in quantity, and the iron chloride solution so obtained is again electrolysed to recover the chlorine.—A. W.

Gold and other Precious Metals, Impts. in and relating to the Recovery of. T. G. Bowick, London. Eng. Pat. 15,569, June 30, 1897.

In preference to running the ore, pulp, or slime containing the gold over stationary surfaces, it is carried over a series

of revolving cylinders, tables, or endless bands, so as to avoid the friction between the ore and the amalgamated plates. The surfaces of the cylinders, &c., consist, preferably, of amalgamated copper, and may be connected, if necessary, with the poles of an electric battery.—A. W.

Copper, Metallic; Elimination of Arsenic from, Impts. in connection with. B. Thomas and J. W. Thomas, both of Llanelly. Eng. Pat. 17,911, Aug. 13, 1896.

The copper is melted in a reverberatory furnace, and is there subjected to the conjoint action of fluxes and air. For the former, soda ash is preferred as being cheap and effective, but sodium nitrate or a good black oxide of copper may be employed. About 1 to $1\frac{1}{4}$ cwt. of flux is commonly required for a charge of 10 to 12 tons of copper. The air is introduced at a pressure of 35 lb. to 40 lb. per sq. in. by means of ($\frac{3}{4}$ to 1 in.) iron piping protected by a silicious coat, and is delivered 3 or 4 ins. below the surface of the metal. The slag fuses and is removed from time to time; it contains 0·8—1 per cent. of Cu, which may be reduced to 0·25 per cent. by introducing it into the ore-smelting furnace. In some cases the copper may be removed from it by direct carbon-reduction. A special mixing machine is also described, by which the fluxes may be conveyed regularly by a screw into a hopper, through which they are carried on by and with the compressed air, and thus passed through the metal itself. By this process the arsenic in 10 or 14 tons of copper may be reduced from 1·25 to a mere trace in 12 hours. The copper may easily be brought up to 99·5 or 99·75 per cent., and even in the presence of bismuth the metal is tougher after treatment.
—W. G. M.

Aluminium Alloys, Impts. in the Means for and Method of producing certain kinds of Metal especially adapted for use in the Manufacture of the Tubes and other of the Component Parts of Cycles of all kinds, Autocars, and other Wheeled Vehicles, which Improvements are also applicable for other Industrial Purposes. W. Van Wart, F. W. Popp, and J. J. Bradley, Birmingham. Eng. Pat. 19,640, Sept. 5, 1896.

The mixture generally used consists of aluminium about 100 parts, copper 1 to 10, zinc $\frac{1}{4}$ to 6, tin $\frac{1}{2}$ to 4, silver $\frac{1}{4}$ to 6, phosphorus $\frac{1}{16}$ to 1. Sometimes the copper is omitted, from $\frac{1}{2}$ to 4 of regulus of antimony being substituted. In the first case the copper is all melted, then the zinc and silver are added, then the aluminium, and finally the tin and phosphorus. In the second case the silver is melted first, then the aluminium is added gradually, then the zinc and regulus of antimony, and finally the tin and phosphorus.
—J. H. C.

Steel or Iron, Annealing of; Impts. relating to the. D. C. Dalzell, Handsworth, and A. Fairley, Birmingham. Eng. Pat. 22,976, Oct. 19, 1896.

In the annealing of ("malleable") castings of iron or steel, the objects are coated over with a wash of red hæmatite ore, or with clay or lime, prior to placing them in the furnace, and the usual packing in hæmatite may then be dispensed with.—W. G. M.

Toughening or Strengthening of Metal, Impts. in or relating to the; particularly applicable to Steel Tubes and Brazed Joints. R. S. Lovelace, Henstrige, Somersetshire. Eng. Pat. 21,175, Sept. 24, 1896.

The hot metal is quenched in a mixture of linseed oil, borax, soda, and india-rubber.—F. H. L.

Blast Furnaces, Iron; Impts. in the Method of Working, so as to utilise the Gases evolved for producing Motive Power. B. H. Thwaite and F. L. Gardner, both of London. Eng. Pat. 23,597, Oct. 23, 1896.

This is an extension of the principle of Eng. Pat. 15,684 of 1896. The gases from blast furnaces may be utilised even at times when the furnace would ordinarily be shut down, by running it on fuel, sand, and limestone, whereby a vitreous material or slag would be produced (and no iron), and the plant depending upon the gas for its motive power would not be caused to stand idle. The value of the gas is indicated by burning a small quantity in a motor and testing with a speed-indicator. The hot gases are passed through a number of tubes, to cool them before purification, and their heat may thus be given up for the warming of the air for cold-blast furnaces. The air for hot-blast furnaces may be heated by alternating stoves; but instead of using the furnace gases for this purpose, it is preferable to employ the latter as motive power, and to burn a cheaper fuel to operate the stoves. In this case, if the blast furnaces should from some cause cease to work, a gas supply may still be obtained by employing that from the coarser fuel.
—W. G. M.

Alloys (Iron) [Non-Magnetic], Impts. in the Manufacture of. R. A. Hadfield, Sheffield. Eng. Pat. 24,703, Nov. 4, 1896.

Alloys suitable for dynamo brackets, or for similar purposes, are made by melting together iron and carbon (commonly about $3\frac{1}{2}$ per cent.) with other materials, to give a composition of 7—30 per cent. of Mn, and either $2\frac{1}{2}$—8 per cent. of Si, or 2—8 per cent. of Al, or $1\frac{1}{4}$—8 per cent. each of aluminium and silicon. Appropriate alloys may be made from No. 1 hæmatite pig, silicious pig, ferro-manganese, and aluminium to yield, *e.g.*, $3\frac{1}{4}$ per cent. of C, 13 per cent. of Mn, and 6 per cent. of Si; $3\frac{1}{2}$ per cent. of C, 13 per cent. of Mn, and $4\frac{3}{4}$ per cent. of Al; or $3\frac{1}{4}$ per cent. of C, 13 per cent. of Mn, 6 per cent. of Si, and 2 per cent. of Al. The metals may be melted together, or they may be melted separately and then mixed. The more manganese added, the less magnetic will be the alloy, but the more difficult it will be to change the carbon from the combined into the graphitic form, as is necessary.—W. G. M.

Steel, Manufacture of, by the Bessemer or Open-Hearth Process; Impts. in. C. H. Foote, W. R. Walker, E. A. S. Clarke, all of Chicago, U.S.A. Eng. Pat. 8763, April 6, 1897.

This process relates mainly to the making of the final additions of carbon and manganese to the decarburised steel. The manganese is added direct from the spiegel blast furnace without remelting in cupolas. Two small smelting furnaces are employed, the duplication being provided as a safeguard against a breakdown of any part of the furnace. The spiegel may thus be made of any required grade, and should be conveyed from the furnace to the steel plant in a ladle sufficiently large to hold one or more casts of the furnace, and thus to serve as a mixer; or it may be carried in small ladles to a mixer. It is then tapped from the ladle or mixer in suitable quantities for use. The spiegel in the mixer is diluted with pig-iron, or is mixed with irons of such compositions that material having exactly the required analysis may be used in the steel-bath. In this way the nature of the spiegel is under perfect control, and may be altered at a moment's notice. But as the smelting of high-grade spiegel is expensive, owing to the costly nature of the ores to be used, it is recommended that a metal be prepared with 8—12 per cent. of Mn in the miniature furnace, by using cheap low manganese ores, and that it be brought up to the required percentage by the addition of ferro-manganese to the metal in the mixer, either in the molten condition or (preferably) in pieces the size of a hickory nut, which have been heated to a red heat. These fragments are best added in the runner leading to the mixer, so that a thorough incorporation may be effected. The ladle used for conveying the spiegel from the furnace should be provided with an oil or a gas-burner, or the metal should be covered with charcoal or coke dust, to retain the heat; and after the addition of the higher-grade ferro-manganese, a bar of wood should be introduced into the bath to ensure thorough incorporation of the whole.
—W. G. M.

Metals (Copper Iron, &c.), Impts. in or relating to Uniting or Welding. J. Andrews, Allegheny, U.S.A. Eng. Pat. 8780, April 6, 1897.

A process for uniting copper or alloys thereof, to other metals, such as iron in plates, &c. The iron is first coated with a film of copper by cleaning and placing it in an acid solution of sulphate of copper, and is then clamped to the plate of copper to which it is to be welded. The whole is

heated, and the iron and copper plates, with the deposited copper between them, are united by pressure or by passing through rolls.—A. W.

Melting and Casting Metal, Impts. in or relating to Apparatus for use in. L. Rousseau, Paris. Eng. Pat. 9421, April 13, 1897. (Under Internat. Convention.)

These improvements have chiefly for their object a perfect combustion of gases from the fuel and utilisation of the heat produced; the air is admitted as much as possible in a direction opposite to that in which the gases travel. —J. H. C.

Composite Metal Plates, Impts. in the Manufacture of. H. Wachwitz, Nuremburg. Eng. Pat. 14,550, June 16, 1897.

Thin sheets of copper (about 0·1 mm.) are laid on aluminium sheets (of about 10 mm.), and heated between red hot plates, and then rolled or pressed. The composite sheets thus produced, can be worked like sheet copper, brazed, tinned, or galvanised.—J. H. C.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(A.)—ELECTRO-CHEMISTRY.

Colloids, Electric Migration of. Coehn. Zeits. für Elektrochem. 1897, **4**, 63—66.

In a former research, the author drew the conclusion from the fact that a coloured fluid followed the direction of the current during electrolysis, that coloured ions existed. A substance free to move, will move in or against the direction of the current if it be electrically charged. This is the case with the ions during electrolysis, as shown by a lecture experiment of Nernst's, in which the MnO_4 ions, during electrolysis of potassium permanganate, move towards the anode; it is also true of fine suspended solid particles in water or in turpentine, their static charges carrying them towards the oppositely charged electrode, while the liquid particles move towards the other one.

The author and Euler have found that colloids—caramel, starch, and tannin—non-electrolytes, behave in a manner exactly similar to such solid particles, moving in water towards the anode; this fact has already been noticed in the case of tannin, and is the foundation of the process of electrical tanning.

Colloids in which electrolytic dissociation might possibly take place—ferric hydroxide, for example—have also been investigated. Dialysed ferric hydroxide had the last traces of chlorine removed from it by electrolysis with a silver anode, and was then submitted to the current between two layers of water. A dark layer moved from the anode, while a more rapid movement of a lighter-coloured layer took place towards the cathode. This layer would not pass a parchment membrane, and hence could not contain iron ions. The different behaviour of these electrolysable colloids from the others is being further investigated.—J. T. D.

PATENTS.

Acid-Proof, Electrical Non-conducting Articles, and Composition therefor; Impts. in. W. Morison, Montclair, New Jersey, U.S.A. Eng. Pat. 18,485, Aug. 20, 1896.

This composition for a new non-conducting acid-proof and acid-tight battery case, consists of asphaltum, gutta-percha, asbestos, and sulphur mixed and beaten together at a temperature of about 60° C. It can be readily moulded, and, possessing a polished surface, does not require any further treatment.—F. H. L.

Secondary Battery [Lead] Plates, Impts. in Active Material for. The Sussmann Electric Miners' Lamp Co., Ltd., and S. A. Rosenthal, London. Eng. Pat. 21,749, Oct. 1, 1896.

"Active material, for application to a lead conductor to form a secondary battery plate, consisting of a finely powdered lead oxide made into a stiff paste by intimate mixture with a thin india-rubber solution, sulphate of ammonium solution, and, if desired, powdered inert material [asbestos, slag wool] . . . ."—J. C. R.

Electric Currents, Controlling the Direction of Flow of, or Retarding such Flow, and for like Purposes; An Improved Cell for. [Aluminium Anodes.] W. P. Thompson, London. From Dr. Leo Gratz, Munich, Germany. Eng. Pat. 23,084, Oct. 17, 1896.

This invention has for its object a cell for controlling the direction of flow of electric currents, or retarding such flow, and for like purposes, and is based on the property possessed by an aluminium anode of strongly diminishing the flow of current in an electrolyte, which separates oxygen at the anode directly, or by means of secondary processes.

"This property of aluminium has been attributed to the formation of a very bad conducting layer on the anode, that is to say, of a resistance; but the correct explanation should be sought in the production of a kind of polarisation of very considerable extent, or of a condenser action; and measurements undertaken for this object have shown that the counter force resulting thereby amounts to 22—25 volts."

Claims: (1) "Cells having different resistances for the two directions of current flowing through them (choking cells), consisting of an electrode of aluminium or aluminium alloy arranged in an electrolyte which separates oxygen at the aluminium as anode, directly or by secondary processes (for example, alum solutions, alkali sulphate, alkali nitrate, caustic alkali solutions, acids), in connection with either (*a*) another electrode of carbon or any suitable base or noble metal (with the exception of platinum), or of a metal alloy that is not soluble by chemical action in the liquid used; or (*b*) any desired electrode in an electrolyte (acids, salts solution) which is divided from the former by a diaphragm . . . ." Other (four) claims relate to applications of these "choking cells" for electrical purposes. —J. C. R.

Batteries, Electric Primary and Secondary; Impts. in. V. Jeanty, Paris. Eng. Pat. 12,827, May 24, 1897. (Under Internat. Convention (France), Nov. 19, 1896.)

The object is to separate the "depolarising liquid" from the "exciting liquid" without employing porous partitions. The electrodes are arranged horizontally in troughs, with the depolarising solution or oxide occupying the lower position.

Claims: (1) "In electrical batteries (primary or secondary) the provision of separate impervious baths of either circular or rectangular shape, arranged horizontally, and adapted to contain depolarising or any other suitable liquid, as well as one of the electrodes . . . ."—J. C. R.

Electric Accumulators [Lead: Active Material], Impts. in and relating to. I. Celestre, Syracuse, Italy. Eng. Pat. 26,491, Nov. 23, 1896.

(1) "The process of preparing active material for accumulator plates by means of simply sulphating oxides of lead . . . . . and with the complete exclusion of all foreign substances, whether as an adhesive material or as a reacting material." [" Oxides of lead, either alone or mixed with electrolytic lead, are treated with a solution of sulphuric acid; the mass is then worked into a paste with carbonate or sulphate of lead, the quantity of the latter ingredient varying according to the degree of porosity which the mass is desired to possess . . . ."] (2) "The use of carbonate or sulphate of lead in the preparation of this material, by means of which there may be obtained the degree of porosity desired." (3) "Frames for accumulator plates provided with curves of dilatation [so that the dilatation of the active material can take place without any hindrance] . . ."—J. C. R.

Wood and other Cellular or Fibrous Substances, Improved [Electro-Capillary] Process and Apparatus for Treating or Saturating with Liquids. L. A. Bretoneau and

A. L. C. Nodon, Paris, France. Eng. Pat. 13,242, May 28, 1897.

The apparatus consists of a vat of brickwork cemented internally, the lower portion of which contains the anode and the solution with which it is desired to impregnate the wood or other substance. The wood, which only dips to a depth of a few centimetres into the liquid, rests on a support of felt or other porous material, which covers a perforated lead anode supported by a wooden floor resting on blocks. The bottom of the tank is sloped and provided with an opening through which it may be emptied. A porous partition covers the wood and ensures contact between it and a porous vessel resting on it, filled with pure water, and on the bottom of which is the cathode, which is preferably an iron plate. This vessel is supplied with water, which is constantly withdrawn by means of a siphon. The current density employed should not exceed one-third of an ampère for every square decimetre of material treated.
—G. H. R.

Primary Batteries or Cells [*Amalgams*], *Impts. in.* I. Kitsée, Philadelphia, U.S.A. Eng. Pat. 14,508, June 15, 1897.

Relates to the construction of a battery having as electrodes an alloy of mercury and a metal, as zinc, a metallic rod surrounded with "a high oxide of lead," and dilute sulphuric acid as electrolyte. Both electrodes are surrounded or enveloped by some inert porous material or cups, as wood or plaster of Paris.—J. C. R.

Electric Batteries [*Manganese*], *Impts. in.* L. W. Pullen, Camden, New Jersey, U.S.A. Eng. Pat. 16,069, July 6, 1897.

An "electric battery having the cathode arranged in a depolarising mass, consisting of finely divided carbon [plumbago, coke], a salt of manganese [sulphate], and concentrated sulphuric acid uniformly distributed throughout said mixture."—J. C. R.

Electric Accumulators [*Lead*], *Impts. in or relating to the Electrodes of.* E. W. Jungner, Stockholm, Sweden. Eng. Pat. 16,362, July 10, 1897.

"In positive electrodes of electric accumulators, in which the active support consists of metallic lead or lead alloy, and the electrolyte of a suitable acid, the method of covering the surface of the metal, by mechanical, chemical, or electrical means, with a conducting compact coating of peroxide of lead, between the molecules of which a suitable material ['paraffin'] is introduced for the purpose of binding the same together . . ."—J. C. R.

New Galvanic Element [*Zinc - Carbon*]. C. D. Abel, London. From C. N. Sedneff, St. Petersburg. Eng. Pat. 17,026, July 19, 1897.

(1) "A galvanic element with 'circulating depolarisation,' characterised by a depolarising mass, which is composed in such a manner that the oxygen of the depolariser becomes continuously regenerated by the production of exchange operations . . . ." (2) "In the element specified in the first claim, the use of a diaphragm, which is only permeable to water and gases . . ."—J. C. R.

(*B.*)—ELECTRO-METALLURGY.

Slimes, Battery; Treatment of. J. R. Williams. Trans Chem. and Metall. Soc. of S. Africa, July 17, 1897, 14—18.

The first slimes-plant to be operated (that of the Crown Reef Gold Mining Co.) has been in use for 12 months, and, after several small troubles, is working successfully, having paid its way from the very first. The slimes may be passed through a large sand separator, but this is rarely required, as the proportion of sand leaving the tailings plant is negligeable; the liquid then runs through a launder, in which milk of lime is added to precipitate the slimes (in a flocculent form). The lime must be neither in excess or in insufficient quantity; and the introduction of an automatic lime feeder, invented by Seymour, has effected a saving at the rate of 1,200*l.* per annum in lime alone. The mixture passes through two settling pits, each 20 ft. by 20 ft., by 10 ft. deep, in which 80 to 90 per cent. of the slimes deposit, the overflow passing to another tank 40 ft. by 40 ft. by 10 ft., the effluent from which is practically clear. These pits come down to a point, forming an inverted pyramid, and are each connected with a centrifugal suction pump, which delivers the settled slimes into eight tanks for treatment, together with about 10 per cent. of the water originally contained in them, which cannot thus be separated. These tanks are 32 ft. in diameter, and 10 ft. deep, with conical bottoms, and are arranged in series of four. The first receives the sludge, which is allowed to settle in it, and the effluent is then drawn off. The sludge is then pumped into the second tank, a jet of cyanide solution being used to sluice it into the suction of the centrifugal pump, and the tank is then filled up with cyanide, the strength of the whole being made up to 0·01 per cent. of KCy. It is found that 80 per cent. of the gold is dissolved in passing thus through the pump; nevertheless the mixture is agitated by a circulation pump for two hours, and is then allowed to settle, and the solution is drawn off through a series of side-cocks (to which siphon pipes are preferable). The settled slimes are then pumped into a third tank, where they are agitated with a very weak solution of cyanide and allowed to settle, the resulting solution being used for the first treatment when transferring from the first to the second tanks. Then the residual slimes are finally pumped, together with still more dilute cyanide liquor, into the fourth tank, the liquid from this being used in transferring the material from the second to the third tank. The comparatively clear solution from the second tank is run into settling tanks 15 ft. in diameter and 5 ft. deep, and when perfectly clear is passed through the electrolytic precipitation boxes from below upwards. These boxes are 30 ft. long by 6 ft. wide by 4 ft. 9 ins. deep, and have iron and lead anodes; the effluent is used over again. In using this plant the gold tends to concentrate in the liquor, so that the solution passed to the precipitating vats assays 60 grs. The cost of treatment per ton is 3*s.* 9·05*d.*, which is made up as follows:—

	d.
Management and European wages	9·44
Kaffirs and food	3·29
Electric light	0·40
Power	2·75
Cyanide	5·51
Lime	6·19
Lead foil	1·75
Oil and waste	0·89
Assaying	1·26
Sundries	0·63
Royalty	4·77
Maintenance (labour, 4·43*d.*; stores, 3·47*d.*)	8·17

This estimate is based on two months' working (April and May), during which 6,640 tons were treated, with an assay value of 5·32 dwt., yielding 1,067 oz. of gold, with an actual extraction of 60·48 per cent., and giving a profit of 3,058*l.*

In regard to rate of precipitation by electrolysis, the result of an experiment by Butters, with a solution containing 2 dwt. 12 grs. of Au per ton, showed that the liquid, after successive periods of treatment of three hours each, assayed 1 dwt. 4 grs., 20, 12, 8, 4, 2, and 0·5 gr.; whilst after 24 hours only a trace of gold was left.—W. G. M.

Nickel, The Electrolytic Separation of, from Solutions of the Sulphates and Chlorides. F. Foerster. Zeits. f. Elektrochem. 4, 160—165.

The author describes experiments undertaken in order to judge of the possibilities of the successful production of nickel, by electrolytic methods upon an industrial scale of operation.

Electrolysis with Soluble Nickel Anodes.—This is the method used in electro-plating with nickel, but only very thin coatings of nickel have been obtained.

If the electrolysis be continued, the deposit becomes scaly in character, and small lamellæ break away from the surface of the electrode and curl up.

The author has found that this difficulty can be overcome by use of a hot electrolyte. At temperatures ranging from 50° to 90° C., tough deposits of nickel of any desired thickness may be obtained from either the sulphate or the chloride salt solution. The former is, however, to be preferred. The solution used contained 145 grms. of commercial nickel sulphate per litre. A thick plate of nickel was used as anode, and a thinner sheet of nickel as cathode. The nickel used contained from 1·5 to 2·0 per cent. of impurities, chiefly iron and cobalt, with smaller amounts of carbon, silicon, copper, and manganese. The anode was covered with parchment paper in order to maintain a clear electrolyte. This parchment covering was not attacked in the hot sulphate solution; but in the chloride solution it quickly rotted, and was dispensed with altogether in later experiments. Circulation of the electrolyte was maintained during the deposition, and apparatus was used to supply the deficiency in water or acid caused by the evaporation at the high temperatures used. Current densities are all expressed in ampères per sq. decimetre. (1 ampère per sq. decimetre = 9·24 ampères per sq. foot.) Using the nickel sulphate solution of the concentration named at temperatures of from 50° to 90° C., and current densities varying from 0·50 to 2·5 ampères, satisfactory and bright deposits of a light grey or tin-white colour were obtained. The deposits increased in lightness of colour and in smoothness, with increasing current density.

From a more concentrated solution of nickel sulphate (= 100 grms. nickel per litre) a current of 0·50 ampère density deposited the metal in a dull and rough state, whilst from the same solution a current of between 2 and 2·5 ampères in density yielded the metal in a bright silvery-white form at the cathode. An equally good deposit can be obtained from the solution first named with a current of only 1 ampère in density. Bubbles of hydrogen at the surface of the cathode are sometimes the cause of irregularities and roughness in the deposit. The preliminary experiments led to the performance of one on a larger scale, in which 0·50 kilo. nickel was deposited upon a cathode of 2 sq. decimetres superficial area. The solution used, contained 100 grms. of nickel per litre: the current density employed was 1·5—2·0 ampères; the temperature was maintained at 60° C.; and the E.M.F. required, with the electrodes at a distance of 4 cm., was between 1·0 and 1·3 volts. The nickel obtained was exceedingly tough, and the author concludes that, using hot solution, the electrolytic deposition of nickel upon any scale of operations is possible. An analysis of the nickel deposited in these experiments, showed that practically the whole of the iron and cobalt contained in the crude nickel had passed over to the cathode with the nickel. The author considers that in the sulphate solution no separation of these three metals is possible by alterations of the E.M.F. and current density employed. Such a separation is, however, possible according to Basse and Selve, and also to Vortmann, if the solution of the nickel salt be treated with tartaric acid and excess of sodium hydrate. The character of some of the alloys of nickel and iron obtained in the attempts to deposit nickel electrolytically from solution containing much iron, are described by the author.

In the experiments made with the nickel chloride salt solution, the electrolysis of the cold solution yielded similar deposits to those obtained from the sulphate solution. Heating the solution does not solve the difficulty, since a deposit of green basic chloride makes its appearance under these conditions at the cathode. This formation is prevented by the addition of 0·25 grm. of hydrochloric acid per 100 c.c. of fluid to the electrolyte. On account of the loss that occurs during the electrolysis of the hot solution, it is necessary to add fresh acid from time to time. Under these conditions entirely satisfactory deposits of nickel were obtained from solution containing 5—12 grms. of metal per 100 c.c., at temperatures ranging from 50° to 90° C., by means of currents of from 0·70 to 3·0 ampères in density. The deposit was lighter in colour and tougher, the greater the current density and the degree of concentration of the electrolyte. At temperatures below 50° C., the deposit was brittle, and dark grey in colour. The use of an acid electrolyte causes a greater evolution of hydrogen than in the previous case, and the tendency towards roughness in the deposit is consequently increased. One experiment, in which filtering cloth was used to envelop the anode in order to retain the slimes, yielded a very brittle, scaly deposit of only one half the expected weight. Both the electrolyte and deposit were found to contain carbon; and the unsatisfactory nature of the deposit was ascribed to the passage of organic matters from the cloth into the hot electrolyte.

Electrolysis with Carbon Anodes.—The experiments made with carbon as anode did not yield successful results. The carbon disintegrated in the hot nickel chloride solution, and the effects produced in the electrolyte and upon the deposit of nickel were similar to those just described above. The author intends to continue these experiments with other forms of carbon, and also with lead peroxide as anode in a nickel sulphate solution. The conditions governing the electrolytic deposition of cobalt will also receive attention.

—J. B. C. K.

PATENTS.

Quantity of Metal which a Galvanoplastic Bath should Deposit on the Object treated, Apparatus intended to determine. J. Ducot, Paris, France. Eng. Pat. 18,537, Aug. 21, 1896.

The amount of metal to be deposited is determined by an automatic apparatus by a system of double weighing. The apparatus consists of a balance lever resting on two supports and a knife edge; the left portion of the lever carries a fixed counterweight, which it passes through lengthwise so as to produce a progressive resistance during the second weighing. The right arm of the lever is a rack bar, on which moves a sliding counterpoise, whereby the weight of the object treated is counterbalanced. From the same arm, and near its centre, is suspended a rod carrying the pieces which dip in the bath. The left arm carries a pointer, which moves over a dial until a movable handle is reached, which has been set at the predetermined weight. On the pointer and the handle making contact, an electro-magnet is actuated which breaks the circuit and stops the deposition, simultaneously ringing a bell. The fixing in point of the balance is facilitated by an indented piece, and compensation in the motion of the lever is assured by the placing out of centre of the fixed counterweight, and by an indented bar secured diagonally on the sliding counterpoise, and further by a spherical regulator above the balance knife edge.—G. H. R.

Metals or Alloys, Impts. in or connected with the Obtainment of, by Electrolysis or Hydro-electro-chemical Action. P. Marino, Brussels, Belgium. Eng. Pat. 27,776, Dec. 5, 1896.

The electro-depositing bath for a single metal may be made by mixing two solutions of salts of different acids of the same metal so as to produce double decomposition, or better results may be obtained by replacing one of the solutions by a solution of less than one equivalent of the salts of one of the alkaline earth metals or of lead, so as to produce incomplete double decomposition and a precipitate which is removed by decantation or filtration. In the case of alloys, the bath is composed of a mixture of the solutions of two salts of the respective metals, or of the metals and the salt of the alkaline earth or of lead, prepared as already described. Either before or after the incomplete double decomposition a saline solution or a mixture of saline solutions may be added to any of the baths prepared by this method, or free organic acids or free boric acid may be added to such baths.—G. H. R.

Anodes, Impts. in, for the Electro-Deposition of Nickel. T. R. Canning, Birmingham. Eng. Pat. 28,288, Dec. 10, 1896.

The improved anode is built up of blocks or cubes of nickel just as they come from the refiner, except that they are moulded with suitably shaped recesses on two opposite sides, so that when the blocks are built up into an anode, vertical holes are formed between superimposed layers which serve to hold connections of carbon or other suitable material. The blocks are retained in a frame lined with carbon, and provided with carbon lugs, and having a

lattice or wicker-work front and a hinged back, so that the anode may be raised, and any individual block replaced as required.—G. H. R.

Zinc, Impts. in Electrolytes for the Deposition of, and for Use in Secondary Batteries. H. Leitner, London. Eng. Pat. 1572, Jan. 20, 1897.

This electrolyte is an improvement on that described in Eng. Pat. 24,473, Nov. 1896, and is composed as follows:—

Concentrated solution of zinc sulphate	50 parts.
Sulphuric acid (pure)	10 „
Concentrated solution of manganese sulphate....	10 „
Ammonia alum in a concentrated solution, in which also a further amount of zinc sulphate has been dissolved	30 „

Zinc sulphate must be added, if necessary, till the solution approaches concentration. The solution may also be prepared by taking 9 parts of water with 1 of concentrated sulphuric acid, adding to this 1 part of concentrated solution of manganese sulphate, and saturating the resulting solution with crystals of zinc sulphate and alum, preferably ammonia alum. The electrodes, as described in the previous patent, consist of peroxide of lead plates, and plates of lead or other suitable base, which have been coated with zinc in the foregoing electrolyte, and this coating preferably consists of zinc layers, the first being slightly amalgamated with mercury before the second is given.—G. H. R.

XII.—FATS, OILS, AND SOAP.

Oils, Lubricating. F. Jean. Rev. de Chim. Ind. 1897, **8**, [91], 193—196.

The author considers that a lubricating oil should have a fairly constant viscosity at 50° and 100°, and that oils rendered viscous by the addition of soap should be proscribed altogether.—C. A. M.

Cerotic Acid and Ceryl Alcohol. R. Henriques. Ber. 1897, **30**, [11], 1415—1418.

In the course of his work on cold saponification (this Journal, 1896, 299), the author examined two specimens of Chinese wax obtained directly from China. Of these, one melted at 81·5° and had a radiating crystalline structure, whilst the other was more granular, and melted at 83°. Both contained ceryl cerotate as their chief constituent. After four crystallisations from petroleum ether, boiling from 115° to 135° C., this ester was obtained pure. Its melting point was 81·2° (Brodie, 82°), and its saponification number 73·6. The latter value was determined by dissolving 3 grms. of the ester in hot petroleum spirit, adding 25 c.c. of normal alcoholic soda, and either leaving for 24 hours in the cold or boiling for an hour under a reflux condenser. The excess of alkali was then titrated back with seminormal alcoholic HCl, since the aqueous acid was found to give erroneous results, owing to the ready decomposition of cerotic salts by water.

The formula given by Brodie for ceryl cerotate, $C_{54}H_{108}O_2$, corresponds to a saponification value of 71·1, whereas the author's saponification value corresponds to the formula $C_{52}H_{104}O_2$. Hence it was considered that Brodie's formula was doubtful, as well as his formula for cerotic acid ($C_{27}H_{54}O_2$) and ceryl alcohol ($C_{27}H_{55}.OH$). C. Hell and O. Hermanns (Ber. **13**, 1721) also expressed doubt as to Brodie's formula for cerotic acid, and more recently Marie (Ann. Chim. Phys. **29**, [7], 145) came to the conclusion that it should be $C_{25}H_{50}O_2$. To decide as to this point, the author prepared pure cerotic acid (see this Journal, 1890, 661) and ceryl acetate, and determined their saponification values. The pure ester was saponified, and the cerotic acid precipitated as the calcium salt, which was washed, dried, and extracted with acetone, the acid being liberated from the residual salt and purified by crystallisation from benzine or acetic acid. It melted at 78°·5 (Marie found 77°·5, uncorrected), and gave, as the mean of six determinations, a saponification number of 140·9. Hence its formula was $C_{26}H_{52}O_2$, which has by theory 141·4 for its saponification value.

The ceryl alcohol in the saponified ester was converted into the acetate by means of acetic anhydride, and this was purified by crystallisation from benzine. It then melted at 63°·5 and had a saponification value of 131·4, corresponding to the formula $C_{26}H_{53}.C_2H_3O_2$, with a theoretical saponification value of 131·1. According to this the formula for ceryl alcohol is $C_{26}H_{54}O$, and for ceryl cerotate $C_{26}H_{51}O_2.C_{26}H_{53}$.
—C. A. M.

Soaps, Disinfectant. S. Rideal, Brit. Pharm. Conference, Aug. 1897. Pharm. J. 1897, [1416], 158—160.

An oleic acid soap is stated to be preferable to one containing palmitic or stearic acid, as the sodium salts of the latter separate out on the addition of hot water. Superfatted soaps are practically useless for disinfectant purposes, since the free fat hinders the action of the disinfectant.

Sulphur and alkaline sulphides blend well with soap, but the former, even as milk of sulphur, is very slow in its action, on account of its insolubility. The principal objection to alkaline sulphides is, that a prominent feature of their antiseptic action is the gradual evolution of sulphuretted hydrogen. With regard to metallic soaps, if an internal effect is wished for, a metallic oleate soap will succeed, but if a local disinfectant or antiseptic action be required, a mixture must be used, such as a mercuric iodide soap (in which the mercury is incorporated in the form of the double iodide of mercury and potassium) or a zinc soap, in which the metal is added in the form of a neutral zincate solution, which will yield the metal in a soluble form to water. Carbolic and cresylic soaps, which are usually sold as of 10 per cent. strength, frequently contain much less of the disinfectant. All soaps of this form have a pronounced odour, and soaps containing homologues of cresol always possess a tarry odour, even when disguised by the addition of eucalyptus, gaultheria, or other scents. Ethereal oils have such a low antiseptic value that, to be effective, they require to be used in such quantities as are liable to cause serious irritation to the skin.

Different medicated soaps were examined bacteriologically in comparison with a curd soap containing 32·5 per cent. of water and 60·8 per cent. of fatty anhydrides, using for the experiments a 2 per cent. solution. Inoculation with active bouillon cultures gave results which are summarised in the following tables, a + sign indicating growth and a — sign sterility:—

I.—Bacillus Coli Communis in Vigorous Growth.

Time.	Curd Soap.	A Scented Curd Soap.	Carbolic, 10 per Cent.	New Disinfectants. A.	New Disinfectants. B.	HgI_2, 3 per Cent.	Formalin, 0·4 per Cent.
5 mins.	..	..	..	..	..	—	+
15 „	..	..	..	..	..	—	+
25 „	..	..	..	..	..	—	+
30 „	+	+	+	+	+	—	—
1 hour	+	+	+	+	+	—	—
1½ „	+	+	+	+	+	..	..
2½ hours	+	+	—	+	+	..	..
3½ „	+	+	—	+	(Much attenuated.)	..	..
4 „	—	—	—	—	—	—	..

II.—Staphylococcus Pyogenes Aureus.

Time.	Curd Soap.	A Scented Curd Soap.	Carbolic, 10 per Cent.	New Disinfectants. A.	New Disinfectants. B.	HgI_2. 3 per Cent.	Formalin, 0·1 per Cent.
10 mins.	+	+	–	+	+	–	+
20	+	+	–	+	(Attenuated)	–	+
30	+	–	–	–	–	–	–
Between 1 and 4 hours	–	–	–	–	–	–	–

The soaps were tried as sold. The relative amounts of disinfectants present in the solutions of the same strength (2 per cent.) would be carbolic, 0·2 per cent., or 7 in 500 of phenol; A, 0·06 per cent., or 7 in 1,666 of phenol; B, 0·03 per cent., or 7 in 3,332 of phenol; HgI_2, 0·06 per cent., or 7 in 1,666 of HgI_2; formalin, 0·008 per cent., or 7 in 12,500 of formaldehyde.

It was further found by bacteriological tests that many of the so called disinfectant soaps have little, if any, greater germicidal action than good curd soap itself.—A. S.

Linseed Oil, Investigation of. H. Amsel. Zeits. f. angew. Chem. 1897, [12], 369.

See under XXIII., *page* 829.

American Cotton-Seed Oil in Marseilles. U.S. Cons. Reps. 1897, **54**, [202], 430.

See under Trade Rep., *page* 839.

Soap, Analysis of. R. Moreschini. Annali del Laborat. Chim. Centr. delle Gab. III., 107.

See under XXIII., *page* 831.

PATENTS.

Soap, Impts. in the Manufacture of, and in Frames used in the Manufacture of, and in the Method of Cutting or Slabbing. H. Hadfield, Whaley Bridge, Chester. Eng. Pat. 19,903, Sept. 9, 1896.

In place of the ordinary frame with removable sides and ends, the patentee employs a frame cast in one piece, or built up in sections, and fitted with a false bottom capable of being raised or lowered by chains, bands, hydraulic pressure, &c. This is used in combination with an arrangement of cutting wires with which the soap comes in contact when raised from the frame, and by means of which it is cut into slabs, &c. Another cutting frame with the requisite number of wires, and provided with a longitudinal movement, completes the division into bars when the soap has been raised to a sufficient height.—C. A. M.

Soap, Dry, or Soap Powder: Impts. in the Manufacture and Production of. F. W. Wright and The United Alkali Co., Ltd., both of Liverpool. Eng. Pat. 23,025, Oct. 16, 1896.

A mixture of ordinary soap with crude bicarbonate of soda (as obtained in the ammonia-soda process), dried either by the addition of anhydrous monocarbonate of soda or by gentle heat.—C. A. M.

Soap, Impts. in the Manufacture of. E. G. Scott, Liverpool. Eng. Pat. 26,285, Nov. 20, 1896.

The improvement, which relates to the manufacture of soft soap, consists in separating the oil or oils into glycerin and fatty acids by means of superheated steam or otherwise, recovering the glycerin and saponifying the fatty acids.
—C. A. M.

Fragrant Substances, Method of Producing Highly Concentrated Fat Solutions of. H. Mack, Ulm-on-the-Danube. Eng. Pat. 8387, April 1, 1897.

A solution of the fragrant substance in alcohol is diluted with water and shaken up with a scentless fat or with vaselin, so as to extract the perfume. The fat or vaselin, with the fragrant substance in solution, is separated from the liquor, and mixed with a "pulverous" material, such as starch, for use as a washing substance.—C. A. M.

Soap, Improved Process for Making. R. Gesell, Berlin. Eng. Pat. 17,783, July 28, 1897.

Soap mass mixed with pulverised wood fibres, impregnated with a suitable antiseptic and (or) perfume, together with pulverised bran, oatmeal, and asbestos, in suitable small proportions.—C. A. M.

XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

(A.)—PIGMENTS, PAINTS.

Paint Trials. M. Toltz. Chem. News, 1897, **76**, [1970], 104; and J. Assoc. Eng. Soc., June 1897. A paper read before the Civil Engineers' Society of St. Paul, Dec. 7, 1896.

The author tested the following paints as to their comparative value for protecting iron surfaces:—True asphaltic varnish paints; so-called asphaltic varnishes of inferior qualities; black carbon paints, of which the vehicle is practically a varnish; iron oxide paints; and graphite and silica graphite paints. Red lead was not tested, as, although it is still used to a certain extent, it is generally mixed with graphite. To ascertain the resistance to moisture, a set of sheet-iron dishes, 12 ins. in diameter by 0·5 in. deep, were painted, the scale or skin being first carefully removed. Two dishes were coated with each kind of paint, one receiving one coat, and the other two coats, the first being allowed to dry at least a week before the second was laid on. When the paint was perfectly dry, a given amount of water was placed in the dishes and allowed to evaporate at the ordinary temperature, this being repeated until the dishes showed more or less rust. After most of the water had evaporated, there remained, at the junction around the edge, a thin film of water, which, in contact with the carbonic and other acids of the air, acted on the paint in such a way that the iron beneath it began to rust. In actual practice, the same thing happens, except that the rust extends under the paint, and does not show as plainly as on the dish. Only paints which will withstand this test, are stated to be desirable for the protection of iron and steel structures. The cheap asphaltum paints and iron oxide paints failed under this test. Sheet iron, coated with the various kinds of paints, was also exposed to a temperature of 220°—300° F., for the purpose of ascertaining whether they would keep their elasticity or become so brittle as to be easily removed from the surface.

As a result of the experiments made, it is stated that iron oxide paints are not very desirable, at least for the first coat or two, for iron or steel; but as a third coat, for the protection of the underlying paints, they may be recommended. However, as graphite paints in paste form, well ground and mixed with boiled linseed oil, do not cost very much more than the cheapest iron oxide paints, they are recommended in preference to the latter for the second or third coat, as, if properly applied, they withstand the action of moisture much better. Asphalt varnish paint or carbon paint is recommended for the first coat, but the surfaces of iron and steel must be as free from moisture as possible while the structures are being painted, otherwise there is great danger that the coating will not adhere very firmly, and the value of the paint will thus be nullified. This precaution is less important in the case of iron oxide or red-lead paints simply mixed with linseed oil, as the latter readily absorbs moisture, whilst carbon and asphalt paints do not; but the lack of this property in the two last-named paints is one of the principal reasons why they are

superior to any other class. The author describes in detail a system for efficiently protecting iron and steels structures by successive coats of paint.—A. S.

PATENTS.

Lead Sulphite for Use as a White-Lead Pigment, A Process for the Manufacture of. A. C. J. Charlier, Glasgow. Eng. Pat. 19,788, Sept. 8, 1896.

A current of sulphurous acid gas is led, at a pressure of 10 or 20 lb. per square inch, through a number of vessels containing finely ground lead oxide, made into a paste with water, and maintained at "the proper temperature" by means of steam coils and agitators. The operation may be conducted so as to produce either a basic or a normal sulphite of lead; and if the former, it may be neutralised with sulphuric acid in order to yield a mixture of sulphite and sulphate. If preferred, carbonic acid may also be introduced simultaneously into the vessels, when the resulting precipitate will consist of the carbonate as well as the sulphite. The advantages of the sulphite of lead are said to be that it is non-poisonous, and has greater covering power and body than any other white lead at present manufactured.—F. H. L.

Zinc-White, Impts. in the Manufacture of. H. H. Lake, London. From G. Harmegnies, Brussels. Eng. Pat. 23,716, Oct. 24, 1896.

Waste zinc ("rag-picker's zinc") is dissolved in hydrochloric acid, the solution filtered, decomposed with the theoretical quantity of magnesia, and the oxide washed and dried. The process is stated to be more economical than the usual method of distillation in air, inasmuch as the hydrogen can be burnt, and the filtrates and washings containing magnesium chloride may be concentrated to a density of 1·4, and then heated to 250° C. to regenerate fresh acid and magnesia. Presence of lead in the original zinc is immaterial; but if the solution contain more than 0·5 per cent. of iron, it should be removed by means of zinc oxide.—F. H. L.

Paint for Ships' Bottoms, An Improved. G. L. Burnham, Providence, U.S.A. Eng. Pat. 2108, Jan. 26, 1897.

10 galls. of linseed oil are boiled for five hours at 315° C. with 8 oz. of litharge and 8 oz. of lead acetate. When cold, the varnish is diluted with oil of turpentine to working consistency; and it is then mixed with "1 pint of seaweed, which is first ground—while green and wet, just as it is picked from the seashore—with oil." The composition is stated to yield a smooth enamelled surface, and it is also claimed to be an effectual preventative against barnacles and worms.—F. H. L.

Enamel Paint, and Method of Applying the same, An Improved. F. Bovling, Brisbane, and C. L. Garland and A. Ogden, Sydney. Eng. Pat. 14,105, June 9, 1897.

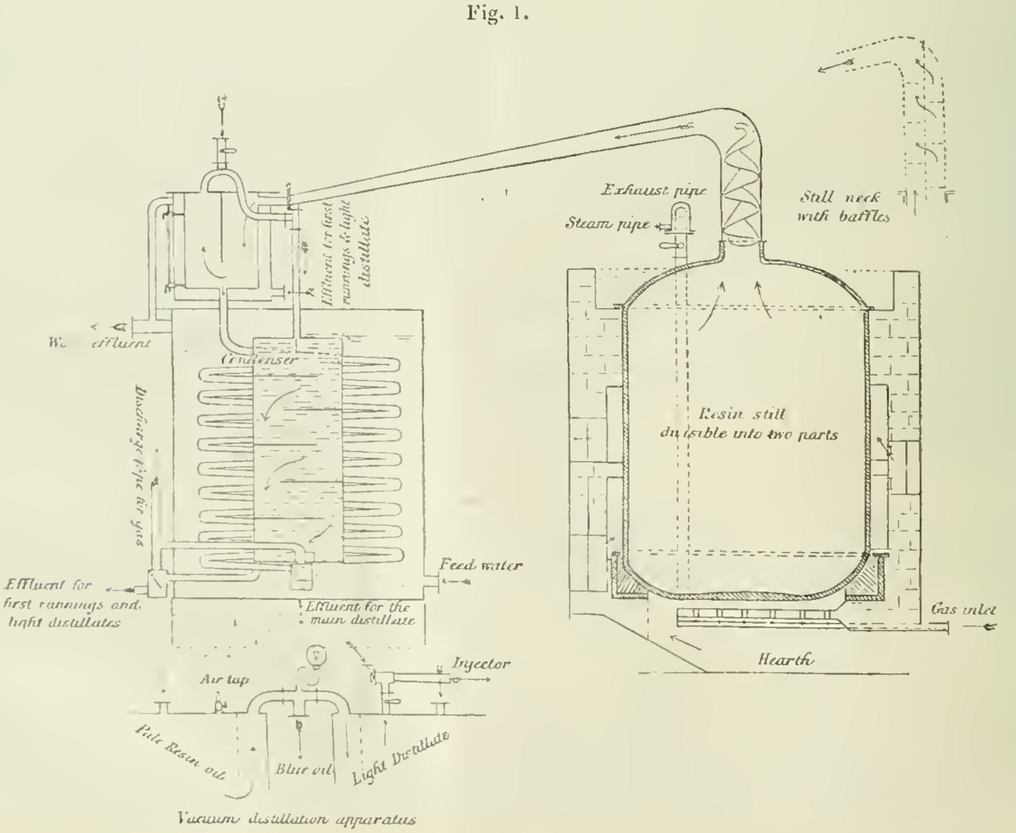
The desired pigment, suspended in gold size, is mixed with carriage varnish to a thin paste, about 20 per cent. of a 5 per cent. solution of shellac in methylated spirit introduced, and the whole thinned to working consistency with the carriage varnish already employed. For use on leather, 10 per cent. of raw linseed oil is added, and the material is stoved at 60° C.; but in the case of metal, the temperature should be about 120°. The composition may also be employed without stoving, like an ordinary paint, or as a coating for ships' bottoms.—F. H. L.

(B.)—RESINS, VARNISHES.

Crude Turpentine and Resin, Impts. in the Treatment of. Fr. Boleg. Chem. Rev. Fett- u. Harz-Ind. 4, [12], 159—161; [13], 173—176; [14], 187—189; [15], 201—205; [16], 218—220.

Resin Distillation.—The best form of still is said to be a cylindrical vessel capable of holding 5 tons of resin when three-quarters full (73 by 92½ ins. without the lid dome), preferably in two parts, and made either of cast iron or iron plate, with cast rounded bottom. The cast-iron lid is 12 to 14 ins. high, rounded, and attached by screwing. It is fitted with a manhole, safety-valve, and a central flanged

Fig. 1.



Vacuum distillation apparatus

pipe, 8—10 ins. high and about 14 ins. inside diameter, to support the copper neck, which, in order to facilitate the production of thin oil free from resin, should be at least 40—60 ins. high, and be fitted with baffles extending up to the bend and causing the vapours to pursue a spiral course. The neck tapers down to a diameter of $2\frac{1}{2}$—3 ins. at its junction with the condenser. For the latter a simple worm condenser is insufficient for the production of better-class oil, and a double condenser is requisite, of the kind shown in the annexed figure, wherein the first runnings—acid water and strong-smelling light oils—can be passed through a separate apparatus to that used for the main distillate. Moreover, the pungent green oil coming over last of all must be led into an entirely separate condenser.

With this apparatus a simple fractional distillation is generally effected, and is usually sufficient. For special purposes, however, and particularly in the production of *drying oils*, a partial vacuum, equivalent to 15—25 (more rarely 50—60) c.m. of mercury, is produced by means of an exhaust attached to the receiver. This arrangement is also useful for certain purposes of rectification, 3—5 per cent. of fresh powdered lime being at the same time used to ensure sufficient decomposition of the distillate.

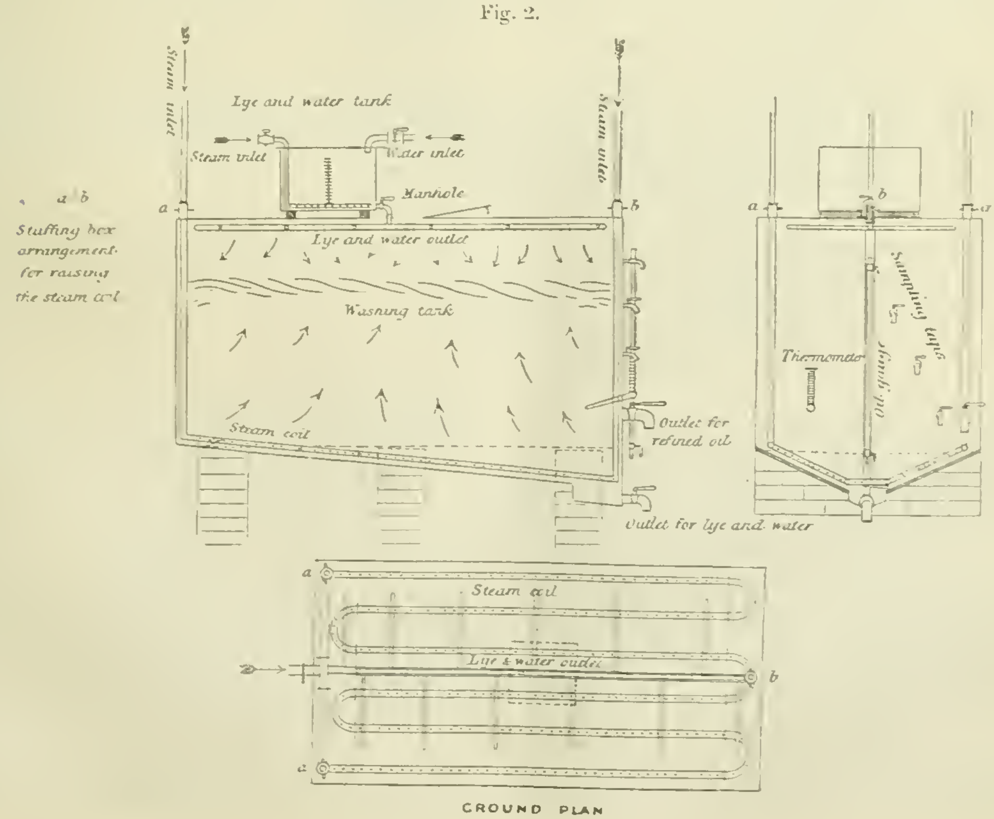
In large distilleries the only suitable method of firing, is to combine coal with resin gas. The bottom of the still is protected from the direct contact of the flame by means of a firebrick arch or dome, and the hearth must be long and broad, and not at too great a distance from the arch. A mixture of lump coal with nuts and smalls is recommended for firing, the proportion of the latter increasing as distillation progresses. The gas jets must be situated close under the arch, and adjoining the openings therein. By an intelligent use of gas-firing, as much as one-third of the coal bill can be saved, and a considerable quantity of resin spirit (pinolin) recovered. The best form of flue round the cylindrical part of the still, is one 6 ins. wide by 18 ins. high, running once forwards and once back, on either side, and closed with dampers.

The still being charged with resin, and the fire kindled, a saving of time is effected by pouring in from $2\frac{1}{2}$ to 5 per cent. of pale paraffin oil, of suitable density and boiling point, which will prevent the frothing up and boiling over, due to water in the resin, and will thus enable the still to be closed at once. This addition, moreover, ensures an additional 5 per cent. in the yield of pale distillate. The products obtained are: resin essence (pinolin) and acid water, 6—8 per cent.; pale resin oil, 50—55 per cent.; "blue" oil for refining or waggon-grease making, 20—15 per cent.; "green" oil for waggon-grease or carbolineum, 6—7 per cent.; gas, 5—7 per cent.; loss (and worthless residue), about 10 per cent.

If the still be charged overnight and the fire banked up under the melted resin, work can begin at 5 a.m., and the distillate will begin to run in about $1\frac{1}{2}$ hour's time. By 8 p.m. (or two hours earlier when working with a vacuum) everything will have passed over except the green oil, which is transferred to a special still either the same night or early next day, and the resin still recharged at once with ready-melted resin, or opened and well aerated to prevent risk of explosion, and then filled as before. In this way the stills can be worked four times a week without night work, thus saving fuel, labour, and wear and tear. The residues from eight distillations are united and distilled for "green" oil in smaller cast-iron stills, the pitchy residue being used for making lacquer for ironwork, shoemakers' pitch, &c.

Treatment and Utilisation of the Resin Spirit (Pinolin).—The rectification of the crude spirit is preferably carried out by the aid of direct and indirect steam, a vacuum, and a double condenser. This ensures a paler, purer, and milder product than by the ordinary method, and an increase of 5—7 per cent. in the yield. Instead of selling this spirit it can be advantageously worked up by treating it with direct steam and 3—$3\frac{1}{2}$ per cent. of soda lye of 36—40° B., at a temperature of 80°—100° C., carefully removing the lye, washing with lukewarm water, and then mixing with 2 per cent. of sulphuric acid of 66° B. (at 15°—25° C.). After the acid has settled down—in 3 to 4 hours—another 2 per cent. of soda is added, to effect thorough neutralisation, and the spirit is finally rectified, like oil of turpentine, *in vacuo*, by direct and indirect steam, and $\frac{1}{2}$ per cent. of soda or 3—5

Fig. 2.



per cent. of lime water. The resulting product is very similar to commercial oil of turpentine, and may be still nearer approximated thereto by an admixture of true oil of turpentine, essence from root-stock- or wood-tar oil, or crude turpentine, either before or during rectification. A considerably enhanced value is thus obtained, the product being fit to serve either as an adjunct to, or substitute for, oil of turpentine, and sure of a more ready sale than resin spirit of the ordinary quality.

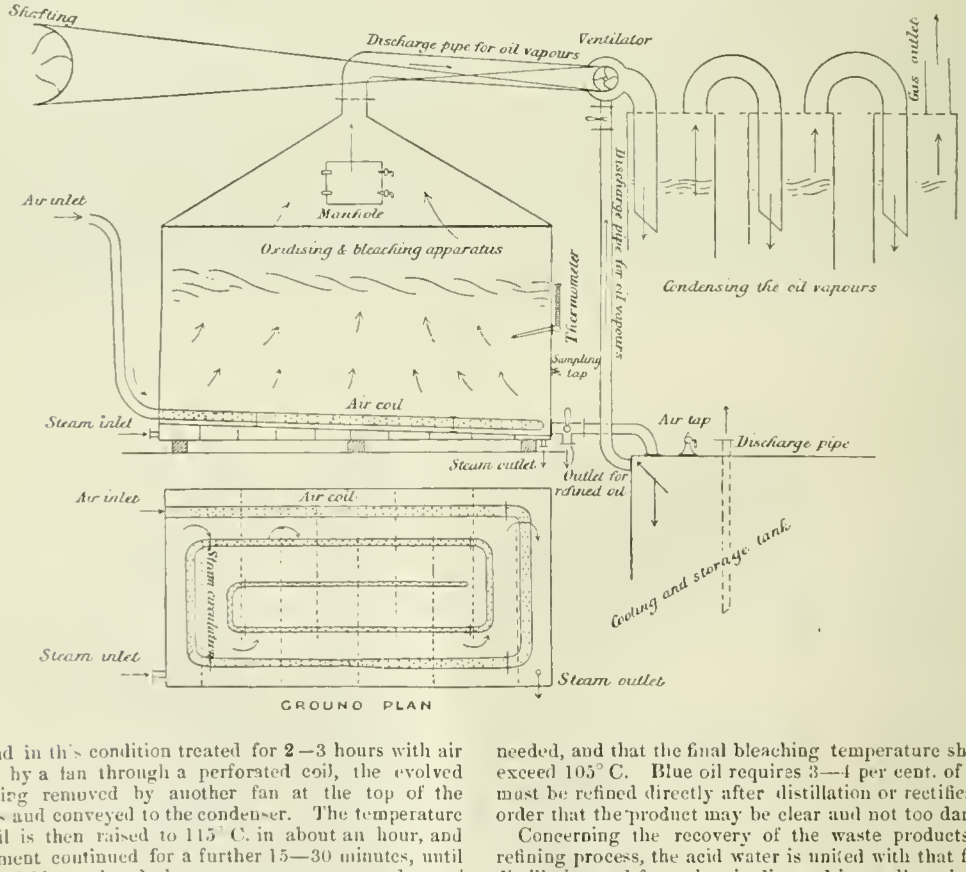
No success has attended the attempts hitherto made to recover the washing lyes or the waste products therein, though the lye used for the second washing can be employed for the first washing of the succeeding batch of spirit.

Refining and Utilising the Resin Oils and Waste Products.—The fractions refined are the pale yellow or "blonde" oil and the blue oil. When distilled in the apparatus already described, they will not require a preliminary rectification, since the percentage of saponifiable resin constituents does not exceed 10—11 per cent., as compared with 20—26 per cent. in the crude oils from the ordinary distillation methods.

The apparatus consists of a washing tank, 120 by 60 by 60 ins. (Fig. 2), and a bleaching tank of similar size (Fig. 3). A 5-ton charge of "blonde" oil being placed in the former to allow the acid water to settle, about $\frac{1}{4}$ part of hot water is added, and the whole boiled a short time by direct steam, to facilitate the operation. When freed from the acid water the oil is again boiled by direct steam, and treated with 4—5½ per cent. of soda lye of 36°—40° B. sprinkled over the surface, the heating being continued until a sample shows the oil to be free from resin and acid, whereupon the steam must be instantly shut off, or the product will assume a dark colour and turbidity due to dissolved resin soaps. For the same reason the steam coil should be at once lifted out of the liquid, by means of the stuffing-box arrangement *a*, *b* (Fig. 2), and cleaned by a short blast of steam before being used again. By taking this precaution a second vessel for the final washing may be dispensed with, and a clearer and paler product obtained.

At the end of an hour's time the water is drawn off, and the oil washed again by about 33 per cent. of water at 50°—60° C. and direct steam (the temperature not to exceed 105°) until a sample clarifies quickly. In case the operation does not require repeating, the oil, after the water has subsided, is drawn off into the bleaching and oxidising apparatus (Fig. 3), where it is warmed up to about 60° C. by indirect

Fig. 3.



steam, and in this condition treated for 2—3 hours with air blown in by a fan through a perforated coil, the evolved gases being removed by another fan at the top of the apparatus and conveyed to the condenser. The temperature of the oil is then raised to 115° C. in about an hour, and the treatment continued for a further 15—30 minutes, until the oil is debloomed and the vapours assume an odour of camphor or peppermint. In order to render the oil inodorous, it is necessary to continue exhausting the vapours until the temperature of the oil has fallen to about 30° C., and the addition of 10—15 per cent. of salt water during the bleaching process exerts a favourable influence, by preventing the renewed formation of oxide- and empyreumatic resins, which otherwise occurs, the formative substances being absorbed and removed by the brine. The oils thus treated will be found free from resin and acid, and suitable for the preparation of drying-oils for varnish-making.

In refining rectified blonde oil, the same treatment is applied, except that only 3—3½ per cent. of lye will be needed, and that the final bleaching temperature should not exceed 105° C. Blue oil requires 3—4 per cent. of lye, and must be refined directly after distillation or rectification, in order that the product may be clear and not too dark.

Concerning the recovery of the waste products of the refining process, the acid water is united with that from the distillation and from the pinolin, and in small works is used to decompose the resin soap lyes. In large works, however, it is more advantageously purified, concentrated, and worked up in the manufacture of acetic acid, pyroligneous acid, or acetates. The resin soap lyes are boiled for about five minutes by direct steam, and left for an hour. The oil collecting on the surface is removed, to be returned to the washing tank, and the residue treated by blowing in steam and carbon dioxide (preferably liquid commercial) until the thick oil is separated from the residual (soda) lye. The former is either redistilled for refining or sold for making waggon grease, and the lye is causticised and used again. In smaller works the thick oil is separated by means of the

acid water in a similar manner, but in this case the residual liquor is valueless.

Some of these resin soap lyes can be utilised for facing certain common grades of paper, as also for cheap resin-curd and soft soaps, disinfectants, and in the manufacture of water-glass; so that there is no waste. The light essences and vapours collected from the bleaching tank are mixed with the crude pinolin, to be decomposed into spirit and oil.—C. S.

Varnish, Gold-coloured; for Tin-Plate. Dingler's Polyt. J. 1897, **305**, [7], 168.

For the preparation of this varnish, 50 grms. of crystallised copper acetate are ground to a very fine powder in a porcelain mortar, and the powder is spread on a flat plate, and kept for some time in a warm place. The water of crystallisation and part of the acetic acid are thus volatilised, and the light-brown powder obtained, is triturated with oil of turpentine, and finally to the mixture is added with constant stirring, 150 grms. of copal oil varnish, warmed to 70° C. The copper salt, if it has been well ground, dissolves fairly well with stirring in a quarter of an hour, and the varnish is kept in a warm place for some days, with frequent agitation. The tin-plate is coated evenly with the varnish and then dried in an oven, and the tone of colour depends on the heating: a greenish colour first forms, changing on stronger heating to yellow and dark yellow, then to orange, and finally to reddish brown. The colour is fast to daylight, and better than the English gold lac with regard to fire. If the copal varnish used be of good quality, the varnished tin may be pressed and bent in any shape without injury to the coating. To produce a green colour, the metal is given five coatings, letting each one dry before the next is applied, and the varnished plate must be dried by only a mild heat.—A. S.

Balsams, Resins, and Gum Resins. K. Dieterich. Chem. Rev. Fett- u. Harz-Ind. **4**, [15], 205, and [16], 215.

See under XXIII., *page* 829.

PATENTS.

Manufacture of Sealing Wax, Impts. in the. P. S. Watkins and P. J. Nunn, London. Eng. Pat. 15,180, June 24, 1897.

This patent refers more especially to the cheaper kinds of sealing wax, such as those used for bottles, &c., and the improvement consists in simply mixing the powdered ingredients together instead of melting them and casting the product into cakes or sticks, as is the usual custom.

—F. H. L.

Compositions containing Nitrocellulose and in Articles treated therewith, Impts. in. P. M. Justice, London. From "The Marsden Company," Philadelphia, U.S.A. Eng. Pat. 17,602, July 27, 1897.

For use as a waterproofing composition, 100 parts of a solution of nitrocellulose are mixed with 55 parts of lanolin, and the whole spread over the desired material in the usual manner, or made into thin sheets by allowing it to dry on glass plates, from which it is ultimately stripped. As a flexible covering for leather, &c., 12 oz. of nitrocellulose are dissolved in 1 gall. of amyl acetate, 10 oz. of lanolin, and 3 oz. of "corn oil" added, and finally some colouring matter, such as aniline black, introduced.—F. H. L.

(C.)—INDIA-RUBBER, &c.

Rubber Hose, Mending Cracked. Leipziger Färber Zeit. **46**, [7], 312.

To mend cracked rubber hose, the damaged part, previously well cleaned and dried, is painted over with hot oil of turpentine. A thin sheet of gutta-percha, softened by heat, is put round it so that the edges meet, and is pressed against the hose with a knife blade. The edges are finally cemented together by touching the seam with a moderately hot iron rod.—I. S.

Gutta-Percha. J. A. Montpellier. Rev. de Chim. Ind. 1897, **8**, [91], 200—204.

Gutta-percha was first introduced into Europe by Joseph d'Almeida in 1843. It is contained in the milky juice of a variety of *Isonandra-gutta*, a tree belonging to the genus *Bassia butyracea*, which occurs principally in the Dutch Indies, Malacca, and the Malay Archipelago, where it attains a height of 40 feet.

The natives formerly extracted the juice by felling and barking the trees, but it is now collected in a similar manner to caoutchouc, by making incisions and collecting the exuding juice. This consists of an aqueous and a pasty portion, the latter constituting the gutta-percha. When sufficiently coagulated it is separated from the liquid portion by squeezing with the hands. In another method, now abandoned, the juice was first gently warmed for several days in order to evaporate part of the aqueous liquid.

In 1888, Serullas undertook a mission with the object of attempting to acclimatise the *isonandra* in different colonies and of cultivating it on a large scale. From experiments on samples forwarded by him, Jungfleisch found that gutta-percha was present in all parts of the tree, and that it could be extracted by means of a solvent, such as toluene. The method now used industrially as the outcome of these researches consists in digesting and extracting the dried and powdered leaves with some such solvent at 100°, and removing the solvent by means of the prolonged action of low pressure steam. The yield varies from 9 to 10·5 per cent., and the product is much purer than the ordinary commercial gutta-percha, which has a reddish colour, due to the presence of fragments of bark, earth, &c. Shortly after this process was brought out, an analogous method was proposed by Dieudonné Rigole, in which the leaves were extracted with carbon bisulphide in a special apparatus.

Crude gutta-percha is of very varying quality, the most esteemed varieties coming from Singapore and Borneo. Much of the commercial article is not derived from the *isonandra* alone, but has been mixed by the natives with the juice of similar trees, and in all cases it requires cleansing from particles of bark and dirt. This is most simply effected by dividing it in a machine, and washing it first in a current of cold water and then in one of hot, which causes the denser foreign bodies to fall to the bottom. The cleansed gutta-percha is then kneaded in a special apparatus heated by steam, where it agglomerates and loses most of the water taken up in the washing.

As thus obtained, the purified gutta-percha is of a brown colour, conducts heat badly, and is readily electrified by friction. Between 0 and 2° it is very tenacious, elastic, and fairly supple; between 25° and 30° its flexibility increases, and when the temperature reaches 50° it can be easily flattened out. From 100° to 110° it fuses to a paste, boils at 120°, and is decomposed at 130°, yielding, on distillation, colourless oils, consisting for the most part of isoprene and caoutchine, whilst a residue of carbon remains in the retort. It does not possess the elasticity of caoutchouc, but, on the other hand, unlike that substance, does not lose its elasticity at − 10°. It also differs from caoutchouc (which is apparently structureless) in having a cellular structure, which becomes fibrous when subjected to tension. It is very porous, so that its density often appears to be less than that of water.

Its best solvents are carbon bisulphide, chloroform, and toluene in the cold. It is partially soluble in anhydrous ether and alcohol, and in benzine at 25° and higher. It is readily soluble in warm essence of turpentine from a solution in which it can be precipitated with alcohol. Sulphuric acid (monohydrate) attacks it in the cold, liberating sulphur dioxide. Fuming nitric acid has a very energetic action upon it, liberating nitrogen peroxide, and producing, according to Oudemans, among other substances, formic and hydrocyanic acids. Hydrochloric acid attacks it slowly, changing its colour to brown, and making it friable. As it is not attacked by hydrofluoric acid, it is used for the manufacture of vessels to hold solutions of that gas. It resists the attack of different ferments, and is unacted upon by alkaline or saline solutions. When obtained pure by dissolving it in chloroform or carbon bisulphide, filtering, and allowing the solvent to evaporate, it is white or greyish-white and opaque, but under the combined action of light and air it alters, turning yellowish, hardening, increasing in weight, and becoming more soluble in alcohol. This

appears to be a process of oxidation, which is favoured by a suitable temperature (25°—30°), and a fine state of division. It does not take place when the gutta-percha is immersed in water.

The first chemical research on gutta-percha was made by Payen in 1851, who found it to consist of three proximate constituents, which he named *gutta, albane*, and *fluavile*. —C. A. M.

PATENTS.

Manufacture of India-rubber Substitutes as applied to the Covers or Jackets for Pneumatic Tyres, for Cycles, Carriages, and all Classes of Vehicles, Impts. in the. J. Cooper, London. Eng. Pat. 17,387, Aug. 6, 1896.

This material consists of india-rubber 6 lb., gutta-percha 2 lb., magnesium carbonate 6 lb., antimony sulphide 2 lb., and shellac 8 oz., together with sufficient sulphur and lime to vulcanize. If the composition is to be used for the manufacture of belts or packing, only one-half of the above-mentioned amount of gutta-percha is employed. —F. H. L.

Gutta-Percha from the Leaves and Twigs of the Isonandra Gutta and Kindred Plants, Improved Process and Apparatus for the Extraction of. Siemens Bros. and Co., Westminster, and E. F. A. Obach, Old Charlton. Eng. Pat. 19,046, Aug. 28, 1896.

The stems and twigs are crushed in such a manner as not to produce any dust, and then extracted with light hydrocarbons of the paraffin series in steam-jacketed vessels fitted with inverted condensers. The extracts and washings are cooled to precipitate the gutta, and the latter is finally washed with fresh solvent till colourless. Melted paraffin wax can also be used as the extracting agent, for, on cooling, the gutta remains imbedded in the solidified hydrocarbon and protected from the air, so that it is convenient to handle. Separation is then effected by dissolving out the paraffin by means of petroleum spirit.—F. H. L.

Elastic Filling, An Improved, more especially applicable for Cycle Tyres, Cycle-Handles, Cushions, and the like, and in the Method of Manufacturing the same. J. Deborde, Berlin. Eng. Pat. 8905, April 7, 1897.

It is proposed to fill tyres and saddle-cushions of bicycles, and cushions generally with a gelatinous or glutinous substance which has been transformed into a spongy or foamy condition by mechanical or other treatment. For this purpose 25 parts of gelatin are liquefied by heating, 25 parts of glycerin and ½ part of formaldehyde added to it, and this mixture, after being stirred or beaten into a frothy mass, is filled into the above-mentioned articles. Or, it is cast into appropriate moulds and the solidified castings are then provided with a tightly-closing cover of india-rubber or leather.—C. O. W.

Extracting and Purifying Gutta Percha, Impts. in. W. Ramsay, London. Eng. Pat. 17,936, July 30, 1897.

The essential feature of this invention consists in the substitution of rosin oil for the toluene which is often employed in the extraction and purification of gutta percha, *e.g.*, Eng. Pat. 654, 1896 (this Journal, 1897, 52). The leaves, &c. are washed in alkali and water, extracted with rosin oil in a closed vessel at 80° to 130° C., the gums precipitated from their solution by acetone, and the latter recovered by distillation.—F. H. L.

XIV.—TANNING, LEATHER, GLUE, SIZE.

PATENTS.

Leather, a Substitute for, and Process of Manufacturing the same and other Analogous Substances. From F. Billing and A. Letalle, Birmingham. Eng. Pat. 22,965, Oct. 16, 1896.

The substitute is prepared by immersing sheets of cotton wool or other suitable fibrous material in a solution of gelatin and methylated spirits or naphtha which has been mixed in the form of spray by means of a suitable apparatus. The composition may be made supple by the addition of treacle, glycerin, or like substances. It is dried and subjected to the action of a solution to coagulate the gelatin. Formalin, alum, tannic acid, or a mixture of such substances may be used. The product is dried and embossed with patterns between engraved plates, in imitation of morocco or other leather.—J. T. W.

Tanning Process, An Improved. From A. M. Clark. By Les Fils de F. P. Heinrich, Schlettstadt. Eng. Pat. 24,812, Nov. 5, 1896.

This is a rapid tanning process in which naphthalene is added to the weak liquors. The hides are suspended successively in three tanning liquors containing naphthalene, of the respective specific gravities 1·2°, 2°, and 3° B. They are then transferred to a drum with a strong liquor of 15° B. also containing naphthalene. Five kilos. of naphthalene are used for about 17,600 gallons of liquor. The action of the naphthalene causes the grain to remain soft, and a thick ox hide can be tanned in 50 hours.—J. T. W.

Tanning, Impts. in Processes of. C. S. Dolley, Philadelphia, U.S.A. Eng. Pat. 12,099, May 15, 1897.

Hides or skins, prepared in the usual way for tanning, are subjected to the action of an aqueous solution of formaldehyde, of a strength gradually increasing from 3 to 10 per cent. About one hour's treatment completes the process. Or, the hides are exposed in a close chamber to gaseous formaldehyde, either by itself or in conjunction with aqueous or alcoholic vapours.—C. O. W.

Blood and other Albuminous Matter, Impts. in the Treatment of, both Animal and Vegetable, for Use in Manufacture and the Arts. P. G. Sanford, London. Eng. Pat. 12,449, May 20, 1897.

The invention consists in the treatment of animal or vegetable albuminous matter with ammonium fluoride, or ammonium, sodium, or potassium borofluoride.—C. O. W.

Grease or Fatty Matter, Impts. in the Method of and Apparatus for Removing, from Sheep Skins and other Leather and the like. W. Sagar, sen., A. P. Sagar, and W. Sagar, jun., Colne. Eng. Pat. 13,778, June 4, 1897.

A special form of extraction plant for degreasing sheep skins and the like with suitable solvents at moderate temperatures and without undue waste of solvent.—C. O. W.

XV.—MANURES, Etc.

Mineral Phosphates [Manures], Estimation of Lime Alumina, and Iron in. L. Lindet. Comptes Rend. **125**, [4], 246.

See under XXIII., *page* 828.

XVI.—SUGAR, STARCH, GUM, Etc.

Molasses, Commercial Analysis of. M. Molhaut. Bull. Assoc. Belge de Chim. **11**, [3], 86.

See under XXIII., *page* 830.

Starch Manufactures, The Digestibility of certain By-Products of. B. Schultze. Jahresbericht der agr. Vers. Station zu Breslau, 1896.

See under XVIII. A., *page* 820.

PATENT.

Sugar, An Improved Process for Refining and Purifying. G. Ranson, Phalempin, France. Eng. Pat. 19,815, Sept. 8, 1896. (See also this Journal, 1897, 688—689.)

The syrup or sugar to be purified is made slightly alkaline with barium hydrate or sodium carbonate, and hydrogen peroxide added in the proportion of from ½ to 5 litres per 100 kilos. of sugar according to the colour of the product. The decolorisation is effected gradually. 100 grms. of finely powdered animal charcoal, from which all the phosphate has been removed by acid, to every 100 kilos. of sugar, is added, to expedite the liberation of oxygen

from the hydrogen peroxide so that the organic compounds capable of being decolorised may be oxidised. The excess of oxygen is removed from the syrup by adding hydrosulphites of aluminium, barium, &c., or an aqueous solution of hydrosulphurous acid, or by producing this acid in a nascent condition in the syrup.

To prepare the hydrosulphites of aluminium, barium, &c., the corresponding "bisulphate" (? bisulphite) is placed in a vessel capable of being hermetically closed, the bottom of which contains powdered or granulated zinc. The reduction is allowed to proceed for 30 minutes, the liquid cooled, decanted, and added to the syrup. A solution of hydrosulphurous acid may be made by adding 20 grms. of zinc powder to a solution of sulphurous acid containing 100 to 200 grms. of acid per litre. To prepare nascent hydrosulphurous acid, sulphurous acid gas is passed into the syrup until the acidity is 0·1 to 0·2 grm. per litre, then 10 grms. of zinc powder are added for each 100 litres of syrup and the whole is stirred.

The two periods of oxidation and reduction leave an excess of sulphite in the juice, which is converted into sulphate by adding the necessary quantity of hydrogen peroxide. The temperature of the mixture is then raised to 78° C. and the syrup filtered. The heated mass being thus freed from solid substances, the excess of sulphurous acid escapes as soon as the liquid boils, becoming thereby alkaline and preventing inversion.

In purifying sugars by acid treatment the syrup is saturated at a temperature below 50° C. with sulphurous acid. The organic matter is decomposed, and the alkaline salts converted into sulphites, which latter are oxidised to sulphates by treatment with hydrogen peroxide. The syrup is filtered, and again saturated with sulphurous acid. 10 to 50 grms. of zinc powder to each litre of syrup are added, and the whole mixed for 15 to 20 minutes; nascent hydrosulphurous acid is formed, which effects decolorisation very energetically. The temperature is raised to 75° C. before filtering, because at this point the hydrosulphurous acid is decomposed, taking oxygen from the organic matter in the syrup. The peroxide of hydrogen must then be added in the same manner as in the first-mentioned alkaline process.
—J. L. B.

XVII.—BREWING, WINES, SPIRITS, Etc.

Lupin Seeds, The Proteids of. T. B. Osborne and G. F. Campbell. J. Amer. Chem. Soc. 1897, **19**, 454.

See under XVIII. A., *page* 819.

Cow Pea, The Proteids of the (Vigna Catjang). T. B. Osborne and G. F. Campbell. J. Amer. Chem. Soc. 1897, **19**, 494.

See under XVIII. A., *page* 820.

Adzuki Bean, Proteid of the White Podded (Phaseolus Radiatus). T. B. Osborne and F. Campbell. J. Amer. Chem. Soc. 1897, **19**, 509.

See under XVIII. A., *page* 820.

Sunflower Seed, The Proteids of the. T. B. Osborne and G. F. Campbell. J. Amer. Chem. Soc. 1897, **19**, 487.

See under XVIII. A., *page* 820.

Barley, The Cell-Wall dissolving Enzyme of. F. Reinitzer. Wochenschr. f. Brau. 1897, **14**, 304—307. From Hoppe-Seyler's Zeits. f. Physiolog. Chem. 1897, **23**, 175.

The author considers that the cytase which, according to Brown and Morris, is present in germinating barley, does not exist. The barley only develops diastase, which is able to dissolve cellulose or hemicellulose. The diastase of germinating barley possesses the property of dissolving certain very easily hydrolysable hemicelluloses, and by warming to 60° C. the author finds this property is weakened but not completely annulled. The hemicelluloses, which are easily hydrolysed, are very widely distributed in the vegetable kingdom: they connect the cell walls of the mealy portion of the barley; they form the middle layers in the parenchyma of the potato tuber and carrot, and the walls of the young parenchymatous cells of germinating maize. There are also numerous hemicelluloses which are not attacked by the diastase of germinating barley. The walls of the aleurone granules of barley contain, besides small quantities of cellulose, a large amount of certain hemicelluloses. The grains in which these hemicelluloses are stored up as reserve material, in the form of the thickening of the cell walls, show, on germination, an enzyme different from malt diastase, and which might be known as cytase.—J. L. B.

Globulin, Effect of Minute Quantities of Acid on the Solubility of, in Salt Solutions. T. B. Osborne and G. F. Campbell. J. Amer. Chem. Soc. 1897, **19**, 482.

See under XVIII. A., *page* 820.

Diastase, The Solvent Action of, on Ungelatinised Starch. E. v. Sigmond. Wochenschr. f. Brau 1897, **14**, 412.

A repetition of Lintner's investigations (this Journal, 1890, 402) on the same subject, and carried out in the same way, the object being to determine the comparative resisting power of the starches (purified) from various cereals, to the solvent action of cold-water extract of malt, at temperatures ranging from 50°—65° C. In the first place, it is stated that Lintner's *modus operandi* leads to unsatisfactory results, on account of the starch tending to form partially gelatinised lumps. A continuous stirring of the mash is essential in preventing this "balling." The author's results differ from Lintner's, chiefly in the order of stability of the various starches. Thus maize starch is found to be far more readily dissolved than rice starch at 60° and 65° C.; whilst Lintner brackets the two as being equally resistant. Similarly, in opposition to Lintner, the author finds that rye starch is considerably less stable at 55° and 60° C. than wheaten starch.—H. T. P.

Caroubinase: A New Hydrolytic Enzyme. M. J. Effront. Comptes Rend. 1897, **125**, 116—118.

The carbohydrate isolated from the seed of *ceratonia siliqua* (this Journal, 1897, 749) is readily hydrolised by dilute acids and by the enzyme *caroubinase*. This enzyme is formed during the germination of the seed of the carob, and the transformation of the carbohydrate (caroubin) and the secretion of the enzyme may be observed on allowing the isolated embryo of the seed to germinate in the presence of caroubin. The liquefaction and absorption of the latter take place much more rapidly when chlorophyll is formed in the plant. Caroubinase acts energetically at 40°, and its action increases with a rise in the temperature to 45°—50°, which may be regarded as the optimum temperature; at 70° its action becomes very feeble, and at 80° the enzyme is destroyed. Its action is very feeble in a neutral medium, and is accelerated by the addition of 0·01 to 0·03 c.c. of formic acid to 100 c.c. of the liquid.—A. K. M.

Cider, Salicylic Acid and Calcium Sulphite as Preservatives of. E. H. S. Bailey and C. M. Palmer. Kansas University Quarterly, 1897, **6**, [3], 111—116.

In order to determine the preservative action of salicylic acid and calcium sulphite, the following experiments were made:—Salicylic acid, in quantities varying from 1 part in 500 to 1 part in 20,000, was added to separate lots of fresh cider, which were then set aside at temperatures ranging from 12° to 22°. Portions of the cider were distilled at the intervals given in the table at the head of the next page, and the alcohol determined. With the last of these determinations the acidity was also estimated, and the acid was again determined two months later.

Hydromel ["Mead"], or Honey Wine. J. Graftian. Bull. Assoc. Belge des Chim. **11**, [3], 82—97.

Since honey contains on an average between 80 and 85 per cent. of fermentable sugars, 1 kilo. will yield 480 c.c. of alcohol, and, consequently, to obtain 1 litre of alcohol, 2·08 kilos. of 80 per cent. honey must be taken; so that to prepare 100 litres of honey wort it is necessary to employ x 2·08 kilos. of honey, x representing the percentage by

Strength.	Alcohol.					Acetic Acid.	
	Interval.					Interval.	
	24 Hours.	72 Hours.	8 Days.	22 Days.	52 Days.	52 Days.	112 Days.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
No. 6. Blank	0·3	1·0	3·3	5·9	3·2	3·60	7·38
„ 1. 1 in 20,000	0·3	0·8	3·2	5·9	1·5	5·80	7·38
„ 2. 1 „ 10,000	0·3	0·7	3·2	6·0	2·3	5·00	7·87
„ 3. 1 „ 5,000	0·3	0·5	2·7	6·0	2·6	4·70	7·79
„ 4. 1 „ 1,000	0·3	0·3	0·3	4·0	4·8	0·40	0·40
„ 5. 1 „ 500	0·3	0·3	0·3	0·4	0·4	0·58	0·60
„ 6A. Blank	1·0	1·2	2·3	5·3	6·3	1·60	3·48

A similar series of experiments with calcium sulphite gave the following results :—

Strength.	Alcohol.						Acetic Acid.	
	Interval.						Interval.	
	24 Hours.	72 Hours.	8 Days.	22 Days.	52 Days.	112 Days.	52 Days.	112 Days.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
No. 1. 1 in 250	0·3	0·3	0·2	0·6	0·4	3·9	0·57	0·41
„ 2. 1 „ 500	0·3	0·3	0·3	0·3	5·4	3·9	0·48	0·41
„ 3. 1 „ 1,000	0·3	0·3	0·3	3·9	6·1	3·9	0·41	0·41
„ 4. 1 „ 2,000	0·3	0·4	0·9	4·8	6·3	4·0	0·41	0·41
„ 5. 1 „ 4,000	0·3	0·6	1·5	5·0	6·2	2·2	0·41	3·00
„ 6. Blank	1·0	1·2	2·3	5·3	6·3	2·2	1·62	3·48

—A. K. M.

volume of alcohol desired in the finished hydromel. Moreover, owing to the absence of materials suitable for the nutrition of yeast, it is necessary to add a yeast food similar to that prepared for the purpose by Gastine (Comptes Rend. **109**, 479; this Journal, 1889, 910); otherwise the fermentation will be incomplete. There is no direct evidence that the ancients made or did not make any additions for this purpose; but their honey was less pure, and the fermenting vessels were put to other uses; hence a sufficiency of yeast food might easily be present. An addition of tartaric acid, to supply the requisite acidity for for the development of the yeast, and of tannin for clarifying and preserving the wine, is also advantageous. Other conditions necessary to the normal course of fermentation are: pure water, clean vessels, absence of bad smells, the use of high-class yeast, maintenance at suitable temperature, and a sufficient degree of aeration.

From fermentations made on a small scale by the author at Hay in 1895–1896, with a wort containing 25 per cent. of honey and 5 grms. of Gastine's yeast food per litre, the temperature being maintained at 25° C. (except one sample, kept at 10° C.), it appears that honey wort, whether sterilised or not, becomes infested with mould and ferments if exposed to the air; that cold restricts the fermentation; that the flavouring influence of the different yeasts employed was in this case masked by a bitterness of unknown origin, and not observed on other occasions; and that the proportion of honey was too low: the worts containing an addition of dried raisins or an extra amount of heather honey (30 per cent. in all) giving the best results—a circumstance explicable by the residual extract imparting fulness to the flavour.

In making a hydromel from honey and raisins, the yeast was prepared by exposing 500 grms. of white sultana raisins in 750 c.c. of sterile water to a temperature of 20°—25° C. for five days, with due precautions against infection. The wort was obtained by adding 5 kilos. of the same raisins and 22·5 kilos. of white honey to sufficient warm water to make a total volume of about 70 litres, which, after the solution of the honey, was pitched with the above leaven, and left to ferment at 20°—25° C. in closed vessels. At the end of three weeks, fermentation had subsided, and the first racking was performed, the pulp being pressed and the runnings returned to the bulk. Fermentation then proceeded actively for another week, and gradually terminated, whereupon 5 grms. of tannin were mixed in with the liquid, which was then bunged and placed in a cellar, where, after a second racking, it remained for a year, and was bottled. The resulting hydromel was between Madeira and Champagne in colour, full, of very fine flavour, and contained 13 per cent. of alcohol.

The author recommends the addition of honey to the juice of several varieties of fruit too poor in sugar to produce beverages rich in alcohol though containing an ample supply of yeast food, and he gives the following particulars of the sugar content of each, with the amount of honey to be added per 100 litres of juice :—

——		Apple.	Pear.	Plum.	Sweet Cherry.	Strawberry.	Raspberry.	"Myrtille."	Large Gooseberry.	Small Gooseberry.	Raisins.
Sugar	Per Cent.	10—18	18	6·5	10	6·5	4	5	7	6·5	15
Added honey	Kilos.	20—12	12	23	20	23	26	25	23	23	15

He has also prepared a hydromel with malt wort, 2 litres; honey, 5 kilos.; water, 15 litres; tartaric acid, 15 grms., which fermented satisfactorily, and yielded a very good beverage.—C. S.

Wine, Presence of Acarina (Mites) in. L. Mathieu. Comptes Rend. 1897, **125**, [8], 100—101.

Trouessart (Comptes Rend. 1897, 364) has pointed out that the sediment of wines produced from dried grapes occasionally contains the remains of an acarina (*Carpoglypus passularum*). The author has frequently observed such remains, entire or fragmentary, in wines of undoubted purity (*i.e.*, not produced from raisins), especially in white wines grown in the Champagne and Bordeaux districts. Two species of these mites have been identified (*Glyciphagus cursor*, Gervais, and *Tiroglyphus farinæ*, de Geer), being found in considerable numbers, and multiplying freely, in the growth of mould developed on the

corks of bottles stored horizontally in a dark situation. In a test tube, containing 2—3 drops of wine, these mites also develop rapidly, being able apparently to utilise directly the extractives of the wine for their sustenance. The remains of 50 mites, when macerated for several months with $\frac{1}{4}$ litre of wine, did not impair its flavour in the least. The author concludes that the presence of acarina in a wine does not necessarily indicate its production from raisins. He is of opinion that their presence in the storage cellar may possibly bring about the dissemination of, and contamination of the wines with, *Mycoderma vini*. —H. T. P.

Wine that has lost its Colour ("Casse"), Absorption of Oxygen in. J. Laborde. Comptes Rend. **125**, [4], 248.

When wines that have deteriorated in, or lost colour ("casse") are repeatedly agitated by the injection of a known volume of air, absorption continues during four to eight days, and then ceases, the amount of oxygen taken up and of CO_2 formed, being in three instances as follows:—

Sample.	O absorbed per Litre.	CO_2 formed per Litre.	Ratio $\frac{CO_2}{O}$
	C.c.	C.c.	
No. 1	51·8	32·4	0·63
„ 2	81·0	38·0	0·47
„ 3	110·2	63·8	0·58

The $\frac{CO_2}{O}$ ratio, which is variable, and is greatest in the initial stage with wines much off colour, is always less than unity.

The effects of "casse" may be artificially produced by inoculations with laccase or with diastatic liquid from *Botrytis cinerea* (this Journal, 1897, 156), and this liquid is itself capable of absorbing 49·6 c.c. of oxygen and producing 13·8 c.c. of CO_2 per litre—*i.e.*, it acts very similarly to wine off colour, and, like the oxydase of the latter, thereby loses its oxidising power, probably as a result of the combustion of its elements.

Heat decreases the activity of the oxydase, the absorption of oxygen being found to be reduced to the normal when a wine liable to much deterioration through "casse" was heated in bottle to 75° C., though at 60° C. the oxydase remained unaffected.

In the proposed remedial SO_2 treatment (Bouffard), the absorption of oxygen remains practically the same as before, and since it is found that some of the SO_2 is still unoxidised when absorption has ceased, it would appear that oxydase has a superior affinity for oxygen, and also that the undoubted efficacy of the SO_2 is neither due to its oxidisability nor to destruction of the oxydase.—C. S.

Sweet Wines, Determination of Glycerin in. G. Fabris. Ann. del. Lab. Chim. Centr. delle Gabelle, III., 225.

See under XXIII., *page* 830.

PATENTS.

Pure Yeasts. A New or Improved Method of and Apparatus for the Manufacture of. G. E. Jacquemin, Malzéville, France. Eng. Pat. 21,011, Sept. 22, 1896.

About a hectolitre of wort, or other fermentable liquid containing matter suitable for the nutrition of yeast, is carefully sterilised, cooled to 28°—30° C., and passed into a second sterilised vessel employed for the production of the first yeast. The selected pure yeast is introduced at the opening of the thermometer, the latter replaced, and the contents of the vessel aërated. When the fermentation is proceeding actively, the liquor is run into a third receiver of larger capacity than the second; sterilised wort is subsequently added and an augmentation of yeast effected. This product is aërated for about 12 hours, and a certain quantity drawn off into a fourth receiver, which is supplied with sterilised wort. In this way a third yeast is obtained, which, as soon as made, is directed according to the requirements of manufacture into fermentation vats. The quantity of liquor containing yeast taken from the third receiver is replaced by an equal volume of sterilised wort.—J. L. B.

Fruit Juices (Fruit and Grape Wines), Process for the Production of Non-Alcoholic or Feebly Alcoholic, which may be Kept or Preserved without Change. H. Müller-Thurgau, Waedensweil, Switzerland. Eng. Pat. 978, Jan. 13, 1897.

After the fruit juice, freed from skins and stones, is heated to 60° or 70° C. for half an hour in a closed vessel, it passes into a sterilised settling vat; the juice is then filtered, and sterilised by heating to 60°—70° C. If the product is to be naturally coloured, the juice is heated and passed through a receiver containing the skins, the subsequent treatment being the same as above. On the other hand, the juice and skins are heated together, the latter separated, and the filtered liquor sterilised. Should a small amount of alcohol be necessary, fermentation is allowed to proceed to the desired extent prior to the first heating.

—J. L. B.

Wine, Beer, and Other Liquids; Protecting Bottles and their Contents against Injury or Loss during the Operation of Sterilising of Pasteurising; A Method or Means of. C. Ameye, Iseghem, Belgium. Eng. Pat. 16,091, July 6, 1897.

A double wire, bent over in the form of a hook at one end, is suspended inside the neck of the bottle before the cork is inserted; the minute passage thus formed permits the escape of steam, carbonic acid, &c., and prevents the accumulation of pressure within the bottle. After sterilisation is finished, the wire is withdrawn and the cork pushed home.—L. A.

XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

(*A.*)—FOODS.

Lupin Seeds, The Proteids of. T. B. Osborne and G. F. Campbell. J. Amer. Chem. Soc. 1897, **19**, 454—482.

Both the yellow and blue lupin have long been cultivated in Europe, on account of their readiness to flourish on sandy or gravelly soils. By their help, poor or "worn out" land has been rendered fit for agricultural purposes, as these plants furnish much fodder, and when ploughed in green, they rapidly impregnate the soil with humus.

Ritthausen (J. Prakt. Chem. **103**, 78; *ibid.*, New Series, **24**, 222, and **26**, 442) found the characteristic proteid of yellow and blue lupin seed to be conglutin; his preparation from the yellow lupin contained 1 per cent. of sulphur, whilst that from the blue variety contained 0·5 per cent.; in other respects the two conglutins were similar. This author also obtained a body which he believed to be legumin.

The authors find that both yellow and blue lupin seeds contain very little proteid matter soluble in water, that of the yellow lupin amounting to 0·37 per cent. A part of this consists of proteose, but whether the remainder is albumin or a globulin soluble in very dilute salt solutions, was not determined. Peptone is not contained in the freshly ground seed, but is formed in small quantity after prolonged contact with water. The greater portion of the proteid matter contained in these seeds is soluble in saline solutions, the yellow lupin yielding 26·2 per cent. of conglutin. Preparations from the blue lupin are usually much purer than those from the yellow, for the latter contain a considerable quantity of a substance containing sulphur, from which conglutin may be separated by fractional precipitation of dilute salt solutions. This explains why Ritthausen's conglutin (*loc. cit.*) from the yellow lupin contained twice as much sulphur as that from the blue lupin. When purified, no difference in properties and reactions could be detected between preparations from the two seeds.

The conglutin from the yellow and blue lupin were found to contain carbon, 50·91, 51·13; hydrogen, 6·88, 6·96;

nitrogen, 17·93, 18·11; sulphur, 0·52, 0·32; oxygen, 23·76, 23·58 per cent. respectively. Conglutin is easily soluble in sodium chloride solutions containing above 5 per cent. of salt. On dilution it is precipitated. In a salt solution it is apparently unaffected by prolonged heating in a boiling-water bath; but, on standing, a solid opalescent jelly is formed, which becomes clear and fluid on again heating. Unlike other globulins, conglutin does not yield insoluble (coagulated) products by washing with alcohol after drying.

The preparation of proteid soluble in 0·2 per cent. potash, gave the following numbers on analysis:—Carbon, 51·40; hydrogen, 6·79; nitrogen, 16·13; sulphur, 1·03; oxygen, 24·35 per cent. Owing to the insolubility of this substance in any medium but alkalis, and the difficulty of making pure preparations, nothing further was learned respecting it.

—J. L. B.

Starch Manufacture, The Digestibility of certain By-Products of. B. Schultze. Jahresbericht der agr. Vers.-Station zu Breslau, 1896.

By artificial digestion with gastric juice and pancreas secretion, it was found that 98 per cent. of the proteids of gluten meal, obtained in the manufacture of wheat starch, and 76 per cent. of the proteids of molasses pulp (80 per cent. of potato pulp and 20 per cent. of molasses) were rendered soluble. These two substances are therefore useful foods.

—A. L. S.

Cow Pea, The Proteids of the (*Vigna Catjang*). T. B. Osborne and G. F. Campbell. J. Amer. Chem. Soc. 1897, **19**, 494—500.

As a result of these experiments the authors conclude that the principal proteid of the cow pea is a globulin, much resembling the legumin of the pea and vetch, but differing from them in composition and properties. It is named vignin. On analysis this proteid was found to contain carbon, 52·64; hydrogen, 6·95; nitrogen, 17·25; sulphur, 0·50; oxygen, 22·66. Besides vignin, the cow pea contains a globulin having the composition and—so far as could be determined—the properties of phaseolin, which occurs in the kidney bean (*Phaseolus vulgaris*), and the adzuki bean (*P. radiatus*).

The cow pea contains also a third globulin, soluble in very dilute salt solutions, which could only be partially precipitated by dialysis in water, but completely, in the coagulated form by dialysis in alcohol. This substance closely resembles in properties and composition, bodies obtained from several other leguminous seeds. On analysis this substance gave: carbon, 53·25; hydrogen, 7·07; nitrogen, 16·36; sulphur, 1·11; oxygen, 22·21 per cent.

—J. L. B.

Adzuki Bean, Proteid of the White Podded (*Phaseolus Radiatus*). T. Osborne and F. Campbell. J. Amer. Chem. Soc. 1897, **19**, 509—513.

By treating the meal of this bean with 10 per cent. sodium chloride solution, and saturating the extract with ammonium sulphate, the authors have isolated a globulin, the sodium chloride solution of which, on dilution with water, and dialysis, yielded three fractions. The analysis of these preparations showed that fractional precipitation had brought about no separation, and that the globulin is identical in composition with phaseolin derived from the white bean (*P. vulgaris*). The phaseolin from adzuki bean was found to contain carbon, 52·56; hydrogen, 6·97; nitrogen, 16·45; sulphur, 0·57; oxygen, 23·45 per cent. The reactions of this globulin are identical with those of the phaseolin from the white bean.

Another proteid not precipitated by ammonium sulphate was obtained; it had the composition: carbon, 52·97; hydrogen, 7·01; nitrogen, 16·31; sulphur, 0·83; oxygen, 21·83. Proteids of similar composition, and obtained in a like manner, are found in the pea, vetch, and cow pea, and the authors believe that there is evidence of a distinct globulin which can only partly be removed from its solution by dialysis in water, but is wholly separated in a coagulated form by dialysis in alcohol.—J. L. B.

Sunflower Seed, The Proteids of the. T. B. Osborne and G. F. Campbell. J. Amer. Chem. Soc. 1897, **19**, 487—494.

Ritthausen (Pflüger's Archiv. 1880, **21**, 89) found that by extracting sunflower meal free from oil, with very dilute alkali a proteid was obtained which, according to the authors, closely resembles the globulin edestin (this Journal, 1896, 665). By extracting sunflower meal in a Squibbs percolator with alcohol of 0·820 sp. gr., an acid was isolated, which the authors affirm to be helianthotannic acid. The meal residue was air-dried, and treated with 10 per cent. sodium chloride solution. The filtered extract was saturated with ammonium sulphate, and from the precipitated proteid a substance was obtained which, on analysis, gave the numbers: carbon, 51·54; hydrogen, 6·99; nitrogen, 18·58, sulphur, 1·00; oxygen, 21·71. This preparation, both in composition and in its behaviour towards reagents, resembles edestin, and it is concluded that this is the principal proteid present in the seed.—J. L. B.

Globulin, Effect of Minute Quantities of Acid on the Solubility of, in Salt Solutions. T. B. Osborne and G. F. Campbell. J. Amer. Chem. Soc. 1897, **19**, 482—487.

The authors have obtained a globulin from the castor seed, part of which was soluble and part insoluble in a solution of sodium chloride. By further treatment of the soluble portion with brine, two fractions were obtained, the one not precipitated by saturation with salt, the other insoluble in saturated brine. Two fractions were also derived from the portion of the original globulin, insoluble in 10 per cent. solution of sodium chloride. These four fractions had practically the same composition, but behaved differently towards certain reagents. When treated with a quantity of acid too small to be detected with litmus or by analysis, changes were produced in the fractions soluble in saturated salt solutions; and products resulted having the same general properties as those exhibited by the fractions insoluble in saturated brine. Similar results were obtained by acting on edestin—the proteid of the hemp seed—with very small quantities of acetic acid. The same change apparently takes place by keeping the proteid for a long time in a dry condition.

It is evident that this change to a condition in which the globulin is precipitated by salt, is an intermediate step towards the development of the insoluble form—the so-called "albuminate" of Weyl; and the authors note that by treatment with warm common salt solution the insoluble matter can be changed into the form soluble in cold salt solution, and precipitable by saturation with common salt.

Attention is drawn to the fact that the only animal globulin obtained from an acid tissue is myosin, and that this substance is not only readily precipitated by saturating with salt, but that it quickly and spontaneously changes to the insoluble form, known as syntonin.—J. L. B.

Raw and Boiled Milk, Reagents distinguishing between. R. Dupouy. Bull. Soc. Pharm. Bordeaux, March 1897; Analyst, 1897, **22**, [257], 211.

See under XXIII., *page* 828.

Caffeïne in Coffee, New Method of Estimating. E. Tassilly. Bull. Soc. Chim. 1897, **17**, [15], 706.

See under XXIII., *page* 831.

Caffeïne, Determination of, in Coffee. E. Tassilly. Bull. Soc. Chim. **17**, [15], 761.

See under XXIII., *page* 831.

(*B.*)—SANITATION; WATER PURIFICATION.

Arsenical Poisoning by Wall Papers, The Cause of. T. Bolas. J. Soc. Arts, **45**, 1897, 1101.

It has long been recognised that arsenical wall papers do serious mischief, but the work of Gosio and of Emmerling seems to have cleared up that mystery which has surrounded the matter. Certain moulds, including the very common *mucor mucedo*, have a remarkable property of decomposing arsenical compounds, with the evolution of volatile products containing arsenic, and the highly poisonous character of

volatile arsenical compounds, coming into the system by way of the respiratory organs, is well known. Arsenious acid is, even in small quantities, a highly antiseptic substance and poisonous to moulds; so the throwing off of the arsenic in a volatile form may be an effort of nature to cast out the poison. The arsenical copper greens, and other colouring matters containing arsenic, are still used, and, paradoxical as it may appear, it is by no means improbable that the most dangerous wall papers are those containing a a mere trace of arsenic, since when the quantity is large the moulds cannot exist. Traces of arsenic may come into wall papers from the imperfect washing of vessels used to contain the more highly arsenical colours. Now that boric acid is very cheap, the old and perhaps forgotten suggestion of Bolley to use a precipitated borate of copper as a green pigment in place of the arsenical green deserves attention. Bolley's green is prepared by taking two parts of blue vitriol (crystallised cupric sulphate) and three parts of borax in separate quantities of cold water and mixing; after which the precipitate is washed and dried. Dyed and printed fabrics now very frequently contain traces of arsenic.

Waste Acid from Metal Works. Public Health Engineer, Oct. 2, 1897, 115.

See under X., page 801.

PATENTS.

Filtering Apparatus [*Water*]. W. Defries, 4, Danes Inn, Strand, London. Eng. Pat. 19,039, Aug. 28, 1896.

The filter is provided or connected with an air-tight chamber for the filtered water, and with a pump arranged to exhaust air and draw water from the chamber. The pump lever may be arranged to also work a second pump to supply water to the filter, &c., a ball-cock being employed to regulate the supply.—R. A.

Filtering Liquids [*Water*], *Impts. in and connected with Apparatus for.* W. Reeves and The Reeves Patent Filters Co., Ltd., both of 39, Victoria Street, Westminster. Eng. Pat. 21,805, Oct. 2, 1896.

The filter can be divided into an upper and a lower chamber (the latter containing the filtering medium) by means of a removable water-tight division or valve. When it is necessary to cleanse the filter, the valve is closed, and the upper chamber is filled with water, which passes through a valve-controlled pipe to the bottom of the lower chamber, then upward through the filtering medium, and is discharged through an overflow pipe at the top of the lower chamber. —R. A.

Tobacco, or of the Smoke therefrom, for the purpose of Increasing its Salubrious Effect on the Smoker, and in means therefor; Impts. in the Treatment of. E. T. C. Weihers, Romford, Essex. Eng. Pat. 22,192, Oct. 6, 1896.

The tobacco, the paper for making cigarettes, or a porous plug for inserting in a pipe, is sprayed with a mixture, in stated proportions, of the following ingredients: benzoic acid, tannic acid, essences of cinnamon, myrrh, juniper, aniseed, and elder flower; benzoin and spirits of wine. In the case of the plugs, borax and alum are first added thereto.—R. S.

Tanks and Filters for Precipitation and Filtration Purposes, and in the Appliances and Fittings arranged for use in the same; Impts. in the Construction of. H. L. Hinnell, Manchester. Eng. Pat. 22,503, Oct. 10, 1896.

The tank described is oblong in shape and divided into compartments by transverse weir walls. Attached to each weir wall by brackets is a vertical scum plate, which dips below the surface of the liquid and keeps back floating matters. The sewage enters the first compartment from a distributing trough (which extends from side to side, and is provided with openings at intervals), flows from compartment to compartment under the scum plates and over the weir walls, and has its exit through a channel containing a strainer formed of parallel bars or plates, on which filter cloth may be stretched.—L. A.

Filters, Impts. in [*Water*], *and in Materials or Compositions for Filtering.* G. S. Marris, Islington Stamping Works, Ladywood, Birmingham, and W. J. George, 36, Cambridge Street, Birmingham. Eng. Pat. 21,051, Oct. 28, 1896.

In a filter having the filtering material in the form of a thin disc, the disc is secured to the tubular opening between the upper and lower chambers of the filter by means of a flanged ring fixed by a bayonet joint, &c.; or, when the disc is of large size, it is supported and clamped on a flange and cross-bar by a perforated disc pressed down by a screw nut. The discs are made of an intimate mixture of cellulose or woody fibre, and animal or vegetable charcoal in fine powder, the mixture being treated by processes similar to those used in the manufacture of porous or unsized paper. The filtering material is thus obtained in the form of a tissue, which is preferably covered on both sides with unsized paper, and cut into discs.—R. A.

Purification of Water in Open Vessels, Impts. in Means for. A. Dervaux, 17, Rue Faidherbe, Lille, France. Eng. Pat. 6511, March 12, 1897.

For purifying water containing bicarbonate of lime, the water is passed through an upright vessel so as to form a downward current, and steam is led into the water at the bottom of the current so as to pass upwards through the same, the supply of water or steam being regulated so that the steam condenses before reaching the upper end of the column of water in the vessel. In the apparatus described the regulation is effected by a float or areometer, placed within the vessel and connected to the water- or steam-supply-cock.—R. A.

(C.)—DISINFECTANTS.

PATENTS.

Formic Aldehyde Vapours, Impts. in the Production of. J. J. A. Trillat, Paris. Eng. Pat. 19,741, Feb. 5, 1896. (Under Internat. Convention.)

Instead of directly transforming methyl alcohol into formic aldehyde by means of the apparatus described in Eng. Pat. 17,177 (this Journal, 1897, 695), the same apparatus may be used to vaporise formalin solution or to project it in a fine state of division into the air. To vaporise the solution, a rapid current of steam, carbonic acid, or compressed air may be employed, and a mineral salt, *e.g.* calcium chloride, may be dissolved in the solution, thus allowing it to be heated above 100° C. For spraying the solution, any of the means already in use for the treatment of vines by copper solution may be utilised. The vapours are preferably warmed in any suitable way, and then scattered into the atmosphere to be disinfected, by means of a revolving fan.—L. A.

Aromatic Fluoro Compounds, A Process for the Production of. F. Valentiner, Leipzic, Germany. Eng. Pat. 9827, April 17, 1897.

As an example of the method employed, the patentee gives the following details:—10 kilos. of phenetidine, dissolved in 22 kilos. of hydrochloric acid and 20 litres of water, are diazotised with 5·11 kilos. of sodium nitrite, and the resulting diazo chloride solution is decomposed with 30 kilos. of hydrofluoric acid. After separating the oil, it is distilled with steam and fractionated. The pure product is a yellow liquid smelling pleasantly of aniseed. It melts at 17° C. and boils at 197° C.—T. A. L.

Disinfection by Means of Formaldehyde, Impts. in or relating to. W. Loebinger, Berlin. Eng. Pat. 17,464, July 24, 1897.

Porous plates of plaster of Paris or earthenware are soaked in formaldehyde solution and suspended in the space to be disinfected, or the solution may be placed in porous flasks similarly suspended.—L. A.

XIX.—PAPER, PASTEBOARD, Etc.

PATENT.

Waterproof Paper, Fabric, and the like: A Process for the Manufacture of. A. Scheufelen, Oberlenningen, Würtemberg, Germany. Eng. Pat. 11,795, May 12, 1897.

There is claimed "the process of manufacturing waterproof paper, fabric, and the like, consisting in fixing compounds of casein on the fibres by means of formic aldehyde."

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

Molybdic Anhydride, Reduction of, by Hydrogen, and the Preparation of Pure Molybdenum. M. Guichard. Comptes Rend. 1897, **125**, 105–106.

Molybdenum dioxide, heated in a current of hydrogen, begins to lose oxygen at about 500°, but the reduction is very slow; at 550° the reduction is very distinct, and at about 600° the metallic state is reached in a few hours: no intermediate oxide is produced. Molybdic anhydride, MoO_3, is completely reduced to the metallic state in about $3\frac{1}{2}$ hours. An oxide of the formula Mo_2O_3 is not produced, and where the reduction is incomplete, the product is a mixture of dioxide and metal. In fact, there appear to be only two anhydrous oxides of molybdenum, namely, MoO_3 and MoO_2.—A. K. M.

Cerium, The Purification of. Wyrouboff and A. Verneuil. Rev. de Chim. Ind. 1897, **8**, [91], 210–212.

Cerium differs from its allied metals in having a higher oxide, Ce_3O_4, which readily forms basic salts. On this depends Mosander's method of purification—treatment with chlorine in presence of alkali—as also Debray's method of fusion with nitre. The authors consider that both these methods, which are usually considered the most satisfactory ones, are not calculated to readily yield pure cerium: for, they point out, there exists an intermediate oxide, Ce_6O_7 ($= Ce_3O_4.3CeO$), which is very stable, and also forms basic salts, and that in presence of lanthanum and didymium, more basic than cerium, a still more stable complex oxide is produced of the type $Ce_3O_4.3MO$, in which $M = Ce + La + Di$. Thus, in Mosander's method, the prolonged action of chlorine does not prevent the formation of the intermediate complex oxide, whilst in Debray's process it is produced on the first application of heat.

After studying the conditions under which the intermediate oxide splits up into the constituents Ce_3O_4, forming basic insoluble salts, and $CeO + LaO + DiO$, giving neutral soluble salts, the authors have worked out the following method of purification:—The oxides left on moderately igniting the mixed oxalates are dissolved in hot nitric acid, which causes partial reduction, and formation of the intermediate oxide. The liquid is evaporated to syrupy consistence, dissolved in water, and ammonium nitrate (to 5 per cent.) added to the hot solution. The intermediate oxide is immediately dissociated, the oxide Ce_3O_4 being precipitated as the basic salt $(Ce_3O_4)_4N_2O_5$, whilst the protoxides remain in solution. When washed with a 5 per cent. solution of ammonium nitrate, the precipitate is quite free from didymia, lanthana, and yttria. The remainder of the cerium (25 per cent.) can be recovered by adding oxalic acid to the filtrate, igniting the oxalates, and repeating the above process.

When the mixed oxides contain more than 50 per cent. of ceria, the oxalates should be dissolved in nitric acid, and hydrogen peroxide and ammonia added. The brown peroxide is transformed into the yellow ceroso-ceric hydroxide by boiling, and this, when washed, is dissolved in nitric acid and the purification carried out as before. The precipitate is still impure, as the whole of any thorium present in the original oxides is simultaneously thrown down. The cerium obtained from the filtrate, however, is quite free from it, and in any case it may be removed by a preliminary treatment of the oxalates or nitrates, as neutral as possible, with a concentrated solution of ammonium carbonate containing ammonia, which readily dissolves the thorium salt. Two treatments remove all but about 1 per cent., and this may be separated by crystallising the sulphates from a solution not containing free sulphuric acid, when the thorium sulphate accumulates in the mother-liquor, forming with cerium an exceedingly soluble double sulphate. Iron is removed by precipitating cerium nitrate or chloride from hot acid solution with oxalic acid; or by dehydrating the sulphate at 400°—450°, which leaves the iron in an insoluble state.—C. A. M.

Ammonio-Citrate of Bismuth, Examination of Trade Samples of. W. G. Stratton. Brit. Pharm. Conf. (through Pharm. J. 1897, **59**, 157).

The examination of 11 samples of the official *Liquor Bismuthi et Ammonii citratis* derived from different sources, showed that great variation existed among them, notably in the amount of ammonia and of citric acid, as shown in the following table:—

—	Sp. Gr.	Per Cent. Bi.	Per Cent. NH_3	Per Cent. $C_6H_5O_7$	Impurities.
Theoretical	1·070	4·375	..	3·956	
1	1·070	4·377	1·048	3·910	Nitrates
2	1·063	4·167	0·927	3·427	
3	1·135	4·476	3·535	14·209	..
4	1 070	4·748	0·746	3·647	
5	1·073	4·585	0·807	4·109	
6	1·073	4·548	0·871	3·817	
7	1·072	4·285	0·991	4·232	..
8	1·150	5·552	3 777	14·736	..
9	1·070	2·968	2·034	6·126	..
10	1·122	5·164	3·200	11·426	..
11	1·029	1·944	0·686	2·663	..

The large excess of ammonium citrate is probably added in some cases to render the solution more stable.—J. O. B.

Menthol Carbonate, Preparation of. H. Erdmann. J. für prakt. Chem. 1897, [13] and [14], 43.

The pyridine method is stated to be very suitable for the preparation of menthol carbonate. 30 grms. of menthol are dissolved in 30 c.c. of chloroform, and 25 c.c. of anhydrous pyridine added. The mixture is cooled by means of a freezing mixture, and 55 c.c. of a chloroformic solution of phosgene (containing 10 grms. of phosgene) slowly added with occasional shaking. After standing one day in the cold, the whole is steamed, thus getting rid of the pyridine and traces of unchanged menthol. In the residue is obtained a nearly quantitative yield of crystalline menthol carbonate, which is washed with hot water and dissolved in a large quantity of hot alcohol. The menthol carbonate, being only slightly soluble in alcohol, separates out on cooling, in the form of long, white prisms, with a constant melting point of 105° C.—A. S.

Tropeïnes of the Triacetone-amine Series. C. Harries. Annalen, **296**, 328–343.

α-Vinyldiacetonamine yields—on methylation, extraction with ether, and treatment with alkali, and subsequently with mandelic acid—a tropeïne without physiological action on the eye.

The corresponding compound of β-vinyldiacetonalkamine is prepared in a similar manner; the methylation is carried out as in the case of the α-compound, but the methylated base is easily purified, distillation of the petroleum ether extract being sufficient to obtain it in crystalline scales. The aqueous solution is strongly alkaline; the base forms a crystalline hydrochloride, chloro-aurate and platinate. Conversion into the tropeïne takes place more rapidly than with with the α-compound. The oil separated by sodium carbonate, crystallises, and is purified by recrystallisation from boiling petroleum ether. So prepared, the tropeïne forms small hexagonal prisms, which sinter at 108° and melt at 113°, and are almost insoluble in cold water.

The alkaloid placed on the pupil of the eye causes dilation; the maximum effect in man is observed after 40 minutes, and disappears after about an hour and a half.
—A. C. W.

Rhodinol, $C_{10}H_{17}.OH$; Preparation and Properties of the Pure. H. Erdmann and E. Erdmann. J. Prakt. Chem. 1897, **56**, 1–5.

Rhodinoldiphenylurethane is a well crystallised compound (H. Erdmann and Huth, this Journal, 1896, 292) melting at 82° (not at 84°, *loc. cit.*). It is, however, not possible to prepare pure rhodinol from this substance, the diphenylamine formed in its decomposition being very difficult to remove. Pure rhodinol was obtained by the addition of sodium chloride to rhodinolphthalate of silver, $C_{10}H_{17}.O.CO.C_6H_4.CO.OAg$, and decomposition of the resulting sodium salt by alkali. It is a liquid with a very pleasant smell of roses; it cannot be distilled under ordinary pressure without injury to its odour; under 16 mm. pressure it boils at 110°·5—111°. The specific gravity at 16° is 0·8812, referred to water at 4°. Warm rhodinol dissolves lithium and calcium chlorides; the compound with the latter salt crystallises, but cannot be used for the isolation of the alcohol, because impurities readily prevent its crystallisation, and when decomposed by water, chlorine compounds are formed.

If one drop of rhodinol be diluted with 5 c.c. of alcohol, and 10 drops of concentrated sulphuric acid added without shaking, the acid sinks, and takes an intense orange-yellow colour; on gently moving the vessel, the colour turns to reddish-violet at the surface of contact of the liquids. Linalool shows a similar reaction, the colour being less stable, and rapidly turning brown. Citronellol gives a feebler coloration of a bluish tint.—A. C. W.

Rhodinol, $C_{10}H_{17}.OH$; Occurrence and Detection of, in Essential Oils. H. Erdmann and P. Huth. J. Prakt. Chem. 1897, **56**, 27—42.

The smell, boiling point, and the sulphuric acid test (see preceding abstract) indicate the probable presence of rhodinol in an essential oil. 1 grm. of oil is heated on the boiling-water bath for 2 hours with 1·5 grm. of diphenylcarbamine chloride and 1·35 grm. of pyridine; the mixture is distilled with steam, and after about 1 litre of water has passed over, the residue, which solidifies on cooling, is crystallised from 30 times its weight of alcohol. In the presence of much citronellol it is more difficult to isolate pure rhodinol-diphenylurethane; crystals are obtained melting between 40° and 50° C., and apparently a molecular compound of the urethanes of the two alcohols. Only after repeated recrystallisation is the rhodinol compound obtained of the right melting point, 82° C. Under these circumstances it is of advantage to cool the alcoholic solution of the urethane to 20°. If sufficient quantities of a natural oil are under investigation, it is better to distil, and subject the fraction coming over at the boiling point of rhodinol to the treatment with diphenylcarbamine chloride and pyridine. If the oil contain no rhodinol, diphenylamine passes over with the steam as an oil, which soon solidifies. Unaltered carbamine chloride is not found in the residue after steam-distillation. Rhodinol was found in the following essential oils: Turkish and German rose oils, French, Réunion and Syrian geranium oils, ginger grass oil, oil of lemons, Turkish palmarosa oil, oil of Néroli bigarade, and Portugese oil of Néroli. The yields were very different: the Néroli oils contain very little free rhodinol; 5 grms. of Turkish rose oil, on the contrary, gave 8·3 grms. of crude urethane, which, on recrystallisation, became 4·4 grms. of almost pure rhodinoldiphenylurethane. The rose oils, when not previously fractionated, showed a peculiar behaviour; very little passes over with the steam, but the urethane has a very low melting point and great solubility; this is not entirely due to the presence of citronellol, which these oils only contain in small quantity. Perhaps they contain a third alcohol which is capable of forming a crystalline urethane.

—A. C. W.

Tertiary p-Butyltoluene [Artificial Musk] and its Nitro Derivatives. M. Białobrzeski. Ber. 1897, **30**, [13], 1773—1776.

According to the researches of Baur (this Journal, 1891, 1024, and 1891, 1218), the same tertiary butyltoluene, $C_6H_4(CH_3).C(CH_3)_3$, is obtained by the action of aluminium chloride and toluene on isobutylchloride, isobutylbromide, and tertiary butylbromide. By the oxidation of this hydrocarbon (in which the butyl group occupies the meta position) with dilute nitric acid, Baur obtained *m*-butylbenzoic acid and isophthalic acid. The trinitro derivative of *m*-butyltoluene, as is well known, is the artificial musk.

By substituting ferric chloride for aluminium chloride, the author has obtained, by the action of toluene on tertiary butylchloride and isobutylchloride, a butyltoluene (b. pt. 189°—190°, sp. gr. 0·8784 at 0°, 0·8614 at 23°) which refuses to be nitrated beyond the dinitro stage—

$$C_6H_2(NO_2)_2(CH_3)C_4H_9.$$

The suspicion that the butyltoluene thus prepared was not identical with the *m*-butyltoluene obtained by Baur, was confirmed by oxidising it with chromic acid, when *p*-butylbenzoic acid of melting point 164° was formed, and this, on being heated with dilute nitric acid in a sealed tube, was converted into terephthalic acid. It therefore follows that by the action of toluene on tertiary butylchloride, *m*-butyltoluene is formed in the presence of aluminium chloride, whilst *p*-butyltoluene is formed in the presence of ferric chloride. And since in the latter hydrocarbon the para position is already occupied, it is impossible to introduce more than two nitro groups, or to prepare artificial musk from it.—J. S.

"Dulcine," or Paraphenetol Carbamide. R. Raggeri. Ann. del. Lab. Chim. Centr. delle Gabelle, III., 138.

See under XXIII., *page* 828.

Carbon Bisulphide, Estimation of, in Alcohol, Carbon Tetrachloride, &c. Purification of Commercial Carbon Tetrachloride, and a New Reaction for the same. W. Schmitz-Dumont. Chem. Zeit. 1897, **21**, [50], 487 and 516.

See under XXIII., *page* 829.

PATENTS.

Condensation Product [Anti-neuralgic] from Vanillin and p-Phenetidine, Preparation of. C. Goldschmidt, Frankfort-on-Maine, Germany. Eng. Pat. 14,954, July 6, 1896.

The preparation of a condensation product—

$$(OCH_3)(OH).C_6H_3.CH.N(C_6H_4)OC_2H_5,$$

by heating vanillin with *p*-phenetidine at 140° C., is claimed. The product melts at 97° C., dissolves in cold water, is only slightly poisonous, and exerts, it is claimed, an anti-neuralgic and soporific action.—A. C. W.

Hair Lotions, A New or Improved Manufacture of. J. Herbst, Munchelberg, Germany. Eng. Pat. 17421, Aug. 6, 1896.

The constituents of the lotions claimed, are, a solution of quinine in benzene, tincture of cantharides, balsam of Peru, gum benzoin, castor oil, and alcohol.—A. C. W.

Oxy-Camphor, Improved Manufacture of. O. Imray, London. Farbwerke vorm. Meister Lucius and Brüning, Hoechst a/Main, Germany. Eng. Pat. 18,035, Aug. 14, 1896.

The manufacture of an oxy-camphor—

$$C_8H_{14}\left\langle\begin{array}{l}CO\\ |\\ CH.OH\end{array}\right.$$

by reduction of camphor quinone in acid, neutral, or alkaline solution, is claimed. The substance melts at 203°—205° C., and is tolerably soluble in water.—A. C. W.

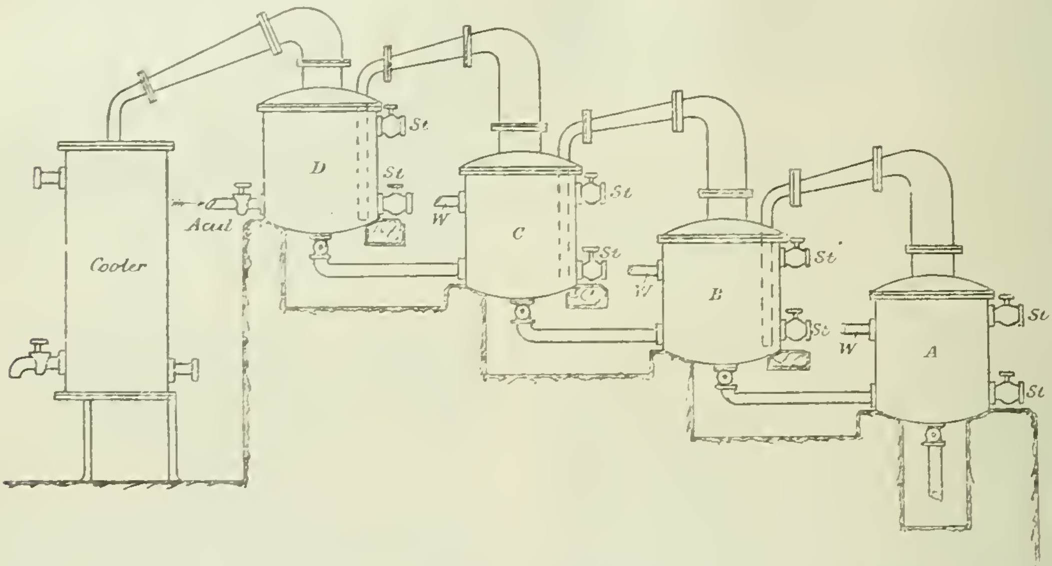
Oxygen, Extracting, from the Air; Process and Apparatus for. A. F. de Villepigue, Rue de la Victoire, Paris, V. Fournier, Rue Caumartin, Paris, and G. Shenton, Copthall House, Copthall Avenue, London. Eng. Pat. 19,044, Aug. 28, 1896.

The air is dialysed through a series of "membranes of tissue" treated with caoutchouc, the proportion of oxygen in the air being increased by the consecutive passages of the air

through the membranes, until it becomes sufficiently pure for practical purposes. The apparatus employed, consists of a spoked wheel mounted on a tubular arbor, the centre of which communicates with one of the hollow spokes of the wheel: a series of bellows arranged round the periphery of the wheel, the fixed walls of these bellows being constructed of "a membrane of tissue" treated with caoutchouc, and so arranged that one of the bellows communicates with the above-mentioned hollow spoke, and the following bellows communicates with the atmosphere, the bellows being connected one with the other by convenient tubes. Two drums are mounted over the wheel, against the periphery of which they press in order to consecutively compress the bellows. —R. A.

Ether from Gases containing Ethylene, Obtaining, and Apparatus therefor. P. Fritzsche, Essen, Germany. Eng. Pat. 20,225, Sept. 12, 1896.

When gases containing ethylene—purified coal-gas, coke-oven gases or oil-gases—are passed through a series of wash bottles containing concentrated sulphuric acid, the hydrocarbons of high molecular weight are mainly absorbed in the first bottle, and their absorption hinders that of ethylene. Moreover, ethylene combines slowly with sulphuric acid at ordinary temperatures, the most favourable temperature is 110°—120°, at which the decomposition of the resulting ethylsulphuric acid is slight. The patentee claims the method for obtaining ethylsulphuric acid, which consists in treating gases containing ethylene with cold



concentrated sulphuric acid, and then with the same substance, or its mixture with ethyl sulphuric acid, at 100°—140° C.

Each of four vessels, A, B, C, and D, is charged with the ethyl sulphuric acid, the contents of A being diluted with 35 per cent., of B with 22 per cent., and of C with 10 per cent. of water, whilst the acid in D is undiluted. The vapours of alcohol, ether, and water pass from each vessel into the one above; ether and water alone are evolved from D; these are condensed. The process is continuous, a current of acid flowing down the series, diluted in A, B, and C by a constant stream of water. The vessels may be heated, as by a steam coil in each vessel having its inlet and outlet *St*. This process is claimed as described.—A. C. W.

Acetosulphanilate Salts [Antipyretics], Manufacture of Pure. F. Hoffmann, La Roche and Co., Basle, Switzerland. Eng. Pat. 29,417, Dec. 22, 1896.

The patent claims the alkaline acetosulphanilates and the process for their manufacture, which consists in boiling sodium sulphanilate with glacial acetic acid, distilling off excess of acid, dissolving in a little water, filtering from sulphanilic acid, precipitating and washing with strong alcohol.—A. C. W.

Thorium Oxide, New or Improved Process for Obtaining, and for Enriching Monazite Sands. W. Buddeus, Berlin, Germany. Eng. Pat. 9996, April 21, 1897.

The process, the various stages and modifications of which are described in the 12 claims, is substantially as follows:—The sand, crushed to such a fineness that the grains are $\frac{1}{4}$—$\frac{1}{2}$ mm. in diameter, is introduced into molten potash or soda at 400°—500° C.; water is added, dissolving the alkaline phosphate and excess of hydrate. The washed residue is made somewhat alkaline and stirred with water; the lighter oxides are suspended and washed away from ferrous titanate and zircon. These oxides are allowed to settle, and treated with an acid (sulphurous acid), which will dissolve the greater part of the ceria, leaving all the thoria.—A. C. W.

Tribenzoyl-gallic Acid, Process for the Production of. L. Landshoff and P. Meyer, Grünau, Germany. Eng. Pat. 10,041, April 22, 1897.

The claim is for the process, which consists in shaking an alkaline solution of gallic acid with benzoylchloride, boiling with water, and purification by recrystallisation. Tribenzoyl-gallic acid is insoluble in water, fairly soluble in hot alcohol, less in cold; is not decomposed by exposure to air or treatment with water.—A. C. W.

Alkoxylated Phenols, A Process for the Production of Indifferent (Neutral) Compounds of. L. Lederer, Munich, Germany. Eng. Pat. 12,963, May 25, 1897.

The patentee claims the process for the production of neutral esters of alkoxylated phenols, which consists in causing alkoxylated phenols in the presence of potassium carbonate or their compounds with potassium carbonate (this Journal, 1897, 758) to react with phosgene, chlorocarbonic esters, acid chlorides, &c. By the introduction of radicals, so as to neutralise the OH group, in the alkoxylated phenols, it is said, "their therapeutic usefulness is increased."—A. C. W.

Ointment, Unguent, or Salve, A New. J. C. Mewburn, London. From The Chemische Fabrik von Hayden Gesellschaft mit beschraenkter Haftung, Radebeul, near Dresden, Germany. Eng. Pat. 16,869, July 16, 1897.

The claim is for "an ointment, unguent, or salve of which the characteristic feature or element is a soluble form or modification of silver in the proportion of 1 to 30 per cent. of the silver to 70 to 99 per cent. of a suitable grease or carrier."—C. A. M.

Therapeutical Compounds [Bismuth Oxyiodide and Tannin], Manufacture of New. Society of Chemical Industry in Bâsle, Switzerland. Eng. Pat. 17,409, July 23, 1897.

In this patent are claimed compounds of bismuth oxyiodide with tannin, another tannic acid or a compound of formaldehyde with tannin, and the process for obtaining these substances, which consists in dissolving one or two molecules of a bismuth salt in dilute acid, adding sodium acetate and a solution of one molecule of tannin and one or two molecules of potassium iodide, and treating the precipitate with sodium bicarbonate.—A. C. W.

XXI.—PHOTOGRAPHY.

Photographic Plates, Action of Metals and other Substances on. W. J. Russell. Proc. Roy. Soc. 1897, **61**, 424—433.

Zinc, laid on a photographic plate in the dark for a week or more, acts on the plate so that, when developed, a dark picture of the zinc is obtained. The metal must be clean, and if it be rubbed with coarse sand-paper the picture shows every scratch made. Other metals produce similar results, mercury being the most active, while magnesium, cadmium, zinc, nickel, aluminium, pewter, fusible metal, lead, bismuth, tin, cobalt, antimony, follow in order. Moisture has no influence on these results, but the action is much stronger and more rapid at high than at low temperatures. Other bodies were also found to produce these results—copal varnish, in a high degree, wood of various kinds, charcoal, printing ink of some kinds, strawboard, and certain brown papers. Among liquids, methylated spirit, turpentine, and oil of cloves gave slight effect; other liquids tried were without action.

Contact is not necessary for the production of these effects; and the action will take place, not only through air, but through screens of various materials, such as celluloid, gelatin, gutta-percha tissues, collodion, parchment and vegetable parchment, gold-beaters' skin, tracing paper, &c. Glass, however, is opaque to the action, and different writing papers show great differences in regard to it. Gelatin may be coloured, or paper may be painted with colours or with Indian ink, without any effect on their transmissive powers.

Further experiments are in progress with the view of discovering the cause of these phenomena.—J. T. D.

Silver in Silver-plating Solutions, Estimation of. T. J. Baker. Chem. News, 1897, **76**, 167.

See under XXIII., *page* 828.

PATENTS.

Ornamenting Objects of Metal, Porcelain, or the like by Photography: A Process for. T. Häusermann, Vienna. Eng. Pat. 14,927, July 6, 1896.

The patent claims "a process for ornamenting objects of metal or porcelain in colours from brown to black, consisting in coating such objects with chrome glue, then exposing the same to light by the aid of a negative or template pattern, and, after washing, heating to 300—400° C."—A. C. W.

Permanent Photographs on China and other Vitreous Surfaces, A New or Improved Method or Process of Producing. J. Slater, Burslem. Eng. Pat. 21,081, Sept. 23, 1896.

A glass plate is coated with nitrocellulose, 100 grains, alcohol, $2\frac{1}{2}$ oz., and ether, 10 oz.; and sensitised in gum arabic, 100 grains; sugar, 60; potassium bichromate, 80 grains; methylated spirit, 1 oz.; and water, 7 oz. It is exposed under a positive, dusted over with enamel colours "in accordance with the lights and shades or natural colouring of the object photographed," washed till free from bichromate, stripped from the support, and laid on the plaque. This is fired as usual, then painted and again fired, and a second film, prepared and dusted exactly as before, superimposed on the image, and burnt a third time. Additional films can be adjusted on the enamel and also burnt in, if so desired. It is claimed that the pictures possess a more life-like tone and a better finish than such as are produced in the ordinary manner.—F. H. L.

Hyposulphites [Thiosulphates], New or Improved Process for removing, from Photographic Negatives and Prints. P. Mercier, Paris. Eng. Pat. 12,182, May 20, 1897.

To remove hyposulphites (thiosulphates) after fixing, this patent claims the use of solutions of alkaline bromides or iodides, with or without the addition of alkaline substances (sodium carbonate); also of an aqueous solution of bromine or iodine, mixed with an alkaline solution and decolorised by heating or other means.—A. C. W.

Prints similar to Photographic Prints, An Improved Chemical Method of Producing. H. Soar and J. W. Cooper, London. Eng. Pat. 13,751, June 4, 1897.

In prints prepared by this process, the material of which the image is formed consists of a metal deposited by purely chemical means instead of by the action of light. This is effected by bringing together, under pressure, a damp sheet of paper containing a salt of some easily reducible metal (such as the gelatino-chloride "printing-out" papers of commerce), and a specially prepared metallic block (made of zinc, bronze, &c.) in which the shadows of the picture are represented by bare metal, whilst the lights are protected by a resist. In presence of the moisture, a reaction between the zinc and the salt of silver sets in, determining the precipitation of the latter metal in the pores of the paper. Other substances besides silver may also be employed, provided they will yield a coloured deposit: salts of gold, lead, nickel, or iron may either be introduced into the emulsion with which the paper is coated, or they may be added to the water with which it is damped.

The plate is prepared by any of the known photo-engraving methods, carrying the process up to the point where it is, in the ordinary way, ready for immersion in the acid bath. A block may also be produced in bichromated gelatin on a glass or similar non-metallic support, being developed in bronze or other metallic powder by "dusting-on." Or, a metallic plate may be coated with a resist and engraved or otherwise cut through mechanically, to give the necessary design.—F. H. L.

XXII.—EXPLOSIVES, MATCHES, Etc.

PATENTS.

Explosive Compounds for Blasting Purposes, Impts. in. J. C. Hamilton, Arnside, Lancaster. Eng. Pat. 22,162, Oct. 6, 1896.

Oxalate of urea (about 15 per cent.) is added to the explosive—either black gunpowder or nitroglycerin class; this addition is said to render these flameless, or reduce the amount of flame during combustion.—W. M.

Denitrating Acid which has been used in the Nitroglycerin Manufacture, An Improved Process for. G. Kynoch and Co., Ltd., and A. T. Cocking, Witton, Staffordshire. Eng. Pat. 22,718, Oct. 13, 1896.

In this invention the waste acid is allowed to flow from a reservoir into a small vessel in which some hot water has been placed. The mixture of acid and water generates sufficient heat to start the decomposition of the nitrated bodies in the acid, and the heat is maintained by the decomposition of these bodies. The purified acid is allowed to flow out of the small vessel, the proper rate of in- and outflow being found by experiment.—W. M.

Explosives, Improved Manufacture of. M. Bielefeldt, Wittenburg, Saxony. Eng. Pat. 18,034, Aug 14, 1896.

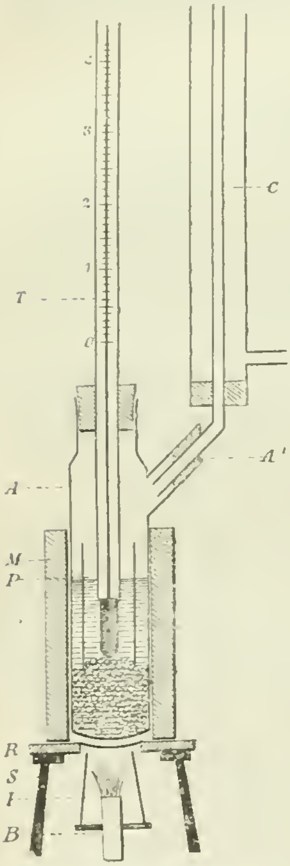
The claim is for an explosive composed preferably in the following proportions: ammonium nitrate 100 parts, potassium nitrate 1·5 parts, and resin 5·5 parts, the ingredients of which have been intimately mixed and then heated until only the resin begins to melt.—W. M.

XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

Boiling-Point Apparatus, for Use with Low- and High-Boiling Solvents. H. C. Jones, Amer. Chem. J. **19**, 581—597.

A glass vessel, A, 18 cm. high and 4 cm. diameter, has its neck narrowed to $2\frac{3}{4}$ cm., and ground to take a glass stopper. Glass beads are dropped in to a depth of 3 or 4 cm., and a cylinder, P, of platinum foil, 8 cm. high and $2\frac{1}{2}$ cm. diameter, is placed in and pushed in so that the beads steady it. Small squares of platinum foil about 7 mm. in the side, and with their corners turned up so that they do not lie flat on one another, are then dropped in so as to cover the beads inside the platinum cylinder. On an iron tripod, S, 8 cm. diameter, is a ring, R, of asbestos, 9 cm. outside and $3\frac{1}{2}$ cm. inside diameter. On this rests a circle of fine copper gauze, which is kept in position by the asbestos cylinder M, 12 cm. high, $1\frac{1}{2}$ cm. thick, made by wrapping asbestos paper round A, and in which A will just move stiffly. A small Bunsen flame, B, protected by a shield, I, is enough for heating purposes. The side tube A, $2\frac{1}{4}$ cm. diameter, will connect with the condenser C, about 40 cm. long, the upper end of which can take a cork with a drying tube when the solvent needs to be protected from moisture.



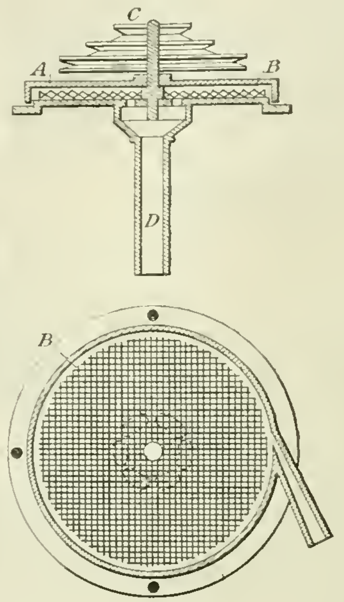
The stopper is placed in the neck of A, the side-tube corked, and the vessel weighed; the solvent is put in (in quantity just enough to avoid boiling over from inside to outside of the platinum cylinder) and the whole re-weighed. The cork carrying the thermometer is substituted for the stopper, the vessel inserted in the jacket and pushed down till the bottom is in contact with the gauze, and connection made with the condenser. Heat is applied till the liquid boils, the flame regulated so as to secure active and regular boiling, and after sufficient time the thermometer read with a hand lens. The apparatus is allowed to cool somewhat, the substance to be investigated added from a weighed tube either through the condenser or by removing the thermometer, and the boiling point of the solution now determined.

The advantages of this apparatus, beyond its simplicity, and ease of construction and of manipulation, are, that it minimises the effect of radiation, prevents the condensed solvent from touching the thermometer till it has been reheated, and renders an outer vapour jacket unnecessary. The results obtained by it show that it meets all practical demands.—J. T. D.

Motor, New Laboratory Water-. H. Tryller. Ber. 1897, **30**, 1729—1731.

The author describes a new laboratory motor, which he claims can be more easily regulated, and which uses less water than the well-known Rabe motor. When working at a low speed it produces no noise whatever, whilst at high speed it is free from vibrations, which are so apt to throw off the driving cord.

The construction of the motor may be gathered from the accompanying figures. A is a shallow cylinder 100 mm. in diameter and 5 mm. high. The interior of the cylinder is almost completely filled by the disc B, consisting of a double layer of ordinary iron-wire gauze, which is firmly attached to the spindle carrying the pulley C. As in one of the older forms of turbine, the water enters at the periphery in a tangential direction, but instead of again leaving by an opening at the side, it passes towards the centre and escapes through a series of holes drilled round the axis into the exit tube D. In thus passing from the exterior to the centre, the water is compelled to flow in spiral paths, and the great resistance offered by the wire gauze B causes the disc to rotate.—J. S.



Extraction Apparatus, A New. E. Diepolder. Ber. 1897, **30**, [13], 1797—1798.

The new extraction apparatus, which is an improved form of that devised by Schwarz (Zeits. Anal. Chem. **23**, 369; this Journal, 1884, 648), is shown in the accompanying figure. The tall, narrow cylinder A, Fig. I., contains the liquid to be extracted, and has sealed into it the bent exit tube F, the longer limb of which must be so arranged as

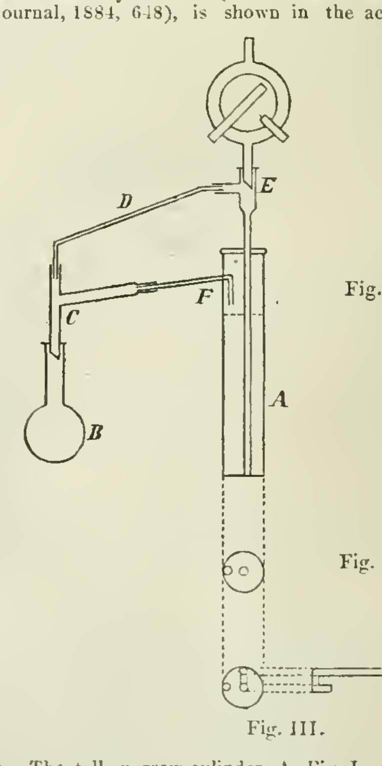


Fig. I.

Fig. II.

Fig. III.

not to act as a siphon. The surface of the liquid to be extracted must be quite free of the opening to F. The solvent is then poured into A, and when sufficient has run over into the flask B the open end of A is closed by means of a split cork. When B is heated, the vapour of the solvent rises up through D, is condensed in the spherical Soxhlet's condenser, and passes down through E. When the hydrostatic pressure of the condensed solvent in the central tube becomes sufficiently great, the solvent passes up through the liquid, extracting the soluble matter on the way, and flows over into the flask B.

In order to distribute the solvent better, the lower end of the tube E may be provided with several small openings.

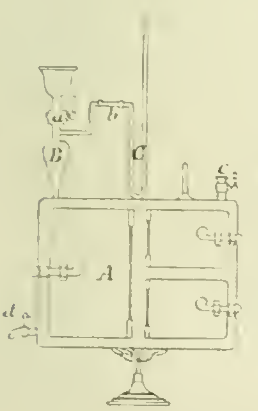
Fig. II. represents a plan of the apparatus with the tube E in a central position, whilst in Fig. III. it is shown at the side, with an upward bend at the lower end, in order to allow the use of a Witt's stirrer to facilitate the mixing of both liquids.

The apparatus as shown is for solvents which are specifically lighter than the liquid to be extracted. If denser solvents are employed, the tube F must be continued to the bottom of the vessel A, whilst E dips just under the surface of the liquid.

By heating or cooling the vessel A, the extraction may be conducted at any required temperature.—J. S.

Hot-Water Oven, A New, for Temperatures over 100° C. F. Gautter. Forschungs Ber. 1897, **4**, [6], 154—155.

The tube from which the steam escapes is connected with a barometer tube containing mercury, so that by regulating the flame, any temperature over 100° corresponding to the pressure may be obtained. A is a double-walled oven, with the usual openings for the thermometer and escape of moisture from the interior. The jacket is filled with water through the funnel B, until it runs out through the tap *c*. The latter is then closed, and the water heated to boiling, and the tap *a* closed. By the pressure of the mercury in the tube C, and regulation of the gas flame, any temperature from 100° to 110°, or higher, may easily be maintained. At the bottom of the oven there is a safety-valve, *d*. —C. A. M.



INORGANIC CHEMISTRY.—QUANTITATIVE.

Copper Sulphide, Solubility of, in Fused Alkali Polysulphides. E. Prost and A. van de Casteele. Bull. Assoc. Belge des Chim. **11**, [3], 99—102.

The summary of the experimental results detailed in this article is as follows:—

1. The amount of copper which can be converted into soluble sulpho salt by fusing a salt of copper with an alkaline carbonate and sulphur, increases rapidly with the proportion of flux used.

2. The action is more energetic when potassium carbonate is used instead of sodium carbonate in the flux (26 grms. of the latter, along with sulphur, converting 65·6 per cent. of 0·1 grm. of copper into a soluble sulpho salt, whereas 26 grms. of the former converted 98·2 per cent. of the 0·1 grm. of copper).

3. The quantity of copper converted into sulpho salt appears to be inversely proportional to the duration of the fusion, at least within certain limits. The maximum is in all cases quickly attained.

4. The presence in considerable proportion of a metal like tin, susceptible of being converted into a sulpho salt, appears to diminish the action of the alkaline sulphur flux on the copper.

5. Methods of separation in which copper is brought to the state of sulphide by fusion with alkali polysulphide, and subsequently determined after elimination by means of water of the sulpho salts of the metals of the sixth group, are to be regarded with suspicion. In certain cases under such conditions the qualitative detection of copper is not possible.—C. S.

Tin, Analysis of. E. Prost and A. van de Casteele. Bull. Assoc. Belge des Chim. **11**, [3], 103—112.

With the idea of reducing the complexity of the ordinary methods of analysing tin, and of estimating in the one sample the arsenic, antimony, lead, copper, bismuth, and iron present, the authors propose to utilise the fact applied by Clarke (Chem. News, **21**, 124) in the estimation of tin, arsenic, and antimony in alloys, *viz.*, that if a solution containing these metals be treated with hydrogen sulphide in presence of oxalic acid, only the two last-named will be precipitated; copper and bismuth are also precipitated, but lead not completely.

The method consists in dissolving 30 grms. of tin at a gentle heat, in nitrohydrochloric acid, neutralising almost completely with potassium hydroxide and adding 130 grms. of oxalic acid in solution. After making up the volume of the liquid to about a litre, a current of hydrogen sulphide is passed for at least two hours, at a temperature of 70°—80° C. The brownish-black precipitate, containing all the arsenic, antimony, copper, and bismuth present, as well as a little tin and some of the lead, is washed and treated, at water-bath temperature, with sodium sulphide to remove the sulphides of arsenic, antimony, and (stannic) tin (solution S), the residue is washed with salt water to prevent it from escaping in the colloid state, and is then dissolved in nitric acid (solution P.).

The original filtrate, which must be kept concentrated and warm, to prevent the deposition of stannic sulphide, contains part of the lead in addition to the iron; it is now treated with potassium hydrate until the stannic hydrate first formed is dissolved. Under the influence of the heat, a white metastannic compound is also thrown down, and, after washing by decantation, may (1) be dissolved along with the iron by very dilute hydrochloric acid, leaving behind the lead sulphide (P^1). The tin is thrown down from this solution by hydrogen sulphide, and the iron estimated in the filtrate. Another method (2) of dealing with the metastannic precipitate is to wash it with salt water on a filter and fuse it, after drying, with sodium carbonate and sulphur. On taking up the mass with water the sulphides of lead and iron are left and, after dissolving in nitric acid, are added to the solution P. This is then evaporated along with sulphuric acid to convert the lead into sulphate (contaminated slightly with tin). Bismuth and copper are thrown down from the filtrate by hydrogen sulphide, then purified from tin by means of sodium sulphide, re-dissolved in nitric acid, and estimated as sulphide and oxide respectively, or in any other way. Iron is estimated in the final filtrate. The residue from (1) is treated as above, except as regards the iron.

The solution S is decomposed by hydrochloric acid, the reprecipitated sulphides being filtered, washed, and flushed into a basin, dried on the water-bath, and the tin and antimony dissolved out by concentrated hydrochloric acid, leaving behind arsenic sulphide, from which, on solution in bromine water, the arsenic is precipitated in the usual manner as ammonio-magnesic arseniate, the filtrate being tested for antimony by hydrogen sulphide. The antimony is separated from tin in the above solution by nearly neutralising with KHO, then adding 10 grms. of oxalic acid and passing hydrogen sulphide. For checking the result, the precipitate may be dissolved in HCl and the antimony thrown down by a rod of tin in a platinum basin.

The tabulated results of analyses performed in the above manner on tin containing known quantities of the foreign metals in question, show that the method is accurate to within 0·001—0·005 per cent. (of total substance) in all cases except lead, where the amount escaping detection is somewhat higher, *viz.*, 0·008 to 0·013 per cent.—C. S.

Silver in Silver-plating Solutions, Estimation of. T. J. Baker. Chem. News, 1897, **76**, [1975], 167.

When estimating silver in residues, precautions must be taken to separate the copper cyanide and other impurities precipitated along with the silver chloride. To avoid this and also the formation of small globules of silver adhering to the crucible, when the impure silver chloride is fused with sodium carbonate and nitre, the following method is suggested :—Precipitate all the silver as cyanide, together with copper cyanide and other impurities, by boiling 50 c.c. of the solution with a slight excess of nitric acid. Wash and dry the precipitate, burn the filter-paper, and wrap up the ash together with the precipitate, in assay lead. Compress into a small bulk and cupel, employing the usual precautions of duplicate assays and checks. The method is said to give good results.—A. S.

Electro-plating and Gilding Solutions, Note on the Assay of. A. H. Allen. Chem. News, 1897, [1978], 199.

In the article of which the above is an abstract, T. J. Baker appears to suggest that the existing methods of analysis for the assay of plating solutions are imperfect. The author gives a method, proposed by him some 20 years ago, which he thinks better than that described by Baker.

20—50 c.c. of the plating solution are largely diluted with water and the whole raised to boiling. Sulphuretted hydrogen is passed through or ammonium sulphide gradually added. The precipitate of silver sulphide, probably contaminated with copper and zinc, is filtered off, washed, and rinsed off the filter into a flask or beaker. It is then treated with an excess of bromine water, and if any sulphur appears to have separated, a drop of bromine should be added to ensure complete oxidation. Boiling water is then added, and the silver bromide filtered off, washed, dried, fused, and weighed. 188 parts by weight of the precipitate represent 108 of metallic silver.

For the determination of the precious metal contained in the solution of the double cyanide of gold and potassium used for electro-gilding, the following method is recommended :—A measured quantity of the solution is cautiously concentrated to a syrupy consistence, in a porcelain crucible. A few grms. of pure red lead or litharge are then added, and the evaporation continued to complete dryness. The crucible is covered and raised to a moderate red heat for a short time. The resulting button of metallic lead is separated from the slag, and the gold contained in it is separated either by cupellation or by treatment with pure nitric acid.

Electro-silvering solutions may be assayed in a precisely similar manner ; but in this case the silver must be recovered by cupellation, since the nitric acid treatment is of course inadmissible.—A. S.

Mineral Phosphates, Estimation of Lime, Alumina, and Iron in. L. Lindet. Comptes Rend. **125**, [4], 246—247.

To avoid the inconveniences attendant on the usual methods of removing the citric acid from the ammoniacal liquid remaining after the precipitation of ammonio-magnesium phosphate, the author proposes to utilise Villiers' method of oxidising organic substances by nitric acid in presence of a manganese salt (Comptes Rend. **124**, 1349; see page 832). To this end, the liquid is saturated with nitric acid, and there are then added 0·5 grm. of manganese nitrate or sulphate, and about 50 c.c. of nitric acid per 20 grms. of citric acid, the mixture being gently warmed and nitric acid added at intervals, as the reaction slackens, until the evolution of gas ceases. The citric acid will thereby be decomposed, and iron and alumina may then be thrown down by ammonia, and separated from manganese in the usual manner.

Vanadyl dichloride, being more energetic, may be used in place of manganese salts, 0·1 grm. sufficing to oxidise 20 grms. of citric acid. The ammonium hypovanadate thrown down with the iron and alumina is insoluble under the conditions prevailing. In place of separating it from the two latter, the weight may be deducted from that of the calcined precipitate, to which end a 1 per cent. solution of the dichloride is used, and is titrated by precipitating 10 c.c. of the liquid by ammonia in presence of a determined quantity of ferric oxide.

Whichever oxidising agent be employed, lime can be estimated in the usual manner after the separation of the iron and alumina.—C. S.

ORGANIC CHEMISTRY.—QUALITATIVE.

Raw and Boiled Milk, Reagents distinguishing between. R. Dupouy. Bull. Soc. Pharm. Bordeaux, March 1897; Analyst, 1897, **22**, [257], 211.

Guaiacol.—Add to 1 c.c. of the sample of milk, an equal volume of a 1 per cent. aqueous solution of guaiacol, and then one drop of ordinary commercial hydrogen peroxide. An immediate yellow colour is produced with raw milk, whilst with boiled milk the mixture remains colourless.

Hydroquinone.—1 c.c. of a freshly prepared 10 per cent. aqueous solution, mixed with 3 c.c. of raw milk and 15 drops of hydrogen peroxide, gives an immediate rose coloration, and the mixture, after standing for 3 or 4 minutes, deposits green crystals of quinhydrone, $C_{12}H_{10}O_4$.

Pyrocatechol.—1 c.c. of raw milk, when mixed with an equal volume of a 10 per cent. aqueous solution of pyrocatechol, gives a yellowish-brown colour after the addition of a drop of hydrogen peroxide.

α-Naphthol.—An aqueous solution of the reagent gives a violet-blue colour with raw milk, after the addition of a drop of hydrogen peroxide. β-naphthol does not give this reaction.

Paraphenylenediamine. — This reagent gives a very sensitive reaction, and can be used with watered and skimmed milks, which, with the preceding reagents, give the colorations, but with less intensity than normal milk. Several centigrms. of the reagent are dissolved in 1 c.c. of water, and to the cooled solution an equal volume of milk and one drop of hydrogen peroxide are added. A very dark violet colour is produced with raw milk.—A. S.

"Dulcine," or Paraphenetol Carbamide. R. Ruggeri. Ann. del. Lab. Chim. Centr. delle Gabelle III., 138—145.

Paraphenetol has been introduced into commerce as a sweetening material under the various names of suciole and dulcine, especially as it has been shown that even repeated doses are innocuous to the human organism.

The first method adopted for the detection of this substance, was proposed by Berlinerblau and modified by Morpurgo, but Thoms subsequently affirmed that the reaction was not characteristic of paraphenetol-carbamide, as phenetidine salts and phenacetine equally give the same result. The method proposed by the author is as follows :—A fragment of the substance is heated in a porcelain dish with a 6 per cent. solution of silver nitrate, or with a 5 per cent. solution of mercuric chloride, operating with 2 c.c.; the whole is evaporated to dryness on a water-bath, when a brilliant violet coloration is produced, which becomes more marked if the residue is heated to 160°, especially if mercuric chloride be used. In the case of foodstuffs, the process proposed by Morpurgo for the extraction of the dulcine, gives satisfactory results. While the paper was in course of preparation, a somewhat similar method, namely, by using mercuric nitrate, was proposed by Jorissen (this Journal, 1896, 620); the author has investigated this method, and finds that 0·01 grm. of dulcine gives a very marked, but 0·001 grm. a slight but sensible reaction.
—V. H. V.

ORGANIC CHEMISTRY.—QUANTITATIVE.

Acetylene, Method of Estimating. Chavastelon. Comptes Rend. **125**, [4], 245—246.

The action of acetylene on an excess of silver nitrate resulting in the liberation of nitric acid, according to the equation: $C_2H_2 + 3AgNO_3 = C_2Ag_2.NO_3Ag + 2HNO_3$ (Comptes Rend. 1897, [24], 1364), the weight or volume of acetylene taking part in the reaction can be determined by titrating the free acid, provided the gases associated with acetylene (in mixtures) are inert towards nitric acid, and are

not themselves acid in character. The only data necessary in the calculation are: the volume, temperature, and pressure of the acetylene or mixed gas, and the final volume and acidity of the liquid.

The operation may be performed in a Raoult eudiometer, 20 c.c. of a 10 per cent. solution of silver nitrate sufficing for a 60 c.c. eudiometer, distilled water being afterwards run in until the pressure is balanced.

The method is also applicable to acetylides of the formula $R.C \vdots C.H$, the reaction in such case being expressed by $R.C \vdots C.H + 2AgNO_3 = R.C \vdots C.Ag.NO_3Ag + HNO_3$.

—C. S.

Carbon Bisulphide, Estimation of, in Alcohol, Carbon Tetrachloride, &c. Purification of Commercial Carbon Tetrachloride, and a New Reaction for the same. W. Schmitz-Dumont. Chem. Zeit. 1897, **21**, 487—488 and 510—511.

For the quantitative estimation of carbon bisulphide the following methods have been recommended: (1) Combining with triethylphosphine and weighing the red compound $P(C_2H_5)_3CS_2$ (Ber. 1880, **13**, 1732). (2) Converting into potassium xanthate, and estimating as copper xanthate by titration with copper acetate. (3) Converting into potassium xanthate, precipitating with copper sulphate in an acetic acid solution and igniting as copper oxide. (4) Converting into potassium xanthate, adding lead nitrate to form lead sulphide and weighing as lead sulphate. (5) Converting into lead sulphide and thiocyanate by the addition of a saturated solution of lead acetate (1 vol.), and alcohol saturated with ammonia (10 vols.). (6) Passing in the form of gas over red-hot copper and determining the sulphur in the resulting sulphide (Ber. **2**, 741). (7) Passing the gas mixed with hydrocarbons (coal gas) over finely divided and moderately heated platinum and absorbing the hydrogen sulphide in lead solution. (8) Converting into iron sulphide by heating in a sealed tube with a solution of iron chloride and strong ammonia and estimating the sulphur as barium sulphate (this Journal, 1884, 295). (9) Conversion into phenylhydrazine phenylthiocarbazinate by the addition of phenylhydrazine (Ber. **24**, 788; this Journal, 1891, 578).

For the estimation of carbon bisulphide in alcohol, the method based on the conversion into xanthate appeared to be the most suitable. To increase its accuracy when dealing with small quantities of carbon bisulphide, the sulphur in the xanthate solution was oxidised into sulphuric acid and weighed as barium sulphate. For this purpose finely powdered potassium permanganate was added to a hot alkaline solution of the xanthate until it assumed a permanent red colour. The excess was then removed by acidifying the solution with hydrochloric acid and adding oxalic acid. The presence of manganese did not interfere with the precipitation of the barium sulphate. On applying the method to the estimation of carbon bisulphide in alcohol, it was found that the formation of potassium xanthate was not quantitative, it being impossible to free the alcoholic distillate entirely from carbon bisulphide. Better results were obtained by using metallic salts in conjunction with the xanthic acid reagent. Test analyses made with lead acetate yielded 98·8 per cent. of the carbon bisulphide originally present in the sample operated upon.

The method devised by the author is based upon the following reaction, which gives quantitative results:—

$$CS_2 + 2C_6H_5NH_2 = CS(C_6H_5NH)_2 + H_2S$$
$$H_2S + Ag_2O = Ag_2S + H_2O$$
$$CS(C_6H_5NH)_2 + Ag_2O = CO(C_6H_5NH)_2 + Ag_2S$$

The process is as follows:—10 c.c. of the alcohol are heated with 50 c.c. of an alcoholic solution of silver nitrate (5 per cent.) and 5 c.c. of aniline in a covered beaker for half an hour at 50°—60°. The silver sulphide is collected on a small asbestos filter, washed with alcohol and ether, and fused in a platinum crucible with about 10 times the quantity of a mixture of sodium carbonate (1 part) and potassium nitrate (2 parts). The mass is then dissolved in water, acidified with nitric acid, filtered, and precipitated with barium nitrate. This process, when applied to carbon tetrachloride, gave equally good results. The author has examined a number of samples of carbon tetrachloride procured by him as being pure productions, in all of which carbon bisulphide was detected, as much as 4 per cent. being found in some cases.

For the purification of carbon tetrachloride prepared from carbon bisulphide, the following method is recommended. About one and a half times the quantity of potash necessary to form the xanthate is dissolved in an equal weight of water. The solution is mixed with 100 c.c. of alcohol, and added to one litre of carbon tetrachloride. The mixture is heated for half an hour to 50°—60 with frequent agitation. It is then shaken up with water to remove the xanthate, and the aqueous solution separated from the carbon tetrachloride. This operation is repeated twice with half the quantity of potash. To remove the last traces of alcohol, the product is washed with 500 c.c. of water. It is then dehydrated with solid potash and distilled over paraffin, which retains carbon trichloride, if present. The distillate was found to be quite free from carbon bisulphide. It had a boiling point of 76·3° at 760 mm. (76·5° according to Regnault and Main, and 76·7 according to Thorpe).

On treating carbon tetrachloride with phenylhydrazine to remove carbon bisulphide, and allowing the mixture to remain quiescent at the ordinary temperature, a gradual separation of crystals in the form of long, lustrous needles took place. On warming, the crystals formed more rapidly in the shape of nacreous laminæ. They proved to be a basic hydrochloride of phenylhydrazine.—D. B.

Linseed Oil, Investigation of. H. Amsel. Zeits. für angew. Chem. 1897, [12], 369—370. (See this Journal, 1895, 605 and 814.)

A RECENTLY published paper on linseed oil, by Lippert, confirms the applicability of the author's method, but to prevent misunderstanding, Amsel wishes to make it clear that his saponification number and "water-reaction" only apply to perfectly clear oils and varnishes. Fresh or insufficiently clarified preparations must either be filtered or be allowed to settle until clear. The presence of resin (colophony) is not shown by the addition of water to the alcoholic soap solution (water-reaction); this treatment only produces a cloudiness when mineral and resin oils are present in the sample. Colophony may, however, be detected by the acid value which for pure linseed oil always lies below 3, whilst for colophony the author has found it to vary between 162 and 172.

These methods are not applicable to thickened oils.

The author points out that, although the drying of linseed oil and varnishes is influenced by light and temperature, it forms, if carried out under uniform conditions and in conjunction with other tests, such as the iodine number, a good indication as to the purity of the preparation.

—J. T. C.

Balsams, Resins and Gum Resins. K. Dieterich. Chem. Rev. Fett- u. Harz-Ind. **4**. [15], 205—208; [16], 215—218.

A. Cold and Fractional Saponification.—By the successive action of ½-normal alcoholic caustic potash and of aqueous caustic potash of corresponding standard, each during 24 hours in the cold, the complete saponification values of the following substances were obtained:—Peruvian balsam, 260—270; Tacamahaca, 19·6—40·6; Sanguis draconis, 95·2—122·2; Euphorbium, 85—91; Lactucarium Anglicum, 75·6; L. Germanicum, 166·6—313·6; Ammoniacum, 148—162; Bdellium, 46·2—81·3; Galbanum, 113·4—141·4; Gamboge, 124·6—128·6. Venice turpentine, asafœtida, and olibanum remain unsaponified, and myrrh and tacamahaca are doubtful.

The advantages claimed for this cold method are, that the treatment with alcoholic potash by itself gives the resin number; the resins may be examined in their natural state; the detection of adulterants is facilitated, and no decomposition products are formed.

B. Acid Number.—In the resins devoid of esters the presence of the so-called resinolic acids complicates the

titration, by combining a further quantity of alkali on prolonged standing. The author therefore prefers to determine the acid number by back titration, and proceeds by dissolving 1 grm. of substance in 50 c.c. of petroleum spirit (sp. gr. 0·700) and acting on the solution with 50 c.c. of ½ normal alcoholic potash for 24 hours at room temperature, then titrating back, with or without the addition of 500 c.c. of water. The following results were obtained:—*Colophony:* Estimation of the acid number by back titration has the advantage that the alkali solution serves both to fix the resin acid and also as a solvent, but no addition of water must be made, or otherwise decomposition sets in, and the results give too low a figure. Acid number, 160—170. *Dammar:* without water, 20—30; with, 20—25. Direct titration gives inferior results. *Guaiacum:* the high colour of the solution renders dilution with water necessary. Acid number, 89·6 (direct, 23—24). *Mastic:* the lye suffices as solvent. Acid number (with water): Levantine varieties, 44·8—53·2; Bombay grades, 109·2—113·4. *Olibanum:* no solvent required, but the time of the operation is extended to 48 hours, the acid number then being 42·0—50·4. This is higher if colophony or sandarac has been added, and when these are suspected, no water should be used in the titration, or results will be below the truth. Dammar reduces the ester number and saponification value. *Sandarac:* results of direct titration unreliable. No solvent is needed, but water should be used. Acid number, 91—105. *Asafœtida:* the conditions resemble those for olibanum. The use of a solvent is objectionable, leading to a loss of volatile constituents. Acid number, direct (alcoholic extract), 11—82; back titration, 68—77. *Ammoniacum and Galbanum* require special treatment to obviate loss of volatile matter, 0·5 grm. being placed in a flask with a little water and distilled by direct steam, until 500 c.c. have passed over into the receiver, which contains 40 c.c. of ½ N aqueous caustic potash. The liquid is then titrated back. Acid numbers: ammoniacum 112—212·8, galbanum 73·50—114·20, those kinds exhibiting the highest number being best suited for pharmaceutical purposes.

In general, the author considers that the drugs themselves should be examined and not merely extracts; furthermore, that back titration and cold saponification afford the most reliable means of attaining the respective objects in view, and that whilst the methods borrowed from fat analysis are generally suitable for all resins, they require to be specially modified in each case. To ensure accuracy, it is desirable to ascertain the acid number and saponification by separate experiments, and the ester number by difference. All results should be referred to the undried drug in its natural condition. The iodine number of the resins is unimportant and sometimes leads to wrong conclusions. Dilution is advisable in all titrations, though care is necessary in adding water, decompositions being sometimes occasioned thereby. In saponification, the fractional cold system is alone capable of giving accurate results. The acid and ester numbers are only valuable in cases where acids or esters are respectively present, *i.e.*, ester numbers for copal, dammar, and sandarac are useless. Finally, the conditions prescribed in methods of examination should be strictly adhered to, or concordant results cannot be expected.

The methods proposed are, briefly, as follows:—

A. Balsams.—B. Copaivæ: (*a*) Acid number—Dissolve 1 grm. in 200 c.c. absolute alcohol, and titrate with $\frac{1}{10}$ N alcoholic potash, with phenolphthalein indicator, multiplying result by 5·6. (*b*) Saponification value.—Dissolve 1 grm. in alcohol, add 20 c.c. ½ N alcoholic potash, and heat for an hour under inverted condenser. Dilute with 100 c.c. of absolute alcohol, and, when cold, titrate back with ½ N H_2SO_4. (*c*) Ester number—by difference.

Bal. Peruvianum.—The acid and ester numbers are determined as above, but for the saponification value 1 grm. of substance is exposed to the action of 50 c.c. petroleum spirit (sp. gr. 0·700) and 50 c.c. ½ N alcoholic potash, for 24 hours at room temperature, in a closed flask, then shaken up with 300 c.c. of water and titrated back with ½ N H_2SO_4. By warming 1 grm. of the balsam with ether, washing on a filter until the solvent runs away clear and has ceased to extract, and then drying at 100° C., the percentage *insoluble in ether* is obtained. To determine the cinnamene present, the ethereal extract is shaken up once with 20 c.c. of 2 per cent. caustic soda, and separated in a funnel, the ethereal solution being then left to evaporate spontaneously and kept all night in the desiccator; the mean of the weighings after 12 and 24 hours gives the weight of cinnamene. The resin ester is thrown down from the alkaline liquid, from the last operation, by dilute HCl, filtered, washed under the aspirator until free from chlorine, and dried at 80° till constant. Both the percentage of the ester and its ratio towards the cinnamene should be calculated.

Bal. Tolutanum.—Acid number, ester number, and saponification value determined as for B. copaivæ, except that for the latter test, 96 per cent. alcohol is used for solution and dilution.

B. Resins.—Benzoin: In addition to the acid, ester, and saponification values, the ash is estimated by incinerating 1 grm., and calcining until the weight is constant. *Colophony:* For the acid number determination, 25 c.c. of ½ N alcoholic potash are used to 1 grm. of resin, without any other solvent. No water must be added before titrating. The specific gravity is estimated by immersion in six standard solutions of common salt, ranging in density from 1·075 to 1·080, or by the Mohr-Westphal balance.—C. S.

Molasses, Commercial Analysis of. — Molhant. Bull. Assoc. Belge des Chim. **11**, [3], 86—88.

Owing to the various changes that have taken place in the methods of manufacture the analytical processes hitherto in use are no longer suitable; the density, for example, giving no indication of the nature of the product; polarisation being unreliable, by reason of the presence of optically active substances other than saccharose, and the Clerget method inapplicable when saccharose, glucose, and raffinose are simultaneously present.

In order to afford the distiller an insight into the probable yield of alcohol obtainable from the molasses, and thus obviate further litigation on the interpretation of the terms "sound, pure, and merchantable," the author proposes to imitate the Denamur method for the commercial analysis of glucose, based on the determination of the fermentable matter in the sample. To this end, 100 grms. of molasses are dissolved in about 200 c.c. of hot water, and boiled along with 1·2—1·5 c.c. of ordinary sulphuric acid, to drive off volatile matters (HNO_2, SO_2, &c.). After filtering and washing with hot water, the filtrate is made up to 1 litre, the density (Balling) estimated, and 500 c.c. of the liquid pitched with 25 to 30 grms. of yeast, for fermentation at 28° to 30° C. during 24 hours. The alcohol and carbon dioxide being then driven off by heat, the volume of residual liquid is made up again to 500 c.c., and the difference of density (Ball.) between this and the original solution gives the amount of fermentable matter. To check the result the alcohol may be estimated by distillation.

—C. S.

Sweet Wines, Determination of Glycerin in. G. Fabris. Ann. del. Lab. Chim. Centr. delle Gabelle III., 225. 23.

The determination of glycerin in wines, presents considerable practical difficulties, especially in those kinds which contain 15—20 grms. of sugar per 100 c.c., such as Malaga, Samos, and others. Various methods have been proposed, but that of most frequent use and ready application is based upon the elimination of the sugar by means of lime, though this method is not free from defect, since the separated glycerin contains small quantities of sugar, colouring matters, as also of nitrogenous and mineral substances. Though it would appear that the proportion of glycerin found would exceed that present, yet as a matter of fact the error due to the above impurities is greatly exceeded by the errors due to other causes, especially in wine rich in sugar. Such errors arise from the bulky nature of the lime precipitate, the subsequent dilution with alcohol, the loss of glycerin in the evaporation of the alcoholic extracts, &c.

With a view of investigating the question, the author made imitation mixtures containing known proportions of glycerin, invert sugar, alcohol, and caramel; for such mix-

tures the German official method gave satisfactory results, but certain observations on the application of the method, as suggested by practice, in the analysis of wines containing 10—20 per cent. of sugar, and highly coloured, such as red Malaga, are considered by the author to be not wholly unnecessary. Thus the results of experience showed that 10 grms. of lime, and not milk of lime, should be added to each 100 c.c. of wine, and 200 c.c. instead of 100 c.c. of alcohol should be added, and the precipitate obtained should be washed with at least 200 c.c. of alcohol to extract the glycerin as completely as possible. But the great disadvantage of the German method is the time required. The author has examined that proposed by Lecco (Ber. 1892, 2094), and a somewhat similar method has been used in the Italian laboratory for some time past; in both cases satisfactory results are obtained if certain precautions are adopted, especially the cessation of the evaporation of the mixture of wine and lime when it has become of the consistency of sludge: if the evaporation is carried beyond this point there is a considerable loss of glycerin.—V. H. V.

Soap, Analysis of. R. Moreschini. Ann. del Lab. Chim. Centr. delle Gabelle III., 107—118.

In the course of commercial practice in the analysis of soaps, the following problems are presented. Firstly to determine the materials, and secondly the proportions in which they were originally mixed; the latter problem is, however, almost impossible, owing to the similarity of the composition of such materials. The author succeeded in obtaining from an Italian manufacturer the specifications of five kinds of soap used commercially under the names of (1) mottled, (2) extra green, (3) green, first quality, (4) yellow, first quality, and (5) yellow, third quality. In the samples not containing resin, determinations were made of the proportion of fatty acids, their acid and iodine values, as also of the Reichert value; for those containing resin, the method proposed by Gladding for its estimation was found to give satisfactory results. The values calculated from the known acidity and iodine numbers of the oils used in manufacture are compared with those found on analysis, and the conclusion is drawn that the methods applied give results sufficiently approximate for the purpose.—V. H. V.

Caffeine, Determination of in Coffee. E. Tassilly. Bull. Soc. Chim. **17**, [15], 761.

Reviewing the various methods which have been suggested for the determination of caffeine in coffee, the author classes them according to the solvent employed for extraction. The first group includes those in which chloroform is the solvent; the second, those extracted by means of hot water; and the third, those in which the solution of an organic salt furnishes the menstrum.

In the first group the process of *Commaille* is the oldest; as modified by *Cazeneuve* this consists in extracting a mixture of coffee and slaked lime, by digestion with chloroform. *Petit* has shown that in order to obtain the whole of the alkaloid in the case of tea, the mixture of the two powders must first be moistened to ensure the subsequent removal of all the caffeine. *Grandval* and *Lajoux* treat the coffee first with ammoniacal ether, and then extract with chloroform by digestion.

In the second group, the process of *Domergue* and *Nicolas* consists in precipitating an aqueous infusion of coffee with mercuric acetate. The filtrate, mixed with sand and magnesia, is evaporated to dryness. The dry residue is then exhausted with chloroform. *Dvorkovitch* makes three infusions, followed by two decoctions, giving a litre of aqueous extract in all from each 10 grms. of coffee taken. This is precipitated with baryta and filtered, the filtrate is washed with petroleum ether; then extracted with chloroform. *Guillot* merely exhausts the substance with successive boilings and washes out the alkaloid with chloroform.

In the third group, *Herlant's* method consists in the use of a solution of sodium benzoate as a solvent, the coffee having first been mixed with lime. The caffeine is removed from the extract by shaking out with chloroform. *Gorges* prefers simple lixiviation with salicylate of sodium.

After adversely criticising each of these processes *seriatim*, the author proposes the method given below.—J. O. B.

Caffeine in Coffee, New Method of Estimating. E. Tassilly. Bull. Soc. Chim. 1897, **17**, [15], 766.

Ten grms. of powdered coffee are treated with three successive volumes, each of 200 c.c. of boiling water, infusion being prolonged for 10 minutes in each case. After decantation, another 200 c.c. of water are added to the residue and the mixture is heated until the liquid begins to be coloured; this is then decanted and the process repeated with another similar quantity of water, so that the volume of the total aqueous extract is 1 litre. This liquid is divided into equal volumes, each of 500 c.c. These are evaporated to dryness on the water-bath, then treated with 1 or 2 c.c. of 10 per cent. sulphuric acid, thoroughly stirred, and then extracted with small quantities of boiling water on filters, until the filtrates cease to give a precipitate with a solution of tannin, or a red coloration with Gaucher's test.

From these slightly acid solutions the caffeine may be extracted in two ways. In operating, as indicated on 10 grms. of coffee, it is as well to treat one 500 c.c. by the first process given below, and the other by the second. Of the two, the latter is to be preferred as the alkaloid is obtained from it in a perfectly pure condition.

(1.) The solution is evaporated to dryness on the water-bath, adding, at the last, 20 grms. of washed sand, and 2 grms. of calcined magnesia. The dry mixture is transferred to an extractor and treated with 100 grms. of chloroform until exhausted, which will be in three or four hours. The solvent is then distilled off, and the residue weighed after drying at 100° C.

(2.) The other 500 c.c. is made faintly alkaline with ammonia, and extracted with several successive portions of chloroform until nothing more is removed by that solvent. The chloroform is then distilled to a small bulk, the residue decanted into a tared dish, evaporated and weighed. The latter method invariably gives slightly higher figures than that in which the solvent is employed hot.

The following are comparative results obtained by the author with various processes, on the same samples of coffee, previously dried at 110° C.:—

	Coffee.		
	New Caledonia.	Liberia.	Mocha.
New process (cold chloroform)	1·44	1·40	1·18
,, ,, (hot ,,)	1·36	1·39	1·08
Domergue and Nicholas' process	1·34	1·39	1·16
Gorges' ,,	1·40	1·36	1·16
Herlant's ,,	1·40	1·35	1·16
Delecour's ,,	1·40	1·35	1·12
Dvorkovitch's ,,	1·40	1·35	1·16

The author has assured himself that every trace of caffeine is removed by his method, and also that no loss of alkaloid occurs; a known amount of caffeine added to one of the two portions of the liquid, after infusion being found again within the limit of experimental error.

—J. O. B.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Iron Chloride, Hydrolysis of. W. Spring. Chem. Zeit. 1897, **21**, [66], 651.

It is stated that ferric chloride in aqueous solution does not change directly into colloidal ferric hydrate and hydrochloric acid, but that an intermediate decomposition first takes place, similar to that of the vapour at a high temperature and resulting in ferrous chloride and chlorine. The chlorine set free, reacts with the water with formation of electrically neutral hydrochloric acid and active oxygen, and the latter combines with the ferrous chloride, forming, so long as the temperature undergoes no change, equal proportions of ferric hydrate, hydrochloric acid, ferric chloride and water.

—A. S.

Acetylene. The Formation of Mixed Hydrates of. De Forcrand and S. Thomas. Comptes Rend. 1897, **125**, 109—111.

By the interaction of acetylene, water and carbon tetrachloride at 0° and under a pressure of about 20 atmospheres, a crystalline product is obtained, which is stable at 5° and at a pressure scarcely above that of the atmosphere; at a higher temperature it decomposes with evolution of acetylene. It is assumed to be a mixed hydrate of acetylene and carbon tetrachloride. Similar mixed hydrates have also been obtained by employing other halogen derivatives in the place of the carbon tetrachloride, and crystals have also been obtained with other gases, namely, C_2H_4, CO_2, SO_2, in the place of acetylene.—A. K. M.

Ethane, Synthesis of; Action of Nickel upon Ethylene. P. Sabatier and J. B. Senderens. Comptes Rend. **124**, 1358.

When a slow current of pure ethylene is passed over the surface of nickel reduced by hydrogen, and heated to above 320° C., the gases obtained after removing traces of undecomposed ethylene by means of bromine are found to consist of a mixture of ethane, formene, and hydrogen, with possibly an infinitesimal trace of higher hydrocarbons. —J. O. B.

"Active" Oxygen (the so-called) and Peroxide Formation. C. Engler and W. Wild. Ber. 1897, **30**, [12], 1669—1681.

In the oxidations produced by free oxygen, it has been frequently observed that a second substance present, which is entirely unattacked by ordinary oxygen, is energetically oxidised. Many explanations of these phenomena have been given: change of oxygen into ozone and antozone (Schönbein), existence of oppositely charged atoms in the oxygen molecule (Brodie and Clausius), formation of free oxygen atoms, &c. M. Traube supposed that in the oxidation of zinc there occurs the process represented by the following equation:—

$$Zn + \begin{array}{c}OH.H\\ \\ OH.H\end{array} + \begin{array}{c}O\\ \vdots\\ O\end{array} = Zn(OH)_2 + \begin{array}{c}H.O\\ \cdot\\ H.O\end{array}$$

and showed that the amount of hydrogen peroxide produced agreed with this hypothesis, which, however, only explains such oxidations as proceed in the presence of water.

Van't Hoff and Jorissen have shown that the spontaneously oxidisable substance renders capable of oxidation exactly as much oxygen as it itself absorbs; they assume that the oxygen molecule splits up into atoms of different electrical charges, one of which is by preference absorbed by each substance present; and Ewan, on this hypothesis, has found that ordinary oxygen must be already slightly dissociated in this way.

The authors put forward the hypothesis that "in spontaneous oxidations not single oxygen atoms but whole molecules are taken up, the double union of the atoms in the molecule being disturbed, with formation of peroxides of the type—

$$\begin{array}{c}R.O\\ \cdot\\ R.O\end{array} \text{ or } R\langle\begin{array}{c}O\\ \cdot\\ O\end{array}$$

These compounds give up (like hydrogen peroxide) one oxygen atom to another oxidisable substance, thus passing into ordinary oxides.—A. C. W.

Aldehydes, Synthesis of Aromatic. L. Gattermann and J. A. Koch. Ber. **30**, 1622—1624.

The method consists in acting on toluene and its homologues with hydrochloric acid and carbonic oxide in presence of aluminium chloride and cuprous chloride. A current of carbonic oxide and hydrochloric acid is passed through a mixture of 30 grms. of toluene, 45 grms. of finely powdered freshly prepared aluminium chloride, and 4 grms. of cuprous chloride, at a temperature of 20°—25° C. with good agitation. The carbonic oxide passes through at the rate of 3 litres per hour, whilst the hydrochloric acid is passed through at half that rate. The dark viscid product of the reaction is poured on to ice, the oil, separated, and the aldehyde and unaltered toluene blown over with steam. The distillate is treated with sodium bisulphite, separated from unaltered toluene (about 8 grms.), the aqueous solution decomposed with anhydrous soda, and the aldehyde again blown over with steam. After taking up with ether and evaporating off, about 20—22 grms. of perfectly pure *p*-tolyl aldehyde remains; this, on oxidation, gives *p*-toluic acid. The method gives satisfactory results also with o-, *m*-, and *p*-xylene, ethylbenzene, mesitylene, and other substances.—T. A. L.

Caffetannic Acid, The so-called (Glycosylcaffeïc Acid), and its Successive Decomposition to Caffeïc Acid, Vinylpyrocatechol and Pyrocatechol. H. Kunz-Krause. Ber. 1897, **30**, [12], 1617—1622.

The tannic acids of coffee and maté, on decomposition, yield sugars and dihydroxycinnamic acid (caffeic acid). Hoffmann states that this acid melts at about 213°, with browning and incipient decomposition; at 200°, however, it is quantitatively decomposed into carbon dioxide and vinylpyrocatechol, $C_6H_3(OH)_2.C_2H_3[C_2H_3 : OH : OH = 1:3:4]$. The latter cannot be distilled *in vacuo* without a decomposition which produces pyrocatechol. The often observed formation of protocatechuic and acetic acids in the fusion of so-called tannic acids with potash, is explained by the following reaction of caffeic acid:—

$$C_6H_3(OH)_2.C_2H_2.COOH + (H_2 + O_2) = C_6H_3(OH)_2.COOH + CH_3.COOH.$$

—A. C. W.

Caffetannic Acid. Cazeneuve and Haddon. Comptes Rend. 1897, **124**, [25], 1458—1460.

Caffetannic acid has usually ascribed to it the formula $C_{15}H_{18}O_8$, and is regarded as a glucoside formed by the addition of caffeïc acid and mannitan, with elimination of H_2O. Preparing a solution of caffetannic acid according to the directions of Hlasiwetz, and treating this with phenylhydrazine and acetic acid, the author obtained yellow well-crystallised needles of an osazone, the analysis of which yielded results (C, 62·8; H, 5·9; N, 13·04 to 13·28) closely agreeing with those (C, 62·7; H, 5·5; N, 13·02) corresponding to the formula—

$$C_6H_3(OC_6H_9O_3.N_2H(C_6H_5)_2.CH(CH)COOH,$$

the osazone of a substance—

$$C_6H_3(OC_6H_{11}O_5)_2.CH.CH.COOH \text{ or } C_{21}H_{28}O_{14},$$

which should therefore be the formula of caffetannic acid. According to this, caffetannic acid must be regarded as formed from a molecule of caffeïc acid and two molecules of a sugar, $C_6H_{12}O_6$, with elimination of $2H_2O$. The authors have this sugar under investigation.—J. T. D.

Oxidation and Chlorination, A Method of [by the Aid of Salts of Manganese]. A. Villiers. Comptes Rend. **124**, 1349.

When an oxidisable body is placed in a medium capable of supplying oxygen, but under such conditions that oxidation does not take place, or proceeds but slowly, the addition of a trace of a salt of manganese brings about, or very materially accelerates the reaction. Thus if a mixture be made of equal volumes of saturated solution of oxalic acid, of hydrochloric acid, 25 per cent., and of nitric acid of the same strength, no gas is evolved even after prolonged contact. If, however, after raising the temperature of the mixture to boiling point, a trace of any salt of manganese be added, such as a few drops of solution of the sulphate, reaction takes place almost immediately, and the evolution of carbonic acid gas continues regularly, particularly if a few fragments of gas carbon be introduced into the liquid. If heating be continued, the whole of the oxalic acid is rapidly used up, and the process can be continued indefinitely by introducing more of that substance, and more nitric acid, to furnish the requisite oxygen. The

manganese salts appear to act as carriers of oxygen, probably by the formation of intermediate unstable oxysalts; their behaviour may almost be compared with that of the oxidising ferments. It is possible that manganese, which is so widely distributed in the animal or vegetable organism, thus plays an important and hitherto unsuspected part in physiology, since its presence may be necessary for the oxidation of carbohydrates and nitrogenous bodies. In therapeutics too, the salts of manganese should prove useful in the treatment of those diseases, such as diabetes and arthritis, which arise from defective physiological oxidation.

Sucrose, glucose, and members of the fatty series are oxidised under like conditions in the same manner as oxalic acid; with substances of the aromatic series, however, even when they contain fatty bodies, oxidation does not result, but chloro-substitution bodies are formed. Thus when benzene or toluene are heated with aqueous solutions of nitric and hydrochloric acid, with a trace of a manganese salt added, reaction takes place slowly even in the cold; the lower surface of the liquid, which is in contact with the aqueous solution, is, in the course of a few days, so far altered that chloro-substitution products are formed, which being of a greater density, fall to the bottom. This method of chlorination promises to be useful for technical purposes. —J. O. B.

Oxidations [Drying Oils], The Part played by Manganese in. A. Livache. Comptes Rend. 1897, **124**, [25], 1520—1521.

Referring to the paper of Villiers (see preceding abstract), the author points out that in 1883 he remarked on the effect of manganese in hastening the oxidation of drying oils, the same oil being completely oxidised in six hours when it contained a little manganese, while with lead it needed 24, and with iron 48. In all such actions the manganese probably acts simply as a carrier of oxygen: no doubt the action of all oxydases (see next abstract) is a similar one.—J. T. D.

Oxidising Action of Salts of Manganese, and the Chemical Constitution of the Oxydases. G. Bertrand. Comptes Rend. **124**, 1355.

It is found that the salts of manganese, *per se*, act as carriers of oxygen. When introduced in aqueous solution together with a body capable of oxidation, such as hydroquinone, into a flask fitted with a stop-cock, and kept continually agitated for a prolonged period, by means of a water wheel, considerable diminution of the amount of oxygen in the atmosphere of the vessel is observed. This varies with the salt of manganese employed, as will be seen from the following results. The same quantity of hydroquinone and water were used in each case:—With nitrate, 1·5; sulphate, 1·6; chloride, 1·8; benzoate, 7·4; acetate, 15·3; salicylate, 15·7; lactate, 16·3; glucinate, 17·6; and succinate, 22·1 c.c.

It would appear that the salt of manganese in aqueous solution undergoes partial hydrolysis, with the formation of free acid and manganous oxide, thus:—

$$R''Mn + H_2O = R''H_2 + MnO.$$

The manganous oxide thus formed, as is well known, is oxidised in the air, and the molecule of atmospheric oxygen thus split up leaves one atom free to combine with any oxidisable body: $MnO + O_2 = MnO_2 + O$.

It is found that a solution of hydroquinone containing manganous oxide in suspension gives at once, on exposure to the air, quinone and manganese dioxide. The dioxide so formed is again reduced in an acid solution, forming a manganous salt and a further quantity of quinone. Thus, a small amount of a salt of manganese may serve as a carrier of oxygen to an unlimited quantity of hydroquinone. The theory of hydrolysis of the salt is supported by the fact that the salts of the organic acids, having a high molecular weight, and a relatively low affinity for the metal, and which are therefore most easily hydrolysed, are those which act most energetically as oxidisers. It would seem, therefore, that in the case of oxydases—which are regarded as manganese compounds in which the albuminoids play the part of very feeble acids, having only just sufficient affinity for the metal to keep it in solution—the oxidising power is due to the manganese, the albuminoids giving to these ferments only their peculiar physical and chemical properties.—J. O. B.

An Organic Substance rich in Manganese extracted from Woody Fibre. G. Guérin. Comptes Rend. 1897, 311—312.

Sawdust is digested for 2—3 days with distilled water containing 1 per cent. of pure caustic potash. The brown solution obtained after filtering, when treated with a slight excess of hydrochloric acid, gives a voluminous precipitate, which can be easily washed by decantation with water containing a trace of hydrochloric acid to prevent dissociation. The product is then dissolved with a few drops of ammonia, reprecipitated with hydrochloric acid, washed as above, and dried. It forms a brown powder, and the particular sample examined from beech sawdust, did not contain any iron, but a considerable proportion of manganese, phosphorus, and sulphur, and gave the following percentage numbers on analysis:—C = 52·762, H = 5·04, N = 4·60, S = 0·666, P = 1·297, Mn = 0·402. The author considers it probable that manganese, combined in a nucleus, exists in all woody fibres.—T. A. L.

PATENT.

Plants, Leaves, and the like; Impts. in or relating to the Treatment and Preservation of. A. J. Boult, London. From R. L. and K. R. Keferstein, Dresden. Eng. Pat. 14,825, June 18, 1897.

In the treatment of the dry parts of plants, leaves, &c., so as to give them a life-like appearance, the ordinary process may be much shortened by a preliminary treatment with more or less diluted sulphuric acid, which thoroughly softens or dissolves the outer coating; the subsequent treatment with glycerin and dyestuff can then be effected simultaneously in the same bath, and the dyestuff being more perfectly absorbed a better result is obtained. The process then consists of (*a*) treatment with acid, (*b*) washing, (*c*) partly drying, (*d*) immersion in a bath of glycerin and dyestuff containing a salt of nitric acid, acetic acid, and gelatin, (*e*) drying sufficiently for (*f*) varnishing. If paint is to be applied as well as or instead of the dyestuff, it may be mixed with the varnish or applied just before.—L. A.

EDUCATIONAL.

Continental Technical Schools.—Report of the Manchester Deputation.

Chem. Trade J., Oct. 2, 1897, 210.

In pursuance of a resolution of the Technical Instruction Committee, confirmed by the Manchester City Council, a deputation, comprising Mr. Alderman Hoy, Mr. Alderman Crosfield, Mr. N. Bradley, Mr. Ivan Levinstein, Mr. John Craven, Mr. Charles Rowley, and Mr. J. H. Reynolds recently visited a number of institutions and schools on the Continent, devoted mainly to scientific and artistic instruction as applied to industrial and commercial pursuits. In their report to the Committee the deputation mention the fact that in 1891 a visit was paid to several of the more important technical schools in France, Germany, Austria, and Switzerland, and that a report was then issued showing the extent to which provision had been made for the supply of instruction of a scientific and technical character in aid of the commerce and of the industries of those countries. "In the meantime," the report proceeds, "the council has undertaken the responsibility of maintaining the technical and art schools of the city, and, with the purpose of giving full effect to that responsibility, has already not only greatly developed these institutions, but has embarked upon the erection of the largest technical school in the country, the proper equipment of which is a matter of the most serious concern and importance. The erection of the new school was begun in August 1895, and its completion, ready for the occupation, is confidently expected at an early date. The Committee therefore feel that it is high time the question of the equipment of the school was considered, especially in respect

of the important departments concerned with the textile industries, with the industrial application of chemistry, and of physics in relation to electrical engineering. . . . Your Committee felt that it would be the height of unwisdom, having in view the serious responsibility of equipping the new Municipal Technical School in Manchester so as to meet adequately and effectively the demands of a great industrial centre such as this, were they to neglect the opportunity of seeing what Continental countries have done of late, and what they are now doing in this regard. Especially is this a necessity with respect to the extraordinary development which has taken place within quite recent years in electrical science as applied to electrical engineering industries, and with the certainty of great extension in the near future. Hardly less important is the great textile industry in its various departments of spinning, weaving, designing, dyeing, and finishing, in some of which we find ourselves at a serious disadvantage (especially those in which chemistry plays a part) as compared with our foreign competitors. It has been found necessary in the dyeing and finishing schools abroad to discard mere laboratory methods, and to equip them on a scale approaching that of the works themselves, and analogous to the practice obtaining in the spinning and weaving schools, so as to give the students who are trained in them a real, practical, and effective knowledge of the processes employed." Reference is then made to what the deputation saw at Crefeld and in its well-equipped textile and dyeing schools. "Your deputation is convinced," says the report, "as a result of the inspection of the Crefeld School, that this district would gain materially by the development of our textile school in the new building on the same lines. 1. By the increase in the number and variety of the looms and of the goods woven upon them. 2. By the establishment of a school of tinctorial chemistry, and of practical dyeing and finishing upon an adequate scale, alike in respect of the completeness and the real efficiency of the machinery employed. 3. By the establishment, in actual touch with the other departments of the school, of a well-organised museum, replete with examples of ancient, mediæval, and modern productions of the best type of workmanship, colour, and design. It is to variety and excellence in these respects that Lancashire must look to maintain and increase its supremacy and reputation as a manufacturing centre." From Crefeld the deputation went to Aix-la-Chapelle, thence to Berlin, Darmstadt, Dresden, Vienna, and other places. After dwelling on the zeal shown by Germany in technical training, the deputation remark: "That Germany is in a prosperous condition, due to her successful manufacturing and commercial enterprise, was plainly evident on every hand in the extension of her cities—the making of new streets and the erection of fine, handsome buildings which is going on everywhere in her large towns. It is not less clear that the schools are the root and base of this surprising industrial development, and are the main contributors to this great economic result; it is no less certain that if we are to maintain our position as a great industrial community it must be by following and adopting the same methods. It is not, however, only in the domain of science that Germany is making great progress. In almost every town visited by the deputation fine industrial art museums were found, arranged with the express purpose of cultivating a knowledge of what has already been accomplished in the production of fine examples of colour, design, and workmanship. Every technical school has its special museum of objects applicable to its purposes. Notably was this the case in Berlin, Vienna, Nuremberg, Crefeld, and at Düsseldorf, in which latter place the Industrial Art Museum is said to be the finest in the Rhineland. These museums help to preserve and hand down the traditions of past achievement and excellence, and stimulate the desire to reach to as high or higher levels to-day." In concluding their report the deputation express their sense of the advantage, whatever the ultimate drawbacks, of a centralised bureaucratic administration, which, taking a careful survey of the educational and industrial needs, placed the schools here or there as circumstances required, brought them into mutual relation, supplied ample means, and effectively assisted, without loss of time, the industrial advance. Something might be lost of "freedom, variety, and elasticity," and that loss might ultimately be serious in its effect upon individual initiative, upon which we as a nation so confidently relied. Which was the better policy the future could alone determine. It might, however, be safely asserted that it was high time the effort was made in this country to give to our youth the educational advantages, general and special, which were enjoyed by their rivals abroad. Exception had sometimes been taken to the size and cost of the new buildings now being erected by the Technical Instruction Committee for the Municipal Technical School, but the deputation, had returned from their visit doubly confirmed in their conviction that every foot of space would be needed, and that even when fully utilised and equipped it would fail to rival in amplitude of resource the splendid industrial schools of Germany and Switzerland.

Commercial Reading Room at the Imperial Institute.

A commercial reading room has been established at the Imperial Institute, and is open for the free use of the general public. It is supplied with the principal British commercial and technical publications, and a number of similar prominent Indian, colonial, and foreign periodicals. Visitors to the reading room can also consult a large collection of directories, almanacks, official year-books and hand-books, Government gazettes, diplomatic and foreign consular reports, market reports, prices current and statistics, trade circulars and catalogues, and an extensive collection of colonial and Indian maps. Inquiries relating to commercial and industrial matters, to emigration, &c., addressed personally, or by letter, to the Commercial Intelligence Department of the Institute, are promptly dealt with.

New Books.

The Principles of Chemistry. By D. Mendeléeff. Translated from the Russian (Sixth Edition) by George Kamensky, A.R.S.M., of the Royal Imperial Mint, St. Petersburg, &c. Edited by T. A. Lawson, B.Sc., Ph.D. In two volumes. Longmans, Green, and Co., 39, Paternoster Row, London; also New York and Bombay. 1897. Price 36s.

Two 8vo volumes, containing preface to the English Translation and author's preface to the Sixth Russian Edition, tables of contents to Vols. I. and II., and in Vol I. 621 pages of subject-matter and 78 illustrations; whilst in Vol. II., following the table of contents, there are 499 pages of subject-matter and 19 illustrations. Just preceding the table of contents in Vol. I. are two tables: Table I. The Distribution of the Elements in Groups and Series; and Table II. The Periodic System and Atomic Weights of the Elements. Forming a kind of frontispiece to Vol. II., is a Table showing the periodic dependence of the composition of the simplest compounds, and properties of the simple bodies upon the Atomic Weights of the Elements. At the end of Vol. II. are alphabetical indexes of authors and of subject-matter for the entire work. The contents of Vol. I. are given and subdivided as follows:—I. Water and its Compounds. II. Composition of Water. Hydrogen. III. Oxygen and the chief aspects of its Saline Combinations. IV. Ozone and Hydrogen Peroxide. Dalton's law. V. Nitrogen and Air. VI. The Compounds of Nitrogen with Hydrogen and Oxygen. VII. Molecules and Atoms. The Laws of Gay-Lussac and Avogadro-Gerhardt. VIII. Carbon and the Hydrocarbons. IX. Compounds of Carbon with Oxygen and Nitrogen. X. Sodium Chloride. Berthollet's Laws. Hydrochloric Acid. XI. The Halogens; Chlorine, Bromine, Iodine, and Fluorine. XII. Sodium. XIII. Potassium, Rubidium, Cæsium, and Lithium. Spectrum Analysis. XIV. The Valency and Specific Heat of the Metals. Magnesium, Calcium, Strontium, Barium, and Beryllium. The Contents of Vol. II. are sketched as follows:—XV. The Grouping of the Elements and the Periodic Law. XVI. Zinc, Cadmium, and Mercury. XVII. Boron, Aluminium, and the Analogous Metals of the Third Group. XVIII. Silicon and the other Elements of the Fourth Group. XIX. Phosphorus

and the other Elements of the Fifth Group. XX. Sulphur, Selenium, and Tellurium. XXI. Chromium, Molybdenum, Tungsten, Uranium, and Manganese. XXII. Iron, Cobalt, and Nickel. XXIII. The Platinum Metals. XXIV. Copper, Silver, and Gold. Appendix:—I. An attempt to apply to Chemistry one of the Principles of Newton's Natural Philosophy. II. The Periodic Law of the Chemical Elements (The Faraday Lecture). III. Argon: A New Constituent of the Atmosphere.

Jahrbuch der Chemie. Bericht über die wichtigsten Fortschritte der reinen und angewandten Chemie. Unter Mitwirkung von H. Beckurts, C. A. Bischoff, E. F. Dürre, J. M. Eder, P. Friedländer, C. Häussermann, F. W. Küster, J. Lewkowitsch, M. Märcker, F. Röhmann, K. Seubert. Herausgegeben von Richard Meyer (Braunschweig). VI. Jahrgang. 1896. Friedrich Vieweg und Sohn, Brunswick. 1897. Price M. 15. H. Grevel and Co., 33, King Street, Covent Garden, London, W.C.

8vo volume containing 525 pages of subject-matter, and alphabetical indexes of subject-matter and authors. The work commences with a short preface by the editor, Richard Meyer, and with regard to the subjects treated of, these are given as follows, and are contributed by the authors following the subjects numbered. I. Physical Chemistry (Küster). II. Inorganic Chemistry (Seubert). III. Organic Chemistry (Bischoff). IV. Physiological Chemistry (Röhmann). V. Pharmaceutical Chemistry (Beckurts). VI. Chemistry of Foodstuffs, &c. (Beckurts). VII. Agricultural Chemistry (Märcker and Naumann). VIII. Metallurgy (Dürre). IX Fuels and Explosives (C. Häussermann). X. Technology of the Carbohydrates and of Fermentation (Märcker and Naumann). XI. Technology of the Fats and Petroleums (Lewkowitsch). XII. Chemistry of Tar Products and Colours (Richard Meyer). XIII. Chemical Technology of Textile Fibres (Friedländer). Photography (Eder and Valenta).

Chemisch-Technisches Repertorium. Uebersichtlicher Bericht über die neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der technischen und industriellen Chemie, mit Hinweis auf Maschinen, Apparate und Literatur. Herausgegeben von Dr. Emil Jacobsen. 36. Jahrgang. 1897. Erstes Halbjahr. Zweite Hälfte. Mit in den Text gedruckten Illustrationen. Berlin: R. Gaertner's Verlagsbuchhandlung, Hermann Heyfelder, S.W., Schönebergerstrasse 26. London: H. Grevel and Co., 33, King Street, Covent Garden.

This number, for the second half-year of 1897, treats of the following subjects of Applied Chemistry:—Foodstuffs. Paper. Photography. Residual Matters. Manures. Disinfection and Sanitation. Soaps. Matches and Explosives. Preparation and Purification of Chemicals Chemical Analysis. Apparatus. Machinery. Electro-technology. Thermo-technology. Adulteration of Trade Products, &c. New Books.

Organic Chemistry for the Laboratory. By W. A. Noyes, Ph.D., Professor of Chemistry in Rose Polytechnic Institute, Terre Haute. 1nd. Chemical Publishing Co., Easton, Pa. 1897. Price 1.50 dols.

This small work is very similar to that of Fischer, Cohen, Gattermann, and others, to whom, indeed, the author acknowledges his indebtedness.

The subject-matter fills 240 pages, and there is an alphabetical index. The work may be defined as one of Organic Chemical Preparations, Reactions, and Separations. It is subdivided as follows:—Chapter I. Acids. II. Derivatives of Acids. III. Halogen Compounds. IV. Nitro Compounds. V. Amines. VI. Hydrazo, Azo, Diazo Compounds, &c. VII. Alcohols and Phenols. VIII. Aldehydes, Ketones, and their Derivatives. IX. Sulphonic Acids and Sulphine Compounds. X. Hydrocarbons. XI. Miscellaneous Compounds. XII. Qualitative Examination of Organic Compounds.

Trade Report.

OFFICIAL NOTICES.

Acetylene Gas.

The Explosives Department of the Home Office has recently had under consideration the question of the restrictions to be applied to the manufacture and keeping of acetylene gas, and has conducted various experiments with the object of gaining information on this matter. The results show conclusively that acetylene gas *per se*, when under a pressure of something less than two atmospheres, is violently explosive; whereas, at a pressure of less than one and a half atmospheres, it appears to be reasonably free from liability to explosion, provided it is not admixed with oxygen or atmospheric air.

For commercial and practical purposes, it is considered sufficient to allow a pressure of 20 ins. of water above that of the atmosphere (*i.e.*, roughly about $1\frac{1}{20}$ atmospheres), and it is accordingly proposed to draw the safety line at this point, and to declare acetylene when subject to a higher pressure to be an "explosive" within the meaning of the Explosives Act, 1875.

In France and Germany, the authorities have fixed the limit of danger at $1\frac{1}{2}$ and $1\frac{1}{10}$ atmospheres respectively, and have imposed prohibitions or restrictions on the keeping or manufacture of the gas when it is at a higher pressure.

Whitehall.
5th October 1897.

Explosives in Coal Mines Order, 1897.

Memorandum.

1. Arrangements have now been completed for testing explosives (at the Home Office Testing Station, Royal Arsenal, Woolwich), with a view to their being admitted to the list of permitted explosives, which it is proposed to substitute for the list contained in the schedule to the above order. The explosives on the permitted list at present in force will have to be submitted to the test, and pay the fee accordingly.

2. Any person wishing to have an explosive tested must fill in the accompanying application form, and forward it, together with a fee of 25*l.*, to the Under Secretary of State, Home Office, Whitehall, S.W.

3. Instructions will then be given as to the samples to be sent for testing.

4. The samples will be tested in the order, so far as practicable, in which they are received; and notice of the day and hour of the test will be sent to the applicant, who will, on prior application, receive a permit authorising him (by or with a representative named by him) to be present at the test. The test is described in an appendix.

5. All explosives which pass the test will be put on the permitted list, but in the event of an explosive failing to pass the test, it will be open to the applicant to apply for a further trial, for which a further fee must be paid of 15*l.*

6. In this further trial the Secretary of State reserves a right to make any variation in the conditions of the test which may appear to him to be necessary to meet any special circumstances.

7. The permitted list is liable to revision from time to time, and explosives on the permitted list will be liable to occasional re-testing.

8. Applications from manufacturers, importers, mine owners, miners, and others interested, will be entertained for the re-testing of explosives on payment of a fee of 2*l.* for a minimum of 10 shots. Additional shots to be paid for *pro rata*.

9. If at any time an explosive is found in practical use to have caused explosions, or on examination or re-testing to be of bad manufacture, or otherwise likely to be dangerous, the Secretary of State reserves to himself the right, after such special inquiry as he may direct, of removing the explosive from the permitted list or of prohibiting its use when made by a particular manufacturer.

10. Facilities will be given for samples to be tested for experimental purposes in connection with the working out of new explosives for use in coal mines, on payment of fees to be determined in each case; but no explosive will be received for firing, even experimentally, which has not previously been placed on the list of authorised explosives issued by the Home Office.

V. D. Majendie, Colonel,
H.M. Chief Inspector of Explosives.

Home Office,
Whitehall, S.W.,
8th October 1897.

TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

New Customs Tariff of Canada.

(Continued from page 706.)

No.	Article.	Rate of Duty.
333	Wood pulp	25 % *ad val.*
341	Vulcanised fibre and like material	25 % *ad val.*
352	Gold, silver, and aluminium leaf, Dutch metal leaf, bronze powder, and gold liquid paint.	25 % *ad val.*
	Minerals.	
353	Asbestos in any form other than crude, and all manufactures thereof.	25 % *ad val.*
354	Plumbago, not ground or otherwise manufactured.	10 % *ad val.*
355	Plumbago, ground, and manufactures of, n.e.s., and foundry facings of all kinds.	25 % *ad val.*
		Dols. Cts.
413	Gunpowder	Per lb. 0 3
414	Nitro-glycerin, giant powder, nitro and other explosives.	„ 0 3
415	Glycerin, when imported by manufacturers of explosives, for use in the manufacture the reof in their own factories.	10 % *ad val.*
416	Torpedoes, fire-crackers, and fireworks of all kinds.	25 % *ad val.*
417	Fertilisers, compounded or manufactured.	10 % *ad val.*
418	Lamp wicks	25 % *ad val.*
419	Photographic dry plates	30 % *ad val.*
	Sugar, Syrups, and Molasses.	
435	All sugar above number sixteen, Dutch standard, in colour, and all refined sugars, of whatever kinds, grades, or standards.	Per lb. 0 1
436	Sugar, n.e.s., not above number sixteen, Dutch standard, in colour, sugar drainings, or pumpings drained in transit, malado or concentrated malado, tank bottoms and sugar concrete (the usual packages in which imported to be free).	„ 0 0½
437	Glucose or grape sugar, glucose syrup and corn syrup, or any syrups containing any admixture thereof.	„ 0 0¾
438	Sugar-candy, brown or white, and confectionery, including sweetened gums, candied peel, and pop-corn	„ 0 0½ and 35 % *ad val.*
439	Maple sugar and maple syrup	20 % *ad val.*
440	Syrups and molasses of all kinds, n.o.p., the product of the sugar cane or beet, n.e.s., and all imitations thereof or substitutes therefor.	Per lb. 0 0¾
441	Molasses produced in the process of the manufacture of cane sugar from the juice of the cane without any admixture with any other ingredient, when imported in the original package in which it was placed at the point of production and not afterwards subjected to any process of treating or mixing, the package in which imported, when of wood, to be free.	
	(*a*) Testing by polariscope 40 degrees or over.	Per gall. 0 1½
	(*b*) When testing by polariscope less than 40 degrees and not less than 35 degrees.	„ 0 1¾
	And in addition thereto, 1 cent per gallon for each degree or fraction of a degree less than 40 degrees.	

Schedule B.—Free Goods.

No.	Article.
462	Philosophical instruments and apparatus imported for use in universities, schools, and scientific societies.
464	Books on the application of science to industries of all kinds.
466	Books printed by any association for the promotion of science.
475	Models of inventions (but not those which can be fitted for use).
476	Aluminium in ingots, blocks, or bars, strips, sheets, or plates, alumina and chloride of aluminium, or chloralum, sulphate of alumina and alum cake, and alum in bulk only, ground or unground.
477	Ambergris; ammonia, sulphate of salammoniac, and nitrate of ammonia; arsenic; bromine, burgundy pitch; cinnebar, cochineal, cyanide of potassium, and cyanogen or compound of bromine and potassium for reducing metals in mining operations; iodine, crude; kryolite or cryolite, mineral; oxalic acid; quinine, salts of; saltpetre; calcareous tufa; alizarine and artificial alizarine; aniline oil, crude; aniline salts and arseniate of aniline; annato, liquid or solid; aniline dyes and coal tar dyes in bulk or packages of not less than one pound weight.
478	Antimony salts; antimony, or regulus of, not ground, pulverised, or otherwise manufactured.
480	Asphalt, or asphaltum; bone pitch, crude only; and resin or rosin in packages of not less than one hundred pounds; and resin oil.
484	Bismuth, metallic, in its natural states; blood albumen and tannic acid.
485	Blast furnace slag.
488	Bones, crude, not manufactured, burned, calcined, ground, or steamed.
490	Boracic acid and borax, ground or unground, in bulk of not less than 25 lb.
501	Celluloid, xylonite, or xyolite, in sheets and in lumps, blocks, or balls in the rough.
502	Chloride of lime, in packages of not less than 25 lb. weight; cobalt, ore of; oxide of cobalt, oxide of tin, and oxide of copper; copper, precipitate of, crude; dragon's blood; gypsum, crude (sulphate of lime); lava, unmanufactured; manganese, oxide of; phosphorus; litharge; saffron, saffron cake, safflower, and extract of; sulphate of iron (copperas); sulphate of copper (blue vitriol); sulphur and brimstone, crude or in roll or flower; tartar emetic and grey tartar; cream of tartar in crystals and argol or argols; verdigris, or sub-acetate of copper, dry; zinc, salts of, and tartaric acid crystals.
505	Clays, including China clay, fire-clay, and pipe-clay; gannister and sand.
506	Coal, anthracite and anthracite coal dust; coke.
507	Coal and pine pitch, and coal and pine tar of not less than 15 gallons.
510	Crucibles, clay or plumbago.
515	Drugs, crude such as barks, flowers, roots, beans, berries, balsams, bulbs, fruits, insects, grains, gums and gum resins, herbs, leaves, nuts, fruit and stem seeds—which are not edible and which are in a crude state and not advanced in value by refining or grinding or any other process of manufacture and not otherwise provided for; egg yolk; fuller's earth, in bulk only, not prepared for toilet or other purposes; lead, nitrate and acetate of, not ground; litmus and all lichens, prepared or not prepared; musk, in pods or in grain; roots, medicinal, viz.:—alkanet, crude, crushed or ground, aconite, calumba, foliæ digitalis, gentian, ginseng, jalap, ipecacuanha, iris, orris root, liquorice, sarsaparilla, squills, taraxacum, rhubarb, and valerian, unground; vaccine and ivory vaccine points; gum chicle or sappato gum, crude; platinum and black oxide of copper, for use in the manufacture of chlorate; potash, chlorate of, not further prepared than ground, and free from admixture with any other substance; and bacteriological products or serum for subcutaneous injection.
517	Dyeing or tanning articles, in a crude state, used in dyeing or tanning, n.e.s.; berries for dyeing or used for composing dyes; turmeric, nut galls and extracts thereof; lac, crude, seed, button stick, and shell; indigo, indigo paste and extract of, and indigo auxiliary or zinc dust; persis, or extract of archill, and cudbear; terra japonica, gambier or cutch, extract of logwood, fustic, oak and oak bark, and quebracho; camwood and sumac and extract thereof, tanner's bark, hemlock bark, and oak bark; ground logwood, ground fustic, patent prepared dyes, and ground oak bark; iron liquor, solutions of acetate or nitrate of iron for dyeing and calico printing; madder and munjeet or Indian madder, ground or prepared, and all extracts of; red liquor, a crude acetate of aluminium prepared from pyroligneous acid, for dyeing and calico printing.
520	Fertilisers, uncompounded or unmanufactured, including phosphate rock, kainite or German potash salts, German mineral potash, bone dust, bone black, or charred bone and bone ash, fish offal or refuse, guano, and other animal or vegetable manures.

Victoria.

Classification of Articles in Customs Tariff.

The Board of Trade have received from the Department of Trade and Customs, Melbourne, a copy of Supplement No. 2 to the Customs Tariff Decisions, 1896, in which the following articles are classified as under :—

No.	Description of Articles.	Rate of Duty.
91	*Essence*—Lemon soluble, "Stevenson and Howell's," contains 8·6 per cent. proof spirit	12s. per liquid gallon.
193	*Glassware*—Instruments of glass for testing brine, as glassware not otherwise enumerated.	25 % ad val.
	Glue, Marine (pitch)	Free.
	Medicines, &c.—	
	Analgen and Argonin : are simple chemicals, and if not packed ready for retail sale or consumption, and not recommended.	Free.
285	Anti Streptococcus Serum ... if packed ready for retail sale or consumption or recommended	25 % ad val.
285	Anti-Diphtheritic Serum ... if packed ready for retail sale or consumption or recommended	25 % ad val.
285	Anti-Syphilitic Serum ... if packed ready for retail sale or consumption or recommended	25 % ad val.
	Calc Glycerophosphas. Ingredients in chemical combination not packed ready for retail sale, and not recommended.	Free.
	Carniferrin and Ferripyrin : are chemicals, and if not packed for retail sale or consumption, and not recommended.	Free.
285	Diuretin, in 1 oz. bottles, a compound not in chemical combination.	25 % ad val.
	Ferri Glycerophosphas. Ingredients in chemical combination, and if not packed ready for retail sale or consumption, and not recommended.	Free.
	Jalapin and Lysidine, in 1 oz. bottles, not packed ready for retail sale or consumption, and not recommended.	Free.
	Lactophenin and Trional : are simple chemicals, and if not packed for retail sale or consumption, and not recommended.	Free.
	Pepsin Aseptic, in 1 oz. packets, "Parke, Davis and Co."	Free.
386	*Milk, Peptonised*—"Savory and Moore's," as milk, preserved.	2d. per lb.
	Sheep Dip—"Hatch's" Colonial Glycerin ...	Free.
	V[illegible], composed of vaseline and mineral oil.	Free.

EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

The Adulteration of Sumach and the Essential Oils of Lemons, &c., in Italy.

Bd. of Trade J., Oct. 1897, 458.

A report, dated 7th September last, has been received at the Foreign Office from H.M. Consul at Palermo, enclosing translation of an Act which has been recently issued, but, on the above date, had not yet been put into force, concerning the adulteration of sumach and of the essential oils of lemon, &c.

The principal articles of the above Act read as follows :—Whosoever manufactures, keeps in stock, or puts on sale essences of "agrumi" (*i.e.*, of the citron family, *e.g.*, essence of lemon, bergamot, &c.), or ground or powdered sumach, mixed with other substances, must indicate clearly the quantity and quality of the added matter, upon the receptacles in which they are contained, upon the way-bills, the invoices, and the registers which refer to the same.

Transgressors will be punished by a fine of from 100 to 500 lire, to which may be added, in case of a repetition of the offence, imprisonment not exceeding one month.

Article 2. Whosoever sells essence of "agrumi," or ground or powdered sumach, which is impure, without the indications prescribed in the foregoing paragraph, or without otherwise letting the purchaser of the same know their quality, is punishable by a fine of from 200 to 1,000 lire, and, in the case of a second offence, by imprisonment not exceeding three months.

If the sale is made for foreign shipment the penalty is increased by one-half.

Warning to Intending Purchasers of Rubber Forests in Brazil.

Bd. of Trade J., Oct. 1897, 388.

The Foreign Office have received a report, dated 28th August last, from Mr. W. A. Churchill, H.M. Consul at Para, stating that he had received a letter from a British firm inquiring as to the possibility of their purchasing rubber forests with a view to successfully exploiting the rubber industry on a large scale.

Mr. Churchill continues as follows :—

"I made the necessary inquiries, and was universally informed that such an enterprise would be followed by absolute failure, and that it has been attempted many times by Europeans and Americans, but invariably ended in disaster. I accordingly replied in this sense, and explained the reasons, the principal one being the climate, which in most of the swampy, inundated rubber districts is fatal to white men, besides being injurious to the natives."

H.M. Consul also states that it has come to his knowledge that several magnificent (pestilential) forests of rubber-bearing trees will be offered for sale in England by speculators, and therefore thinks such steps should be taken as may be considered necessary to warn the British public, adding: "I would most decidedly discourage anyone from investing in such an undertaking."

British and German Drug Trade.

Foreign Office Reps., Annual Series, No. 1977 ; through Pharm. J.

The British and German drug trade for 1895 and 1896 is dealt with in a recent report, which gives some very interesting figures tending to prove that, as far as direct trade between the two countries is concerned, Great Britain not only holds the foremost position, but is increasing her export, while that of Germany is decreasing. The exports of drugs and druggists' goods from Great Britain to Germany were 59,479,000 kilos. in 1895, and 61,863,800 in 1896, an increase of 2,384,800 kilos. British imports from Germany, however, during the two years were 42,520,600 kilos. in the former, and 41,791,400 kilos. in the latter year, a decrease of 729,200 kilos.

The German Chemical trade with England has also suffered more or less during the last year ; still this branch is one of the most important ones for the trade of both countries. Only a few of the chemicals in which the London market specially participates are dealt with in the report, the first being sulphate of ammonia.

Sulphate of Ammonia.

Of the entire German export, 12,651 d.c.,* 7,236 d.c. came to Great Britain, compared with 15,217 d.c. and 7,317 d.c. in the previous year. Over-production has caused this article to suffer a great fall in price, the English market price of sulphate of ammonia falling from 8*l.* 9*s.* per ton in January to 7*l.* 8*s.* in December 1896, the average price being 7*l.* 18*s.* per ton, as against 9*l.* 15*s.* in 1895, and 13*l.* 3*s.* in 1894.

Aniline Products.

The supply of aniline productions by Germany to foreign countries has considerably increased during the past year, though it did not attain the same increase as in 1895 compared with 1894.

Cochineal.

Cochineal has, in consequence of over-production, suffered a serious fall in price. The English deliveries in 1896 amounted to 3,275 serons (bales), compared with 2,457 in 1895.

Glycerin.

The import of refined glycerin into Germany has increased: 16,444 kilos. were imported in 1896, as against 13,768 in 1895. Of this, Great Britain sent 4,905 kilos. The export of refined glycerin amounted to 26,125 kilos., compared with 18,982 kilos. in 1895. Of this quantity Great Britain imported 5,903 kilos. The prices yield at present to the influence of the American market, but it seems probable that this will cease to be the case, as America will produce sufficient to supply her home demand.

* Double centner = 100 kilos.

Sulphate of Copper.

The prices for sulphate of copper have steadily increased since April 1896, the whole year showing numbers as to the consumption and delivery which have never before been reached. The total deliveries in open market (German) amounted to 217,066 tons in 1896, an increase in the European consumption of 50 per cent. The market opened in January at a price of 40*l.* 11*s.* 3*d.*, reaching its highest point in June, 50*l.* 5*s.*, the concluding price of the year being 49*l.* 10*s.*

Mexican Import of Chemical Products.

Foreign Office Reps., Annual Series, No. 1985, p. 5; through Pharm. J.

The Mexican import of chemical products amounted to 338,487*l.* in 1896, compared with 304,623*l.* in 1895. A noticeable feature in this branch of trade was the great increase in the importation of paints and colours, which in 1895 only amounted to 27,812*l.*, while in 1896 it represented a value of 64,063*l.*, or more than 130 per cent. difference.

Export of Chicle from Mexico.

Ibid., p. 9.

The export of chicle from Mexico during 1896 exceeded by nearly 1,000,000 lb. that of the preceding year. Hitherto it has been used almost exclusively as a chewing gum, but experiments are being made to ascertain if it cannot be used for certain purposes as a substitute for india-rubber.

Nitrate of Soda.

Foreign Office Reps., Annual Series, No. 1994, p. 8; through Pharm. J.

Nitrate of soda was introduced into the port of Antwerp during 1896 to the total amount of 133,500 tons, as against 118,370 tons in 1895. Other fertilisers imported were Peruvian guano, 20,000 tons; phosphates, 12,000 tons; sulphate of ammonia, 24,000 tons.

Chemicals in Morocco.

Foreign Office Reps., Annual Series, No. 1995, p. 21; through Pharm. J.

Chemicals to the value of 1,773*l.* (1,303 cwt.) were imported at Laraiche (Morocco) in 1896, as against 1,174*l.* (746 cwt.) in the previous year.

The World's Production of Nickel.

Eng. and Mining J., Oct. 16, 1897, 452.

The production of metallic nickel, including the metallic contents of that sold in the form of oxide, sulphide, sulphate, &c., in 1894—1896 was as follows (the figures being metric tons):—

Year.	Prussia.	From New Caledonia Ore smelted in France and England.	Total New Caledonia.	Canada.	Norway.	World's Total.
1894	522	1,900	2,422	2,226	90	4,738
1895	698	1,850	2,548	1,764	90	4,402
1896	822	2,150	2,972	1,541	90	4,603

Foreign Competition in the Colonies.

Chem. and Druggist, Oct. 2, 1897, 560.

We subjoin a few more abstracts of pharmaceutical interest from the recent Blue-book on foreign goods in the colonies.

Newfoundland.

The Newfoundland report has been compiled from official statistics and information obtained from merchants.

It is stated that only in a few classes of goods, among which are *Medicines*, the United States are superseding Britain. The medicines imported are chiefly proprietary articles. In *Soaps*, America is losing ground. In 1884, Newfoundland took two-thirds of its soap from the United States, in 1894 hardly any. The same is the case in *Painters' colours*. Here are the figures.

——	1884.	1889.	1894.
	Dols.	Dols.	Dols.
Medicines—total import	32,648	30,329	45,835
Of which from U.S.A.	2,125	3,683	10,775
Soap—total import	34,475	22,191	38,290
Of which from U.S.A.	12,250	8,884	2,360

The British West Indies.

Bahamas.

The Bahamians, says Sir W. H. Smith, buy 75 per cent. of their requirements in the United States, and the proportion is increasing.

Here are some plain statements:—

"Every merchant of the colony has some anecdote to relate as to the mode in which his business has been injured by the indifference of the British merchants.

"Attempts have been made to open a trade in English goods with Cuba. Those who have tried it, however, found that the English merchants would not take the trouble to suit the goods to the requirements of the special market, and the attempts have failed.

"The English carrying trade to many places on the Spanish Main, and in Hayti and Porto Rico, has been driven out by the German steam lines, which are worked most economically. The whole of this carrying trade was British, but it has gone in consequence of the German lines paying close attention to the convenience of shippers."

Bermuda.

Most of the trade of Bermuda is with the United States. The tendency is a natural one, on account of the geographical position of the islands, and is increased by the rapid and frequent steam communication, and the frequency with which American commercial travellers visit Bermuda.

Medicines (principally patents) are chiefly imported from U.S.A.

——	1884.	1889.	1894.
	£	£	£
The total value was..........	2,128	3,186	3,630
Of which from U.S.A.	1,399	2,486	2,426

Soap.—Mostly from the United States. "Premiums" are offered to purchasers of certain quantities of soap. American soap is put up in small coloured-paper packets, which makes it attractive; while soap imported from the United Kingdom is put up in long bars, and, although of better quality, the absence of "premiums" causes it to be in less demand.

British Honduras.

The British Honduras Report is, as all such reports should be, the work of commercial experts appointed by the Governor of the Colony. The imports of foreign goods, other than provisions, are small, and mostly find a market because the goods are low in price. The imports or *Drugs and Chemicals* were:—

——	1884.	1889.	1894.
	Dols.	Dols.	Dols.
From United Kingdom	2,206	1,187	1,014
„ United States.........	1,361	1,033	1,076
„ other parts	105	52	39
Perfumery:			
From United Kingdom ...	625	1,055	438
„ other parts	1,118	1,289	1,036

Soap, nearly all British, is imported to the extent of about 6,000*l.* a year.

Jamaica.

The following figures relate to the imports of certain pharmaceutical goods in 1894:—

Glass Bottles.—6,055*l.*, of which 2,613*l.* from U.S.A.

Instruments (Surgical, &c.).—727*l.*, of which 238*l.* from U.S.A.

Soap.—22,381*l.*, of which 380*l.* from U.S.A.

Perfumery.—5,664*l.*, of which 1,975*l.* foreign.

Where foreign goods compete successfully with British, it is because the foreign houses pay more attention to local requirements, give superior finish, lower price, more careful packing without any special charge therefor, cheaper freights, and more rapid communication.

In cordage, twine, certain classes of cotton goods, hardware, and cutlery, the United States have gained ground on Britain. The trade statistics show a large increase of imports from the United States and Germany, but this increase is much more apparent than real, because the tendency throughout the West Indies is to use New York as a port of transit, although much of the merchandise carried through that port is not American. The increase of imports from Germany (90*l.* in 1884, 24,966*l.* in 1894), is due to the establishment of a direct steamship line, and to the British Merchandise Marks Act.

The presence in Jamaica of many German business houses, the cheapness of labour in Germany, the special efforts made by the German Government to extend its foreign trade also assist. The bulk of the Jamaican population want cheap goods, irrespective of quality.

Other West Indian Islands.

Medicines.—Most of the foreign imports are American patents. All other drugs come from Britain. The following are the principal patents used:—

American:—Lung-balsam, 8*d.* per bottle of 4 oz.; Pymy pectoral, 8*d.* per bottle; Canadian healing oil, 8*d.* per bottle; Pain-killer, 6*d.* per 2-oz. bottle; Condition-powders, 7*d.* per pack; Ross's pills, 3*l.* 6*s.* 8*d.* per gross; Williams's pink pills, 4*l.* per gross.

French:—Vinaigre des 4 voleurs, 1*d.* per oz.; Alcolat valvar blanc, 1*d.* per oz.; Eau des Carmes or Eau Mélisse, 1*d.* per oz.; Elixir antiglaireux, 1*s.* 8*d.* per 1-pint bottle; Boule des Mars, 9*d.* per oz.; Beaume tranquille, 1*d.* per oz.; Syrop de flore, 1*s.* a bottle; Syrop de choux, 1*s.* 2*d.* a bottle; Syrop de nafé, 1*s.* 4*d.* a bottle.

All these prices are in bond, delivered West Indian Islands.

Perfumery.—The bulk comes from Britain; "Florida-water" from U.S.A. and Germany.

The following figures represent the approximate imports of medicines, perfumes, and soap in other West Indian Islands in 1894:—

	Medicines.		Perfume.		Soaps.	
	Total.	Including Foreign.	Total.	Including Foreign.	Total.	Including Foreign.
	£	£	£	£	£	£
Dominica, Antigua, Montserrat, &c.	2,000	500	..	..	3,000	350
Windward Islands ..	800	100	..	..	1,150	140
Barbados	5,700	1,500	2,650	1,250	..	..
Trinidad	10,000	3,500	..	..	..	..

There appears to be little or no displacement of trade in these articles from Britain to foreign countries. Where there is, the reasons are better packing, better finish, more suitable patterns.

Mines and Quarries. Mineral Statistics of the United Kingdom of Great Britain and Ireland, with the Isle of Man, for the Year 1896. (C.—8564.)

This is an annual statement prepared by the direction of the Secretary of State for the Home Department. In the introduction it is stated that the present volume contains an account of the quantity and value of the minerals obtained from mines, quarries, brineworks, &c. in the United Kingdom. In an appendix is shown the production of minerals in the British Colonies and Possessions compiled from official reports.

Mineral Produce of the United Kingdom.

Description of Mineral.	1895.		1896.	
	Quantity.	Value.	Quantity.	Value.
	Tons.	£	Tons.	£
Alum clay (bauxite).	10,108	2,506	7,429	1,918
Alum shale	2,063	258	..	..
Arsenic	4,798	52,198	3,616	15,483
Arsenical pyrites	2,951	2,785	8,808	8,007
Barytes	21,170	23,059	23,737	25,590
Bog ore	5,652	1,413	6,652	1,663
Chalk	2,924,235	153,864	3,559,229	157,170
Chert and flint	94,787	16,661	107,967	17,039
Clays	9,796,086	1,839,607	11,341,782	1,442,065
Coal	189,661,362	57,231,213	195,361,260	57,190,147
Copper ore	7,531	21,912	8,970	21,586
Copper precipitate ..	280	2,855	198	2,124
Fluorspar	36	54	394	478
Gold ore	13,266	16,584	2,765	4,257
Granite	1,667,766	547,999	1,756,816	498,074
Gravel and sand	1,014,477	81,107	1,268,310	90,020
Gypsum	177,892	71,835	193,311	74,538
Iron ore	12,615,414	2,865,709	13,700,764	3,150,424
Iron pyrites	9,048	4,114	10,017	4,003
	Lb.		Lb.	
Jet	168	16	294	10
	Tons.		Tons.	
Lead ore	38,412	273,392	41,069	303,398
Limestone (other than chalk).	9,525,039	1,205,261	11,011,350	1,215,604
Manganese ore	1,273	681	1,080	613
Ochre, umber, &c....	7,625	16,989	9,891	24,688
Oil shale	2,246,865	561,710	2,419,525	604,881
Petroleum	15	28	12	29
Phosphate of lime...	500	875	3,000	5,250
Plumbago	40	100	Nil	Nil
Quartz	724	550	519	389
Salt	2,173,253	709,751	2,022,357	666,613
Sandstone	5,230,526	1,366,596	4,507,745	1,417,985
Slag	134,882	5,888	562,293	15,358
Slate and slabs	581,760	1,274,146	586,933	1,338,256
Soapstone	Nil	..	18,042	5,188
Strontium sulphate.	12,273	3,529	7,663	259,928
Tin ore	10,612	370,530	35	1,500
Uranium ore	40	2,071	2,286,999	425,587
Whinstone, basalt, &c.	1,728,350	352,382	43	1,355
Zinc ore	17,178	19,430	19,319	66,553
Total values...	..	69,129,664	..	69,088,366

The following table shows the quantities of metals obtained by smelting from ores mined in the United Kingdom; the values here given are based on the average market price:—

Metals Smelted from British Ore.

Metal.		1895.		1895.	
		Quantity.	Value.	Quantity.	Value.
			£		£
Copper	Tons	579	27,263	556	28,180
Gold	Oz.	6,600	18,520	1,352	5,035
Iron	Tons	4,394,987	10,534,325	4,759,446	11,375,474
Lead	..	29,000	308,734	30,818	350,940
Silver	Oz.	280,434	34,908	283,826	36,365
Tin	Tons	6,648	446,780	4,837	307,678
Zinc	..	6,654	101,695	7,110	123,250
Total values ..		..	11,472,225	..	12,226,912

American Cotton-Seed Oil in Marseilles.

U.S. Cons. Reps. 1897, 54, [202], 430—432.

The supply of sesame, peanut, poppy, linseed, castor, and cotton seed from India having been entirely cut off owing to the bubonic plague, there is a scarcity of seed in the market here for the manufacture of oils, and American cotton-seed oil is now being largely used by soap manufacturers, &c., owing to the higher prices of peanut, olive, and cocoa-nut oils. The cheapest grade of olive oil for machinery purposes is quoted at 54 to 64 fr. per 100 kilos. (reductions made at the rate of 3½ kilos. to the gallon or 32 galls. to the 100 kilos.), or 34 to 39 c. per gallon; and peanut oil at 46 fr. per 100 kilos., or 28 c. per gallon; whilst American cotton-seed oil is selling at 40 fr. per kilos.,

or 24 cents per gallon. The following table shows the receipts of American cotton-seed oil at Marseilles during the past seven years:—

Year.	Quantity.		Year.	Quantity.	
	Barrels.	Kilos.		Barrels.	Kilos.
1890	66,024	10,809,190	1894	12,530	2,105,789
1891	56,302	9,308,217	1895	59,528	10,072,481
1892	36,798	6,093,979	1896	112,627	20,331,125
1893	13,860	2,254,093			

During the year 1896 the total receipts from all other countries than the United States were only 11,335 barrels, 11,109 of which came from British provinces.

The following quotations of the relative prices of American and English cotton-seed oils, during the past seven years, indicate that the latter is inferior in quality to the former:—

Year.	Price per Gallon.		Year.	Price per Gallon.	
	American Oil.	English Oil.		American Oil.	English Oil.
	Cents.	Cents.		Cents.	Cents.
1890	35	34⅓	1894	35¼	32 3/16
1891	34½	33⅓	1895	29 3/16	28 3/16
1892	32	31¼	1896	26⅗	26
1893	43	37			

—A. S.

GENERAL TRADE NOTES.

The Production of Coca and Cocaine in Peru.

Bd. of Trade J., Oct. 1897, 476.

The *Economista*, of Lima, in an article on this subject, says that 20 years ago coca was only cultivated in a few provinces which were most favoured by reason of climate and cheap labour. Since then the industry has rapidly developed. In the province of Otuzco, the most important district for the production of coca in Northern Peru, the number of plants is computed at 2,700,000, and the annual production amounts to 4,700 quintals. It is worth remarking that most of the plants in this province have not yet attained their full growth, and that in a few years the production will increase probably to 10,000 to 12,000 quintals.

The coca is sold by two houses in Truxillo, which dispose of the product to the cocaine manufacturers in Lima. As they exercise a sort of monopoly the price varies from time to time, the Lima manufacturers sometimes being obliged to pay as much as 32 sols (1 sol equals 2s.) per quintal for raw coca delivered free on board at Salaverry. The proprietors of the haciendas of Huayobamba and of Cayanchal are planning to establish cocaine factories in the neighbourhood of their own plantations, and the competition thus engendered will probably lower the price.

In order that the coca may be cultivated under the most favourable conditions it is necessary that the temperature should rarely fall below 24° C., and it should be about 30°. As for the altitude of the plantations, they are usually situated at a height of 3,000 to 4,000 ft. above sea-level. There are very few plants grown at a height of 5,000 ft., and the coca under these conditions is usually of an inferior quality. The quality of the coca varies also according to the soil; that obtained from a dry soil is better than that grown in damp ground. It is for this reason, for instance, that the coca of Chuquillanqui, though sometimes attaining a height of 10 ft., does not by any means possess the strength and the aroma of that which is grown in other parts of Peru. The shipments of coca from the ports of Huanchaco and Salaverry, which so far as the foreign trade is concerned are coincident with all Peru, were as follows for the years 1894 and 1895.

—	Huanchaco.	Salaverry.
	Kilos.	Kilos.
1894	736	611,158
1895	85,716	159,006

The Algerian Phosphate Deposits.

Eng. and Mining J., Oct. 9, 1897, 427.

M. Chateau publishes, in the *Memoirs* of the French Society of Civil Engineers for August 1897, an important monograph on the phosphate deposits of Constantine and Algeria. The paper covers 118 pages, and contains a historical account of the discovery of sedimentary phosphates on the high tableland of Algeria, with a description of the geological structure, the nature of the phosphates, and the theory of their formation. The various deposits are described in detail, and particulars are given of the cost of production, the exports, the future of the industry, and the legislative enactments relating to it.

Cadmium.

Eng. and Mining J., Oct. 9, 1897, 421.

The increase in the demand for cadmium and the shortage in the supply still continue, notwithstanding a little more is being made in Upper Silesia (which is, indeed, practically the only district where cadmium is produced). The Upper Silesian production in the first quarter of the current year was 3,326 kilos. (valued at 11·844 marks per kilo.), against 3,256 kilos. (valued at 10·261 marks per kilo.) in the last quarter of 1896, and 2,436 kilos. (valued at 5·380 marks per kilo.) in the first quarter of 1896. At present, it is said, the metal is lacking entirely, and urgent demands for it cannot be satisfied. The cause for this peculiar situation is unknown. If the new demand proves to be permanent, however, there should be no difficulty in meeting it, since most of the Upper Silesian ore is cadmium-bearing, and the metal can be recovered without difficulty as a by-product at works where the distillation muffles are equipped with the Dagner or Kleeman condensers, and provision is made to collect the escaping fume. With such an arrangement the cadmium fume is deposited especially in the first part of the common flue, furnishing a product which, if saved separately, is rich enough for treatment to recover the cadmium. At present this is done at only two or three works, and others probably with be slow to make the necessary arrangements until it has been demonstrated that the increased demand for cadmium is likely to be permanent, for after all the present business does not amount to much.

Annual Meeting of the German Chemical Industrial Society.

Chem. and Druggist, Oct. 9, 1897, 573.

The Annual Congress of the German Society for the Protection of the Interests of Chemical Industry was held at Baden-Baden last week. In submitting his usual report of the condition of the chemical industry of the country, the President, Dr. Holtz, stated that in 1896 the output of chemical products increased by 8·7 per cent., the average number of workmen employed by 5·2 per cent., and the total amount paid in wages from 103·4 to 113·7 millions of marks, or from 894·16 to 906·58 marks per head. The exports increased by 6·5 per cent. in value, and by 13 per cent. in quantity. As a consequence of the general fall in prices, there was a reduction in the average net profits (as shown in the reports of the principal chemical companies) from 12·71 to 12·3 per cent. Another matter which came up for discussion, and gave rise to an interesting debate, was the question of whether it was desirable that a State examination should be instituted for technical chemists. This is a matter that has of late been widely discussed in the German technical press. Ultimately, a set of rules was adopted, declaring in substance that although German chemical industry was much interested in the most thorough training of technical chemists, they were not prepared to indicate any special means of arriving at this object, espe-

cially in consideration of the fact that effective measures are shortly to be taken to carry out the recommendations of the Union of Directors of Laboratories of German Universities in connection with this matter, and that the Imperial Government has announced that a Commission will be constituted at the end of October to inquire into the matter. The Society therefore intend at present to maintain a neutral attitude. The Commission of inquiry referred to will consist of representatives of the German Universities, of the technical high schools, and of the chemical industry. Dr. Martius read a report on the projected participation of German chemical industry at the next Paris World's Fair. It is suggested that the industry should exhibit as one collective whole, the objects shown being representative of every branch of the industry, because the room set apart for this section is insufficient to enable all firms to have suitable individual exhibits. This plan of the collective exhibit is strongly supported by the German Imperial Commissioner for the Paris Exhibition, Mr. Richter. It was then decided that the matter should be taken in hand by several sub-committees, each representing a special branch of chemistry. Another matter of importance that was discussed was the proposed central bureau for the preparation of a commercial treaty. The new German Customs tariff is in preparation, and this will probably lead to the collection of new commercial treaties with various countries. The German Government intend to consult all branches of national industry on this subject, and the Society adopted a resolution hoping that means would be found to avoid dissension among the various industrial branches.

Inland Revenue Statistics.

Chem. and Druggist, Oct. 2, 1897, 554.

The methylation of spirits is an increasing business. In the year ending March 31, 1896, the quantity methylated in the United Kingdom reached 3,930,072 gals.: in the year ending last March the quantity was 4,143,310, an increase of 213,238 gals. There were 25 makers of methylated spirits licensed in 1896, and 13,801 licensed retailers.

At the Inland Revenue branch of the Government Laboratory, a total of 64,664 samples were examined during the year ended March 31 last. This was an increase of nearly 10,000 samples over the previous year. There were 28,875 samples of beer, and 13,875 of tobacco, tested during the year. Only 60 samples were referred to the laboratory under the Sale of Food and Drugs Act (82 the previous year). The exportation of tinctures, &c., under the drawback system is still progressing. The number of samples tested last year for this drawback was 8,182, against 7,000 the year previously.

The number of chemists who paid licenses for stills in 1896 was 703 in England, 115 in Scotland, and 25 in Ireland.

Cloves and Caffeïne in France.

Chem. and Druggist, Oct. 9, 1897, 575.

At the last meeting of the Paris Syndical Chamber of Chemical Products, M. Adrian mentioned that the Excise authorities had given distillers facilities for preparing oil of cloves, which was formerly not permitted. The cloves are admitted duty free, and, after having been used for preparing the oil, are burnt in the presence of an Excise officer. M. Adrian thinks the same favour might be accorded to chemists for the preparation of certain alkaloids, and he especially referred to caffeïne as being one of the most important on account of its increased use.

The Production of Salt.

J. Soc. Arts, 1897, 1114.

The quantity value of salt produced in the United Kingdom, and used in brine during the five years ended 1895, are as follows:—In 1891, 2,043,571 tons, valued at 976,824*l.*; 1892, 1,956,524 tons, value 861,401*l.*; 1893, 1,924,029 tons and 735,222*l.*; 1894, 2,235,912 tons and 763,629*l.*; and 1895, 2,173,253 tons and 709,751*l.* The corresponding figures for 1896 are not yet available. In commenting upon the returns of salt production in the United Kingdom, it is stated in the report of the Salt Chamber of Commerce for 1896, that the ammonia-soda process has, by it increase, deprived of some portion of their previous markets those who produce salt for sale. The total trade of the United Kingdom exhibits an increase, the slight relapse in 1896 being due to exceptional freight circumstances. The export and coastwise figures, taken together, of the last five years are as follows:—In 1892, 958,306 tons; 1893, 949,785; 1894, 1,066,483; 1895, 1,080,601; and 1896, 1,022,617 tons. No fresh discovery of rock salt or brine has occurred in this period, except the interception of salt in the Isle of Man during some borings for coal; nor has there been in any part of the world any noteworthy expansion of the existing manufacture. Occasional efforts have been made in South America and Spain, and elsewhere, to utilise long known deposits of salt which are hopelessly remote from railway or ocean, from labour or fuel, or otherwise devoid of commercial importance. In the production of the United Kingdom there has been little change, except some readjustment to topographical and other economic advantages. All the salt districts of the country, especially the most advantageous, have been developed far in excess of either their respective local or foreign demand. The natural result has ensued: prices no longer, says the report of the Salt Chamber, offer any inducement to the enterprise of increasing the plethora. Germany having a few years ago reached the limits of her own consumption, has been pushing the salt into the markets of neighbouring countries, and with less success into more remote parts of the world. Her strength lies in the facilities and cheapness of her inland navigation and ports of shipment. In the United States the inflation of native production, caused by the indiscretion of English manufacturers in 1889 and 1890, has been partially corrected, but reports still show considerable struggles between the chief districts producing in excess of demand. The Red Sea has provided more salt for India. It is of inferior quality, but, like the Hamburg article, is mixed with English salt, and goes into consumption at a lower price. When Eastern freight, and consequently the price of English salt, is at a normal level, the market of these lower grades is limited. The Indian Government has powers under Adulteration Acts to restrain this mixture, but has not yet seen fit to exercise them.

The Chilean Nitrate Industry.

Eng. and Mining J., Sept. 25, 1897, 371.

Senor Don Alberto Herrman, in the *Boletin de la Sociedad Nacional de Mineria*, states that the cost of production at three nitrate mines is as follows:—

—	San Jorge.	Puntunchara.	San Pablo.
	Cents.	Cents.	Cents.
Extraction of the caliche.	27·68	32·47	59·40
Carting..................	12·90	23·46	21·26
Dressing.................	32·08	37·62	18·89
General costs	7·22	9·01	12·87
Various..................	0·17	1·32	..
Total	80·05	103.88	112·42

The Chilean nitrate industry has to contend against the competition of artificial soda manures and sulphate of ammonium, the output of which rapidly increases year by year, the discovery of nitrate deposits in other parts of the world, and the competition of guanos rich in nitrogen, phosphoric acid, and potash, like those recently discovered in the island of Corcovado, Peru. Lately, as is well known, the Chilean industry has been suffering a serious depression. The continuance of this is having an alarming effect on both the revenue and trade of Chile. Hundreds of workmen have been thrown out of employment in the nitrate districts, and they are being brought south, where, at the present time, there is no demand for labour. Realising the importance of revivifying the industry, the Government has joined the Permanent Nitrate Committee in the propaganda carried on by the latter to introduce the product in new markets. The ministry promises at least 75,000 dols., which

may possibly be increased to 200,000 dols., toward this object. The duty has been taken off some sample cargoes which have been sent to China and Japan, while special agents have been sent to the United States with a view of increasing the use of nitrate in this country.

Petroleum in Texas.

Eng. and Mining J., Oct. 2, 1897, 399.

Petroleum was discovered early this year at Corsicana, Texas, at a depth of 1,040 ft. At this horizon there is a bluish sandy shale about 20 ft. thick, which is saturated with petroleum. There is no rock, either above or below, the drill passing down the whole way through a hard clay. The Texas Petroleum Company, organised six months ago, has three flowing wells, the latest of which is said to be producing about 20 barrels per day. The Corsicana Oil Development Company has a well which is flowing 25 barrels. A correspondent of the *Pittsburg Times*, who has visited the district, states that according to the testimony of those familiar with the products of the different fields, Texas oil is better than the Ohio oil, but not as good as that found in Pennsylvania. It most resembles that produced at Neosha, Kansas. Its flow is like that of the wells in the Bradford field. An analysis of the oil showed about 90 per cent. of volatile matter and 10 per cent. of residuum. As compared with other oils, results are as follows :—

Crude Oil from	Specific Gravity at 17° C.	Began to boil at ° C.	Came over under 150° C.	Between 150° and 300° C.	Over 305° C.
			Per Cent.	Per Cent.	Per Cent.
Texas-Corsicana	821	80	54·6	40	15·8
Pennsylvania ..	818	82	21·0	38	40·7
Galicia	824	90	26·5	47	26·5
Baku	859	91	23·0	38	39·0
Alsace	907	135	3·0	50	47·0
Hanover	899	170	..	32	68·0

The line of development at present is about $1\frac{1}{4}$ miles long and covers an area of about 150 acres. It costs about 1,500 dols. to put down a well, and the royalty varies from one-eighth to one-tenth.

The present production is used exclusively for fuel. Some is consumed at the wells and in the local manufacturing establishments, and the rest goes to Dallas, Houston, Austin, &c. In the towns named it is being used in making illuminating gas. It is delivered in Corsicana for fuel at 75 cents a barrel. At present there is a market for all that is produced, but if the continued development increases the production, a larger market must be had, or a refinery established.

Saltpetre in Spain.

Chem. Trade J., Oct. 2, 1897, 215.

Saltpetre was at one time imported exclusively from England to *Oporto*, and for a long time after Germany began to compete, preference was given, even at a higher price, to the British article. Germany has, however, considerably improved her manufacture, and, owing to lower prices, is supplanting some English makes which were well known here. German competition is assisted by better terms, *i.e.*, longer credits than those granted by English firms, and also better terms are allowed to the local agents, who thus have a greater interest in pushing the products of that country.

Manufacture of Starch.

Chem. Trade J., Oct. 2, 1897, 215.

In spite of the dearness of the raw material on account of the famine in British India, the year 1896 was not an unfavourable one for *Belgian* rice starch manufacturers. The exports of starch and starch products of home manufacture amounted to 6,329 tons.

Citric Acid.

Chem. Trade J., Oct. 2, 1897, 215.

A factory for the manufacture of citric acid and oil of lemon was established at *San Diego* during 1896. It takes from four to six weeks to condense the juice of 60 to 70 lb. of lemons into 1 lb. of acid. The factory employs seven hands, has steam works, and a capacity for 40,000 lemons a week ; only culls are used. The same company also makes oil of lemon.

Canaigre in California.

Chem. Trade J., Oct. 2, 1897, 211.

Canaigre is the American corruption of the Spanish "cana agria," sour cane. It is also called "Yerba-Colorado" in Mexico, localisms being "red dock," "tanner's dock," and "wild rhubarb." The best way to propagate the plant is by use of small roots rather than by seed. About 1,000 lb. of tubers will plant an acre, and October and November are the best months for putting in the crop; though where irrigation can be practised, planting may be done at any time. The value of canaigre as a tanning agent, either alone or in connection with other tannins, has been proved beyond question. For light leather it gives great tensile strength, and is far better for split leather than gambier, oak, or hemlock. It is a quick tanner, and the yellow colour absorbed by the hide in the process of tanning is considered highly desirable for certain leathers. The sliced and dried tubers, containing an average of 30 per cent. of tannic acid, are worth from 8*l.* to 9*l.* per ton. A yield of from 7 to 10 tons per acre would give $2\frac{1}{2}$ to $3\frac{1}{2}$ tons of the dried product, for which there is a constant demand in Europe and America. Inasmuch as the plant grows wild in this vicinity, and the seed roots are readily obtained, the industry commends itself to the farmer of small means, as it is harvested in such a short period after planting.

A great deal of interest is being taken in the Los Angeles district in the cultivation of canaigre. It was stated in the Californian press in February last, that a syndicate of English and American capitalists has purchased 22,000 acres about 50 miles east of this city for 100,000*l.*, for the purpose of cultivating canaigre. The agent for the San Francisco Savings Union (the vendors) stated that the purchasers, the Anglo-American Canaigre Co., consist of an American and an English syndicate ; that the American syndicate is taking over the land, and the English syndicate is interested only in the product. The vendors acquired the property under foreclosure, and are selling at cost price. The company is planting 8,000 acres this season.

Chemical Works in Sweden.

Chem. Zeit.; through Eng. and Mining J., Oct. 2, 1897, 396.

The manufacture of sulphuric acid in Sweden amounts to about 35,000 tons of chamber acid per annum. The greater part of this production is used for the manufacture of superphosphates. The principal producers are the Joint Stock Company, of Stockholm, and the Scandinavian Joint Stock Superphosphate and Sulphuric Acid Manufacturing Company, of Helsingborg. This latter is the most important one of the kind in Scandinavia. It produces annually about 18,000 tons of chamber acid and about 30,000 tons of superphosphate. It is the only concern, also, in any of the Scandinavian countries that produces the double superphosphate containing as much as 45 per cent. of soluble phosphoric acid.

Sulphuric acid is very largely used in Sweden for the manufacture of sulphate of copper. The "Stora Kopparbergs Bergslags Actiebolag," the works of which are at Falun, produces annually 1,500 tons of sulphate of copper. This company, which possesses the celebrated Falun copper mine, and has a capital of 12,650,000 frs., is the most important in Sweden, and produces, besides sulphate of copper, 50,000 tons of cast iron, 43,000 tons of manufactured iron and rolled and forged steel, 7,000 tons of Thomas slag, 6,000 tons of paper, 1,000 tons of red mineral paint, 2,000 tons of sulphuric acid, 350 kilos. of silver, and 100 kilos. of gold per annum.

Chemical Industry in the Transvaal.

C. Butters. Proc. Chem. and Metall. Soc. of S. Africa, July 17, 1897, 3—10.

Copper has been produced at the Willows and the Albert silver mines, and silver-lead at the Transvaal silver

mines, but the ores are not rich enough to stand the excessive cost of production in this district. There is one sulphuric acid works, producing an acid of low commercial strength, but the manufacture is not firmly established, owing to the small demand for the product, and in spite of a protective import duty of 1*d.* per lb. Dynamite is not made locally, but its manufacture is strongly advocated (of low grade) for blasting purposes. Glass is now being made at the Lewis and Marks distillery in Pretoria. Coke is being prepared, but although daily improving in quality, it is not yet sufficiently good for use in the iron and steel industries. The author is sanguine as to the ultimate success of this manufacture, as well as of the ore of pig-iron and steel. At present the local coke has to be mixed with from one-third to one-half of foreign coke for blast-furnace purposes; it contains about 17 per cent. of ash, as against 11 per cent. in English coke. Although at present the price is abnormally high, there is no reason why it should not be sold presently at from 2*l.* 10*s.* to 3*l.* per ton. The coal supply is practically inexhaustible, and is essential to the working of gold. Methods of handling the coal are imperfect, and it is still impossible to buy from any colliery an even grade of coal. The percentage of ash in the steam coal ranges from 20 to 25 per cent.; that in smithy coal averages 10 per cent. Agriculture is still in a primitive state. Galena occurs abundantly, some samples containing 80 per cent. of lead. The extraction is carried on locally, but it is restricted, owing to the limited demand for the metal. The industry is protected by an import tax of 3*d.* per lb.

The main industry, however, is that of gold extraction. Here cyanide treatment is slowly but surely replacing chlorination everywhere for the treatment of slimes. The author believes that the next development will lie in the direction of stamping with a dilute solution of cyanide instead of with water, no water-washes being thrown away. With very poor ores, preliminary amalgamation could then be dispensed with, any free gold present being caught in the concentrates and washed down over a single plate after cyaniding. Such a method, in conjunction with electrolytic precipitation, would form a strong rival to dry crushing processes; especially as with pyritous ores the material must be crushed sufficiently fine to pass a 200- to 400-mesh screen. The African Gold Recovery Co. tried a similar system in 1892, but failed to work it out, owing to the difficulty (since surmounted) of separating the liquid from the pulp.

—W. G. M.

Production of Metals in British Columbia.

Eng. and Mining J., Oct. 16, 1897, 451.

It was not very long ago that the entire mineral product of British Columbia came to the United States in the form of ore. Smelting works were erected in the province a little later, but the silver-lead and the copper matte were still sent to the United States for refining and further reduction respectively. Now, however, British Columbia is beginning to turn out its product in a higher state of completion. The Trail Creek works have installed a refinery, and are now shipping gold in fine bars, while the Hall Mines, Ltd. (Silver King mine), is converting its product into blister copper, which is going directly to Wales. Incidentally it may be remarked that this company has one of the largest smelting furnaces in operation on this continent. With the entrance of Vivian and Sons into the British Columbia ore market, and a freight rate of only 8·50 dols. per ton from Vancouver to Swansea, American ore buyers will have to look sharply to the business.

Exports of Tinctures in Bond.

Chem. and Druggist, Oct. 23, 1897, 671.

An Inland Revenue order (No. 15, Oct. 7, 1897) has been issued, in which further concessions are made for the exportation of tinctures, medicinal spirits, flavouring essences, and perfumes. The condition allowing essences to be packed in 1-gal. cases, when in bottles not exceeding 4 oz. each, has now been extended to medicinal spirits, but not less than 2 liquid gals. are to be regarded as a legal quantity for exportation in one consignment. Medicinal spirits, essences, and perfumes may be packed in the same case, when so desired, if packed in separate internal packages. It has been made permissible to export 10 gals. (formerly 5) of medicinal spirit or essence in a jar covered with wickerwork. An important concession is the withdrawal of the regulations which prescribed the minimum and progressive sizes of bottles of perfumed spirits, bottles of any size being not allowed, subject to existing conditions as regards internal packages. Non-dutiable goods may also be packed in with duty paid, provided that they are placed in an internal package marked "Free goods."

BOARD OF TRADE RETURNS.

Summary of Imports.

Articles.	Month ending 30th September 1896.	Month ending 30th September 1897.
	£	£
Metals	1,614,578	1,651,108
Chemicals and dyestuffs	450,151	347,745
Oils	676,216	721,991
Raw materials for non-textile industries.	4,541,461	5,463,168
Total value of all imports	33,006,346	35,195,893

Summary of Exports.

Articles.	Month ending 30th September 1896.	Month ending 30th September 1897.
	£	£
Metals (other than machinery)	2,780,378	2,825,884
Chemicals and medicines	639,278	636,378
Miscellaneous articles	2,670,516	2,525,220
Total value of all exports	19,824,125	18,305,275

Imports of Raw Material for Non-Textile Industries for Month ending 30th September.

Articles.		Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
				£	£
Bark, Peruvian	Cwt.	2,724	1,490	3,800	2,612
Bristles	Lb.	437,628	488,181	62,907	63,722
Caoutchouc	Cwt.	32,859	21,700	403,469	230,193
Gum:—					
Arabic	"	2,379	4,282	4,574	13,341
Lac, &c.	"	3,176	10,584	15,531	14,153
Gutta-percha	"	3,204	4,454	32,030	39,841
Hides, raw:—					
Dry	"	23,906	35,349	58,539	86,020
Wet	"	53,046	53,003	119,775	106,020
Ivory	"	1,030	1,020	42,071	46,502
Manure:—					
Guano	Tons	1,396	3,242	12,450	14,937
Bones	"	2,331	2,790	6,068	10,308
Nitrate of soda	"	1,093	8,309	8,475	60,008
Phosphate of lime	"	25,081	37,941	36,930	56,320
Paraffin	Cwt.	78,400	60,654	73,401	49,302
Linen rags	Tons	1,092	1,925	12,352	18,378
Esparto	"	12,120	20,843	59,259	81,012
Pulp of wood	"	28,912	26,804	145,266	126,851
Rosin	Cwt.	108,512	129,731	27,016	31,686
Tallow and stearin	"	97,281	169,397	94,790	159,202
Tar	Barrels	20,548	26,732	17,428	17,336
Wood:—					
Hewn	Loads	258,891	275,874	518,117	596,528
Sawn	"	822,690	1,025,668	1,836,946	2,421,915
Staves	"	14,050	14,604	56,905	54,758
Mahogany	Tons	7,324	9,760	62,992	84,590
Other articles	Value £	..	..	830,370	1,047,333
Total value		..	..	4,541,461	5,463,168

Besides the above, drugs to the value of 55,177*l.* were imported, as against 70,413*l.* in September 1896.

Imports of Chemicals and Dyestuffs for Month ending 30th September.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Alkali Cwt.	10,238	14,483	7,949	8,698
Bark (tanners', &c.) „	25,572	25,685	8,149	8,004
Brimstone „	42,804	16,760	11,068	3,777
Chemicals Value £	..	..	127,102	96,625
Cochineal Cwt.	686	369	4,641	2,282
Cutch and gambier Tons	1,894	1,821	32,960	29,575
Dyes:—				
Alizarin Value £	..	..	19,393	21,035
Anilin and other „	..	..	40,046	40,339
Indigo Cwt.	830	406	11,040	5,494
Nitrate of potash „	24,671	22,063	20,047	16,994
Valonia Tons	2,865	2,418	33,043	24,095
Other articles Value £	..	..	135,013	90,827
Total value of chemicals	..	..	450,451	347,745

Imports of Oils for Month ending 30th September.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Cocoa-nut Cwt.	14,398	41,212	15,694	42,632
Olive Tuns	961	2,086	31,199	71,882
Palm Cwt.	89,644	65,625	91,814	68,092
Petroleum Gall.	15,393,970	15,470,549	276,296	284,396
Seed Tons	2,655	2,892	55,433	63,925
Train, &c. Tuns	2,370	3,300	38,431	47,568
Turpentine Cwt.	72,187	55,660	68,269	57,041
Other articles Value £	..	..	99,080	86,455
Total value of oils	..	..	676,216	721,991

Imports of Metals for Month ending 30th September.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Copper:—				
Ore Tons	10,704	12,596	44,422	48,158
Regulus „	5,685	5,774	154,242	128,621
Unwrought „	5,246	4,433	255,684	218,643
Iron:—				
Ore „	419,855	420,823	296,206	306,830
Bolt, bar, &c. „	7,407	6,149	59,582	48,469
Steel, unwrought „	1,446	2,181	17,731	15,269
Lead, pig and sheet „	12,073	15,409	136,690	196,209
Pyrites „	56,504	49,518	103,153	78,320
Quicksilver Lb.	64,200	81,974	5,680	7,660
Silver ore Value £	..	..	97,162	167,242
Tin Cwt.	67,347	41,876	202,932	125,816
Zinc Tons	5,227	6,386	87,830	108,554
Other articles Value £	..	..	152,964	201,317
Total value of metals	..	..	1,614,578	1,651,108

Exports of Drugs and Chemicals for Month ending 30th September.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Alkali Cwt.	381,326	388,595	100,554	99,083
Bleaching materials „	90,042	110,416	29,992	34,483
Chemical manures Tons	39,556	38,881	159,804	162,453
Medicines Value £	..	..	95,338	91,704
Other articles „	..	..	253,590	248,655
Total value	..	..	639,278	636,378

Exports of Metals (other than Machinery) for Month ending 30th September.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Brass Cwt.	9,280	8,905	40,092	39,468
Copper:—				
Unwrought „	33,049	33,635	81,919	86,413
Wrought „	18,978	30,125	58,619	92,526
Mixed metal „	16,299	22,398	39,780	52,805
Hardware Value £	..	..	175,771	168,490
Implements „	..	..	118,192	115,273
Iron and steel Tons	314,736	283,559	1,978,784	2,013,675
Lead „	3,730	3,224	45,851	43,948
Plated wares Value £	..	..	39,055	35,680
Telegraph wires „	..	..	76,273	46,127
Tin Cwt.	12,048	9,669	38,296	31,583
Zinc „	15,574	11,329	11,925	9,523
Other articles Value £	..	..	74,921	90,373
Total value	..	..	2,780,378	2,825,884

Exports of Miscellaneous Articles for Month ending 30th September.

Articles.	Quantities. 1896.	Quantities. 1897.	Values. 1896.	Values. 1897.
			£	£
Gunpowder Lb.	351,800	769,400	9,823	21,429
Military stores Value £	..	..	71,240	71,862
Candles Lb.	1,746,000	1,947,000	26,067	26,625
Caoutchouc Value £	..	..	99,569	112,486
Cement Tons	34,898	32,134	55,376	55,464
Products of coal Value £	..	..	118,971	103,119
Earthenware „	..	..	137,646	126,111
Stoneware „	..	..	17,238	13,332
Glass:—				
Plate Sq. Ft.	109,823	133,902	7,148	7,291
Flint Cwt.	9,421	7,822	19,400	19,069
Bottles „	69,246	61,473	33,971	30,213
Other kinds „	20,351	19,348	15,515	13,807
Leather:—				
Unwrought „	12,304	13,043	109,709	106,949
Wrought Value £	..	..	44,751	39,931
Seed oil Tons	5,186	3,417	93,399	62,332
Floorcloth Sq. Yds.	1,745,700	1,664,700	74,508	75,787
Painters' materials Val. £	..	..	130,924	118,041
Paper Cwt.	87,779	80,354	137,227	118,751
Rags Tons	4,683	4,459	27,063	23,598
Soap Cwt.	46,976	60,795	52,362	61,309
Total value	..	..	2,670,516	2,525,220

Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—PLANT, APPARATUS, AND MACHINERY.

Applications.

21,665. F. L. Ball. *See* Class II.

21,757. H. W. Jarvis. Improvements in the construction of evaporative condensers for steam or other gases. Sept. 22.

21,989. W. D. Cliff and The Leeds Fireclay Company, Ltd. Improvements in or in connection with inclined retorts. Sept. 25.

22,020. H. Higgins. Improvements in or relating to vacuum apparatus for evaporation. Sept. 25.

22,026. A. W. Stokes and R. A. Lister and Co., Ltd. Improvements in apparatus for automatically measuring off quantities of liquid specially applicable for testing the amount of fat in milk. Sept. 25.

22,027. A. N. Stokes and R. A. Lister and Co., Ltd. Improvements in apparatus for testing the acidity of milk and other liquids. Sept. 25.

22,046. J. Y. Johnson.—From H. K. Mulford, United States. Improvements in machines for compressing powdered materials into tablets. Sept. 25.

22,066. J. Dymond. Improvements in or relating to gas producers. Sept. 25.

22,141. P. F. C. Willcox, H. W. Willcox, and R. J. N. Willcox. Improvements in and relating to apparatus for heating or evaporating liquids by means of steam. Sept. 23.

22,236. J. D. Darling and C. L. Harrison. Improvements in porous diaphragms for electrolytic apparatus. Complete Specification. Sept. 28.

22,704. Sir C. S. Forbes, Bart. Improvements in or connected with hot water heating and drying apparatus. Oct. 4.

22,705. Sir C. S. Forbes, Bart. Improvements in apparatus for condensing steam and vapour, refrigerating liquids and the like. Oct. 4.

22,777. P. R. J. Willis.—From R. McCoy, United States. Improved receptacle for shipping chemicals and like substances. Oct. 5.

22,779. W. Dalton. Improved scientific sanitary filter. Oct. 5.

22,816. J. V. Skoglund. Improvements in nitric acid condensers. Oct. 5.

22,902. J. Kippax. An improved apparatus for injecting anti-erustating fluids. Oct. 6.

22,921. W. Whittaker. Improvements in and relating to mechanical stokers. Oct. 6.

22,940. W. Defries. Improvements in and relating to destructor furnaces. Oct. 6.

22,958. L. H. von Gehlen. Improvements in and relating to air compressors. Complete Specification. Oct. 6.

22,965. D. Cameron, F. J. Commin, and A. J. Martin. Improvements in apparatus for the treatment of sewage or other liquids. Oct. 6.

22,969. A. Brauel. Improvements in apparatus for pasteurizing liquids. Oct. 6.

23,054. H. Higgins. Improvements in or relating to the maintenance and regulation of heat in distillation, refining, chemical manufacturing processes, and the like. Oct. 7.

23,110. H. S. Maxim. Improvements in and relating to apparatus for superheating steam. Oct. 8.

23,178. A. J. Bell and P. A. G. Bell. Improvements in apparatus for the filtration of water, sewage, and other fluids. Oct. 9.

23,195. T. H. Cobley. *See* Class VII.

23,312. F. Arenas. Improvements in apparatus for indicating and recording when the temperature exceeds or falls below predetermined limits. Oct. 11.

23,339. P. R. Allen and J. B. Chadwick. Improvements in or relating to apparatus for evaporating or concentrating liquids. Oct. 11.

23,722. H. Doig. Improvements in refrigerating apparatus. Oct. 14.

23,724. J. B. Hilliard. Improvements in apparatus and process for treating spirits and other fluids. Oct 15.

COMPLETE SPECIFICATIONS ACCEPTED.*

1896.

23,792. R. Thiel. Closing device for receptacles for sterilising. Oct. 20.

25,612. C. G. J. Moller. Apparatus for drying granular and other material. Oct. 13.

25,842. J. G. Chamberlain and J. Weddell. Evaporative surface condenser. Oct. 6.

26,112. J. Critchlow and T. Shore. Filter presses. Oct. 6.

26,441. F. C. Weedon. Means for determining the expansion of solids. Oct. 20.

26,937. J. W. Hall. Regenerative furnaces. Oct. 6.

27,494. E. Riepe and G. Oppermann. Regenerative furnaces. Oct. 13.

28,893. G. Nayler and A. Nayler. Fire retort. Oct. 20.

* *See* Note (*) on previous page.

1897.

18,189. O. Guttmann. *See* Class VII.

II.—FUEL, GAS, AND LIGHT.

APPLICATIONS.

21,508. R. P. Pictet. *See* Class VII.

21,602. P. Cannell-Bunn. Improved mantles for gas and other burners. Sept. 21.

21,608. H. Walker. *See* Class VII.

21,665. F. L. Bill. Improvements in apparatus in connection with the manufacture of gases and other purposes. Sept. 22.

21,681. J. McConechy. Improvements in apparatus for the production of acetylene gas. Sept. 22.

21,747. L. Dufrasne. Improvements in the manufacture of fuel. Sept. 22.

21,799. H. Maxim. Improvements in methods and apparatus for manufacturing and utilising gas, utilising waste coal, and ventilating coal mines. Sept. 23.

21,831. H. H. Lake.—From Wizard Manufacturing Company, United States. Improvements relating to the production of acetylene gas, and to lamps for generating and burning the same. Complete Specification. Sept. 23.

21,833. F. S. D. Scott and T. Hawkins. Improvements in and relating to the generation of gas for motive power, illuminating, heating, and other purposes. Sept. 23.

21,897. R. Douglas. A new or improved acetylene gas generator, more particularly intended for use in connection with lamps for velocipedes and the like. Sept. 24.

22,120. G. Hilgenstock. Improvements in coke ovens. Complete Specification. Sept. 27.

22,327. A. F. B. Gomess. Improvements in or relating to mantles for use in incandescent gas lighting. Sept. 29.

22,518. G. R. Hislop. Improvements in gas producers. Oct. 1.

22,601. W. Knowles. Artificial coal. Oct. 2.

22,625. W. L. Voelker. Processes for manufacturing incandescible materials. Complete Specification. Oct. 2.

22,626. W. L. Voelker. Improvements in incandescible materials and mantles and the process of making the same. Complete Specification. Oct. 2.

22,648. J. A. Ageron and L. P. Wirth. Improvements in and connected with apparatus for the generation and combustion of acetylene gas. Oct. 2.

22,730. A. Flöck. Improved apparatus for producing acetylene gas. Complete Specification. Oct. 4.

22,755. E. A. Prost. Improvements in or relating to apparatus for the production of acetylene gas. Oct. 4.

22,850. W. H. Wheatley.—From C. W. Beck, United States. Improvements in lamps for generating and burning acetylene gas. Oct. 5.

22,918. C. J. Bailey and J. H. Nicklin. Improved apparatus for the generation of acetylene gas. Oct. 6.

22,929. J. Whitehead and A. Slater. Improvements in automatic apparatus for the generation and storage of acetylene gas. Oct. 6.

23,198. F. Bessbardt.—From A. Moler, Argentine Republic. An automatic apparatus entitled "spirit" for the production of acetylene gas. Complete Specification. Oct. 9.

23,237. O. Imray.—From E. Pilous and A. M. Michaelis, Austria. Process for enriching gas of low illuminating power. Oct. 9.

23,273. W. Ackroyd. Improvements in apparatus for generating, delivering, and storing acetylene gas. Oct. 11.

23,287. E. Skriwan. Improvements in and connected with mantles for incandescent lights. Oct. 11.

23,351. O. V. Sigurdsson. Improvements in apparatus employed in the manufacture of acetylene gas. Oct. 11.

23,411. J. Riley and W. Lawson. Improvements in oxygen generaters. Oct. 12.

23,450. J. E. Speller. Improvements in apparatus for generating acetylene gas. Oct. 12.

23,458. M. Wagner and A. Pfemmert. Improvements in apparatus for the production of acetylene gas. Oct. 12.

23,543. T. Edmundson and F. Entwisle. Improvements in apparatus for the generation of acetylene gas. Oct. 13.

23,545. W. Schenk. Improvements in the manufacture of mantles for incandescent lights. Oct. 13.

23,547. A. Wartenweiler and R. Spengler. Improvements in automatic apparatus for the production of acetylene gas. Oct. 13.

23,793. C. J. Lundström. Improvements in the preservation of carbide and its subsequent treatment for use in the production of gas. Complete Specification. Oct. 15.

23,802. L. S. Buffington. Improvements in acetylene gas generators. Complete Specification. Oct. 15.

23,887. B. J. Atterbury. *See* Class XVII.

COMPLETE SPECIFICATIONS ACCEPTED.

1896.

19,677. P. R. de F. d'Humy. Manufacture of fuel briquettes. Oct. 13.

25,221. I. N. V. Bablon. Production of acetylene gas and apparatus therefor. Oct. 13.

25,762. M. Arndt. Gas-absorbing apparatus for ascertaining the quantity of a particular gas in a gaseous mixture. Sept. 29.

26,041. C. C. Connor. Electrodes. Sept. 29.

26,618. J. F. Duke. Manufacture of mantles or incandescing bodies for incandescent gas lamps. Sept. 29.

26,897. J. Vaughan-Sherrin. Apparatus for the production and utilisation of acetylene gas. Oct. 6.

27,574. E. Gossart and H. Chevalier. Means for regulating the production of acetylene gas, and lamps therefor. Oct. 20.

28,279. Sir C. S. Forbes, Bart. Apparatus for automatically regulating the supply of liquid fuel. Oct. 13.

28,290. F. V. C. Brokk. Vaporiser for liquid combustibles for incandescent lighting or heating purposes. Oct. 13.

28,853. W. P. Thompson.—From The Continental-Gas Gluhlicht-Actien-Gesellschaft "Meteor" vormals Kroll Berger and Co. Oil burners applicable for incandescent lighting. Oct. 20.

29,188. W. P. Thompson.—From L. L. H. Gerard. Apparatus for producing acetylene gas. Oct. 6.

29,320. P. de Plesener and H. Luchaire. Apparatus for the manufacture of acetylene. Oct. 6.

29,596. J. O. O'Brien.—From P. P. H. Mace. Feed governors for apparatus for producing acetylene. Oct. 6.

30,037. P. de Plesener. Apparatus for manufacturing acetylene. Sept. 29.

1897.

262. W. M. Ward. *See* Class XVIII.

9762. J. G. A. Kitchen. Burners for acetylene. Sept. 29.

15,261. F. Alomann. Apparatus for generating a vaporous or gaseous mixture from hydrocarbon containing liquid and water for the production of a blue flame. Oct. 6.

15,369. E. F. J. C. Bauweraerts. Carburetting of illuminating gas and apparatus therefor. Oct. 6.

17,927. M. Bernstein. Petroleum blue flame burners for incandescent lighting and heating purposes. Oct. 6.

17,938. A. Rieffel. Apparatus for the production of acetylene gas. Oct. 20.

19,149. W. E. Koehs. Coke ovens and machinery in connection therewith. Oct. 13.

19,823. T. Thorp and T. G. Marsh. Apparatus for the production of acetylene from calcium carbide and water. Oct. 6.

20,052. F. Rhind. Gas generating lamps. Oct. 13.

20,225. L. Freudenthal. Improved solution or fluid for impregnating incandescent bodies. Oct. 6.

20,571. O. Kahns. Process and apparatus for the production of carburetted air for incandescent gas light. Oct. 13.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, ETC.

APPLICATION.

22,716. A. Adiassewich. Treatment of petroleum or bitumen for production of pitch, aromatic hydrocarbons, and antiseptics therefrom, and apparatus for that purpose. Oct. 4.

COMPLETE SPECIFICATIONS ACCEPTED.

1896.

26,771. G. Schultz. Obtaining benzol and similar aromatic hydrocarbons. Sept. 29.

27,527. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier. Manufacture of preparations of guaiacol, creosote, and other higher phenols. Oct. 20.

1897.

5698. A. Basch and H. Graepel. Carbonising, charring, or coking combustible substances. Sept. 29.

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

21,553. A. G. Green and A. R. Wahl. Improvements in the production of colouring-matters for cotton. Sept. 20.

21,825. A. G. Green, A. R. Wahl, and The Clayton Aniline Company, Ltd. The manufacture or production of paranitrobenzaldehyde ortho-sulphonic acid and colouring matters therefrom. Sept. 23.

21,968. I. Levinstein and Levinstein, Ltd. Manufacture or production of aromatic aldehydes and of colouring matters therefrom. Sept 25.

21,991. J. Möller. Ink. Complete Specification. Sept. 25.

22,121. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier, France. Improvements in the production of aromatic aldehydes. Sept. 27.

22,242. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of blue basic safranine azo-dyestuffs. Sept. 28.

22,677. A. Ashworth and J. Bürger. Improvements in the production of colouring matters. Complete Specification. Oct. 4.

23,047. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of chemical compounds of the aromatic series. Oct. 7.

23,248. A. G. Green and A. R. Wahl. Improvements in the manufacture and production of colouring matters. Oct. 9.

23,370. C. D. Abel.—From Chemische Fabrik Rhenania, Germany. Manufacture of para-acetamidophenoxyl-acetamide mono- and di-alkylparaacetamidophen-oxylacetamides and chloral derivatives. Oct. 11.

23,816. A. Ashworth and J. Bürger. Improvements in the production of colouring matters. Complete Specification. Oct. 16.

COMPLETE SPECIFICATIONS ACCEPTED.

1896.

21,198. L. P. Marchlewski, E. S. Wilson, and E. Stewart. Production of dyes. Sept. 29.

25,592. C. W. Anderson, H. Benjamin, and J. Mendess. Liquid compound for dyeing purposes, also applicable for detergent, purifying, and disinfecting purposes. Oct. 13.

26,020. Read Holliday and Sons, Ltd., J. Turner, and H. Dean. Production of sulpho acids and colouring matters therefrom. Oct. 20.

26,139. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Production of chemical compounds of the aromatic series. Sept. 29.

26,210. W. H. Claus, A. Ree, and L. Marchlewski. Production of new azo-colouring matters. Oct. 6.

26,666. A. Zimmermann. — From The Chemische Fabrik auf Actien vormals E. Schering. Manufacture of isomeric, unsymmetrical acetonalkamines, and acidyl derivatives therefrom. Oct. 20.

27,896. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of basic dis-azo dyestuffs. Oct. 13.

28,199. A. G. Green. Production of paranitraniline red. Oct. 13.

29,717. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier. Manufacture of chlorine derivatives of toluene. Oct. 20.

1897.

6581. W. Majert. Method of producing and purifying orthotoluol-sulpho-chloride. Oct. 6.

18,762. A. Ashworth. Manufacture of colouring matters suitable for dyeing unmordanted cotton. Sept. 29.

V.—TEXTILES, COTTON, WOOL, SILK, Etc.

Applications.

21,632. G. C. Dymond.—From Storm and Braakenberg, Holland. Improved process and composition for rendering cloth, cellulose, paper, and the like, fireproof Sept. 21.

21,719. M. Rivizza and R. Schönlin.—From J. R. Bautz, Germany. An improved process for impregnating woollen fabrics intended for keeping the human body at an equal temperature. Sept. 22.

21,801. W. G. Thomson. Improvements in the method of and apparatus for manufacturing inlaid linoleum and other floorcloths. Sept. 23.

21,983. J. Newell. Improvements in the method of and apparatus for singeing, gassing, or genapping yarn. Sept. 25.

22,351. E. Remy. Improvements relating to the washing and like treatment of printed fabrics, and to apparatus therefor. Sept. 29.

22,477. F. Holtschmidt. Improvements in and relating to the manufacture of fabrics. Complete Specification. Sept. 30.

23,670. W. P. Ingham. Improvements in or connected with the manufacture of slag wool. Oct. 14.

Complete Specifications Accepted.

1896.

21,557. R. B. Hardman. Apparatus for cleaning fibres from grease and impurities. Sept. 29.

22,197. W. C. Kipling. Waterproofing silk, wool, or other fabrics. Oct. 6.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

Applications.

22,295. J. Heartwell. Improvements in and connected with bleaching and similar kiers. Sept. 29.

22,450. E. Cabiati. An improved process of bleaching fabrics. Sept. 30.

22,551. W. Lowe. Improvements in printing textile fabrics with basic dyes or aniline black ground. Oct. 1.

22,786. P. Reid and H. Thorp. Improvements in dyeing textile fibres, yarns, and fabrics. Oct. 5.

23,483. T. Trusher. Improvements in apparatus for treating fibrous materials with dye or other liquors. Oct. 12.

23,484. T. Trusher. Improvements in apparatus for treating fibrous materials with dye or other liquors. Oct. 12.

23,485. T. Trusher. Improvements in apparatus for treating fibrous materials with dye or other liquors. Oct. 12.

23,644. A. J. Craig. Improvements in apparatus for scouring, dyeing, and washing woollen, cotton, or other textile materials and fabrics. Oct. 14.

23,688. J. Brandwood. A new or improved machine or apparatus for boiling, bleaching, mordanting, dyeing, or drying vegetable or animal fibres in the raw state, in the hank or in the cop, or on the bobbin or cheese, or other compact forms. Oct. 14.

23,689. J. Brandwood, J. Taylor, and W. Ogden. A new or improved apparatus or appliance for dyeing cops on cop-dyeing machines without spindles or skewers. Oct. 14.

Complete Specifications Accepted.

1896.

27,742. H. W. Kearns and J. Barnes. Production of fast colours upon vegetable fibre. Oct. 13.

28,266. A. F. B. Gomess. Bleaching textile vegetable fibres, yarns, and fabrics. Oct. 20.

VII.—ACIDS, ALKALIS, and SALTS.

Applications.

21,508. R. P. Pictet. Improvements in the process and apparatus for the production of carbide of calcium. Date applied for Feb. 20, 1897, being date of application in France. Sept. 20.

21,608. H. Walker. Improvements in the method of and apparatus for the manufacture of calcium carbide. Sept. 21.

21,910. The Aluminium Company, Ltd., and A. G. Haddock. Improvements in bleaching compounds. Sept. 24.

22,136. F. Lutze. An improved process for the preparation of carbolic acid. Sept. 27.

22,395. W. Garroway. Improvements in the manufacture of lead carbonate, ammonium nitrate, silicates of sodium, and caustic soda. Sept. 30.

22,790. I. Levinstein and Levinstein, Ltd. Improvements in and connected with the concentration of sulphuric acid, and with apparatus therefor. Oct. 5.

23,053. H. Higgins. Improvements in or relating to the evaporation of saline and like solutions, and the desiccation of the product thereof. Oct. 7.

23,163. C. P. F. Desprèz and E. A. Duthuit. Improvements in the manufacture of blocks of calcium carbide. Date applied for March 8, 1897, being date of application in France. Oct. 8.

23,195. T. H. Cobley. Improvements in the production of colloidal tannates for use in land and marine steam boilers. Oct. 9.

Complete Specifications Accepted.

1896.

22,717. G. Kynoch and Co., Ltd., and A. T. Cocking. Method and apparatus for mixing and conveying acids for use in nitroglycerin and other manufactures. Sept. 29.

27,769. E. Dyson. Apparatus for concentrating sulphuric and other acids. Oct. 6.

29,922. J. D. Gilmour. Drying alkaline cyanides. Oct. 6.

1897.

17,834. E. F. Turner. Treatment of metallic chlorides, such as those produced from sulphide ores. Sept. 29.

18,189. O. Guttmann. Apparatus for converting nitric peroxide into nitric acid. Sept. 29.

VIII.—GLASS, POTTERY, and ENAMELS.

Applications.

22,033. T. Krah. Improved apparatus for pressing and moulding glass and the like. Complete Specification. Sept. 25.

23,080. W. S. Hadley, F. W. Sephton, and R. Mills. Improvements in the method of and means for producing indelible designs upon the surface of glass, marble, metal, and similar material. Oct. 8.

Complete Specifications Accepted.

1896.

25,665. J. Duckett and Son, Ltd., J. Duckett, and J. W. Bullock. Construction of muffled and semi-muffled kilns for burning biscuit and glazed ware. Oct. 13.

1897.

19,003. H. Brooke and J. C. Grout. Apparatus for the manufacture of glass. Oct. 6.

19,493. C. C. Hartung. Making plate glass. Oct. 6.

20,388. J. B. Vernay. Automatic machine for the manufacture of bottles and other articles of glass. Oct. 6.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, and CEMENTS.

Applications.

21,921. C. W. Luther. A new way of constructing light fireproof building material possessed of the quality not to be eaten by insects, and therefore specially adapted for hot climates. Complete Specification. Sept. 24.

22,254. E. M. Fox. An improved compound or solution for the treatment of wood or other substances to render them uninflammable. Sept. 28.

22,271. K. Kunze. Improvements in the manufacture of artificial stone. Sept. 28.

23,515. W. Smith. Improvements in the manufacture of plaster. Oct. 13.

Complete Specifications Accepted.

1896.

14,673. F. D. T. Lehmann and P. N. Kohlsaat. Kilns for burning bricks, tiles, lime, and the like. Oct. 6.

21,781. A. C. Ponton. Manufacture of fire-bricks and fireproof mortar. Oct. 6.

24,711. R. Hunter. Manufacture of finely pulverised substances for use in the production of Portland cement, pigments, and the like. Sept. 29.

27,648. A. Taylor. Manufacture of artificial stone for building, paving, and other suitable purposes. Oct. 6.

1897.

1795. La Société Metallurgique de Champignelles et Neuves-Maisons. Manufacture of an improved cement. Oct. 20.

X.—METALLURGY, MINING, Etc.

Applications.

21,552. E. C. F. Otto. A new or improved process for case-hardening iron or mild steel partially applicable to hardening steel. Sept. 20.

21,736. R. W. E. Maelvor. Improvements in the treatment of gold ores and slimes and apparatus therefor. Sept. 22.

21,963. W. J. H. Jones and C. H. Moon. Improvements in the annealing of metals drawn into wire. Sept. 25.

21,984. G. Thomson. Improvements in alloys or metallic compounds containing steel. Sept. 25.

22,256. The General Gold Extracting Company, Ltd.—From L. Peletau, France. Improvements in and in apparatus for the treatment of ores or the like containing gold or silver, or both gold and silver for the obtainment of the precious metal therefrom. Sept. 28.

22,666. C. Jones. Improvements in the construction of furnaces for heating baths or pots of metal for tin plating lead coating, galvanising, or the like. Oct. 4.

22,815. R. Clarke and C. Flower. A new and improved white metal or alloy and the process of producing the same. Oct. 5.

22,938. H. Maxim. An improved method of and apparatus for applying intense heat to the surfaces of metallic and other bodies, and also for the complete fusion of bodies. Oct. 6.

22,961. G. Weil and A. Lévy. Improved processes for coating aluminium or other metals with silver or other metals. Oct. 6.

23,177. W. Allen and A. H. Plante. A solder for uniting aluminium. Oct. 9.

23,267. J. E. Bryant. Improvements in apparatus for the electro-deposition of one metal on another. Oct. 11.

23,278. J. Bedford and J. Ashton. Improvements in the production of compound steel bars for making cutting tools and the like. Oct. 11.

23,335. F. J. Rutter. Improved means applicable for use in obtaining metals from sulphide ores. Oct. 11.

23,883. R. B. Thomas and H. S. Thomas. Improvements in the treatment of iron or steel plates or sheets to be afterwards coated with tin or terne metal or other coating metal or alloy or to be otherwise coated or used uncoated and in apparatus employed in the said treatment. Oct. 16.

Complete Specifications Accepted.

1896.

21,976. A. L. Le Redotte. Manufacture of certain metals and alloys, especially magnesium and its alloys. Oct. 13.

22,791. T. Teague. Bar tin or metal casting machine. Oct. 6.

24,705. R. A. Hadfield. Manufacture or production of metallic alloys. Oct. 6.

25,462. W. P. Thompson.—From La Société Civile d'Etudes du Syndicat de l'Acier Gerard. Pulverisation of metals with a view to the manufacture of steel and for other purposes. Oct. 6.

25,611. H. Maxim. Method and apparatus for manufacturing calcium carbide and for the reduction of metals from their oxides. Oct. 20.

28,727. P. E. Placet. Purification of aluminium and apparatus therefor. Oct. 13.

29,846. S. Danner. Manufacture of crucible steel. Oct. 6.

1897.

6991. H. Griffith, jun.—From V. Coppee and A. E. Kemplen. Improvements relating to aluminium. Oct. 20.

6993. H. Griffith, jun.—From V. Coppee and A. E. Kemplen. The brazing of alumium. Oct. 20.

14,325. C. C. E. Bohne. Treatment of slag resulting from the smelting of tin ore. Sept. 29.

XI.—ELECTRO-CHEMISTRY and ELECTRO-METALLURGY.

Applications.

21,657. W. Decker and G. von Struve. Improvements in and relating to electrolytes for secondary batteries. Complete Specification. Sept. 21.

21,704. S. W. Hart. Improvements in secondary electric batteries or accumulators. Sept. 22.

21,705. S. W. Hart. Improvements in and relating to secondary electric batteries or accumulators. Sept. 22.

21,819. E. J. Wade. Improvements in and connected with electrical storage batteries. Sept. 23.

21,976. F. Greenfield. Improvements in appliances used in connection with the electro-deposition of metals. Sept. 25.

22,044. R. W. James.—From The Bell Electric Company, United States. Improvements in electric batteries, electrodes, and method of making the same. Complete Specification. Sept. 25.

22,045. R. W. James.—From The Bell Electric Company, United States. Improvements in electric batteries. Complete Specification. Sept. 25.

22,203. A. J. Boult.—From F. H. Soden, United States. Process of and apparatus for electrically treating ore. Complete Specification. Sept. 28.

22,549. G. F. W. Morris.—From S. Bash, Argentine Republic. Improvements in single liquid voltaic batteries. Oct. 1.

23,329. G. Platner. Improvements in and connected with substances to prevent polarisation in electric batteries. Complete Specification. Oct. 11.

23,332. E. A. Paris. Improvements in secondary battery plates or electrodes. Oct. 11.

23,352. A. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Schering, Germany. Improvements in the manufacture of electrodes for electrolytic purposes. Oct. 11.

23,489. C. P. Elieson. Improvements in electric storage cells. Oct. 12.

23,602. E. J. J. B. Benoit. Improvements in anodes of electro-decomposing apparatus. Oct. 13.

Complete Specifications Accepted.

1896.

21,273. J. E. Liardet. Electric storage batteries. Sept. 29.

28,139. A. Dodd. Lead accumulators. Oct. 20.

1897.

996. W. Majert. Accumulator plates. Sept. 29.

18,628. C. H. Cole. Portable electric primary batteries. Sept. 29.

19,035. H. S. Jones, of the firm of W. P. Thompson and Co.—From E. Balbach, jun., United States. Electrolytic apparatus. Oct. 20.

XII.—FATS, OILS, and SOAP.

Applications.

21,749. J. C. W. Stanley and T. B. C. Hardman. Improvements in or relating to the extraction of oil from cocoa and other nuts. Sept. 22.

21,832. F. S. D. Scott and T. Hawkins. An improved soap for toilet and other purposes. Sept. 23.

22,123. J. G. Haller. Improvements in the manufacture of glycerine from spent soap leys. Sept. 27.

22,140. E. T. F. Goodwin. Improvements in toilet and other soaps. Sept. 27.

22,810. R. Holliday and Sons, Ltd., and T. Holliday. Improvements in the manufacture of soap. Oct. 5.

22,862. F. Arledter. Improvements in the manufacture of resinous soap. Oct. 5.

23,140. T. Arthur. A solution for washing and cleansing glass and painted and varnished surfaces. Complete Specification. Oct. 8.

23,197. J. Monterrubio. An improved system for manufacturing soaps by the direct employment of oleaginous seeds. Complete Specification. Oct. 9.

23,612. H. J. Gardiner. An improved detergent compound for woollen goods and the like. Oct. 13.

23,700. J. Mendess. Improvements in compounds for cleansing and dyeing purposes. Oct. 14.

Complete Specifications Accepted.

1896.

26,007. W. R. Harrison and E. Stephenson. Apparatus for brightening and clarifying crude oils. Oct. 13.

1897.

19,429. F. Arledter. Manufacture of resinous soap, and apparatus therefor. Oct. 13.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

21,718. R. Strobentz and J. Fried. Process for manufacturing black pigments from carbonaceous materials. Complete Specification. Sept. 22.

22,514. W. Ellis. The combination of india-rubber with other chemical substances, which impart to the product a quality useful in certain manufactures. Oct. 1.

22,792. L. Hunt. A new or improved non-poisonous and permanent white paint or varnish. Oct. 5.

22,808. H. Cotton. A lacquer to be applied for the prevention of atmospheric oxidisation of gold leaf, silver leaf, Dutch metal leaves, and the like, as used by decorators for interior decorations. Oct. 5.

22,867. H. L. Bates. An improved paint. Complete Specification. Oct. 5.

Complete Specifications Accepted.

1896.

23,294. The Non-Corrosive Preserving and Fire-Resisting Paint Co., Ltd., F. White, and J. Thomson. Non-corrosive preserving and fire-resisting paint. Oct. 20.

24,711. R. Hunter. *See* Class IX. Sept. 29.

25,779. H. Helbing and G. Pertsch. Preparation and application of coating and insulating materials for medical and other purposes. Oct. 13.

26,322. J. Fairlie. Manufacture of red lead. Oct. 6.

26,695. F. H. Smith and C. Macintosh and Co., Ltd. Manufacture of rubber compounds. Oct. 13.

1897.

16,490. J. Stocker and H. Zander. Improved weather acid and fire-resistant material. Oct. 6.

19,030. A. H. Eyles. Apparatus for use in the manufacture of white lead. Oct. 6.

19,736. J. E. Bedford and C. S. Bedford. Manufacture of varnish. Oct. 13.

XIV.—TANNING, LEATHER, GLUE, and SIZE.

Applications.

21,601. E. H. Dewson. Improvements in the tanning of hides and skins and the like, for the production of leather and in the apparatus employed therein. Complete Specification. Sept. 21.

22,361. G. Levinstein. Improvements in and relating to the tanning of hides and skins. Sept. 29.

23,194. T. H. Cobley. Improvements in the manufacture of tannin preparations, concentrating and blending same for tanning and dyeing and other purposes. Oct. 9.

Complete Specifications Accepted.

1897.

13,997. W. Turner. Apparatus for working or treating skins, hides, or leather. Oct. 20.

18,332. E. J. Thibaut. Means to be used in tanning hides and skins. Oct. 13.

19,661. H. Bolder. Liquid casein glue. Oct. 20.

19,874. E. Du Bois. Apparatus for tanning. Oct 6.

20,354. E. J. Thibaut. Means to be used in the tanning of hides and skins. Oct. 13.

XVI.—SUGARS, STARCHES, GUMS, Etc.

Applications.

21,533. B. J. B. Mills.—From E. Leconte, France. Improvements in the manufacture of dextrine and applications of the same. Sept. 20.

22,532. G. S. Baker and W. K. Baker. Improvements in or relating to the manufacture of sugar, glucose, and the like, and apparatus therefor. Oct. 1.

23,875. H. Higgins. Improvements in or relating to the manufacture of lactose or sugar of milk. Oct. 16.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

21,878. E. Zdarek. Improvements in the manufacture of aethyl-alcohol. Complete Specification. Sept. 24.

22,481. E. de Meulemeester. A process for treating maize and other cereals, for use in the manufacture of beer and alimentary substances, and in distilling. Sept. 30.

22,617. R. D. Bailey. Improved processes of producing brewers' and distillers' wort for fermentation. Oct. 2.

22,846. A. D. Watson. An improved process for the extraction of the Plasma (cell contents) and the separation of cellulose from yeast. Oct. 5.

23,025. B. J. Atterbury. Improvement in the production of ethyllic alcohol. Oct. 7.

23,249. J. D. Möller. An improved method of improving or oxidizing alcoholic beverages. Complete Specification. Oct. 9.

23,341. M. L. Wheatley. Improvements in or relating to the preservation of brewers' grains and other material. Oct. 11.

23,724. J. B. Hilliard. *See* Class I.

23,887. B. J. Atterbury. Improvements in the production of alcohols and bye products, and in apparatus therefor, and for the production of acetylene gas. Oct. 16.

XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

Applications.

A.—Foods.

21,995 J. A. L. F. Janasz. An improved treatment of the milk of cows or other domesticated animals to render it suitable as food for infants and children. Sept. 25.

22,112. H. A. Lacroix and A. C. de Caudemberg. Process of preserving organic compounds. Sept. 27.

22,686. L. Wacker. Process and apparatus for preserving meat. Oct. 4.

22,838. J. Mecredy. Improvements in and connected with the desiccation of eggs and in apparatus therefor. Oct 5.

22,839. J. Mecredy. Improvements in the desiccation of articles of food and in means or apparatus therefor. Oct. 5.

22,840. J. Mecredy. Improvements in and relating to the desiccation of articles of food. Oct. 5.

23,149. W. J. Engledue. Improvements in or relating to the preservation of milk and milk products. Oct. 8.

B.—Sanitation.

21,982. W. Watson. Improvements in the method of and apparatus for purifying and softening or similarly treating sewage and other impure water. Sept. 25.

22,128. C. R. Bilange. Improvements in the treatment and utilisation of excreta and other refuse and in apparatus therefor. Sept. 27.

22,965. D. Cameron, F. J. Commin, and A. J. Martin. *See* Class I.

23,107. J. Grossmann. Improved method of treating sewage. Oct. 8.

23,428. W. H. L. Galloway.—From J. E. Jordan, South African Republic. Improvements in or relating to machinery or apparatus for treating, softening, and purifying water, sewage, slimes, or other liquids or matters held in solution or suspension. Oct. 12.

23,604. A. Zimmermann.—From H. Krell, Germany. An improved process for disinfecting and in apparatus therefor. Oct. 13.

23,787. C. J. Whittaker and W. C. Bryant. Improvements in the treatment of sewage and in apparatus therefor. Oct. 15.

C.—Disinfectants.

22,716. A. Adiassewich. *See* Class III.

COMPLETE SPECIFICATIONS ACCEPTED.

A.—Foods.

1896.

14,209. R. G. Nash. Aëration and preservation of milk and apparatus therefor. Sept. 29.

24,587. J. Garton, M. E. G. Finch-Hatton, and R. Garton. Treatment of grain and the manufacture of food-stuffs therefrom. Oct. 13.

1897.

899. J. J. Hazard. Preservation of organic matters and apparatus for use in connection therewith. Sept. 29.

15,884. L. Michon and M. E. Goulet. Preserving alimentary products. Oct. 13.

19,446. H. Grous and H. Pateson. Chilling and freezing meat and other alimentary substances and means therefor. Sept. 29.

B.—Sanitation.

1896.

22,768. C. Fell. Filter for sewage effluent and the like. Oct. 20.

26,667. A. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Schering. Disinfecting and apparatus therefor. Oct. 6.

29,009. J. F. G. de Bonardi (Marquis du Menil). Method and furnace for the incineration of so-called garbage and the recovery of ammonia therefrom. Oct. 20.

1897.

262. W. M. Ward. Mode, means, and apparatus for treating smoke for the extraction of the carbon and other residual products resulting from imperfect combustion. Oct. 6.

19,426. J. Desmaroux. Apparatus for sterilising water. Oct. 13

C.—Disinfectants.

1896.

25,592. C. W. Anderson. H. Benjamin and J. Mendess. *See* Class IV. Oct. 13.

XIX.—PAPER, PASTEBOARD, Etc.

APPLICATIONS.

22,630. E. Sonstadt and J. M. Landon. Improvements in the treatment or manufacture of paper. Oct 2.

22,859. C. Endruweit. An improved process for the metallic coating of paper. Complete Specification. Oct. 5.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

APPLICATIONS.

23,004. J. Y. Johnson.—From C. F. Boehringer and Soehne, Germany. Improvements in the manufacture or preparation of adenin. Oct 7.

23,005. J. Y. Johnson.—From C. F. Boehringer and Soehne, Germany. Improvements in the manufacture or preparation of hetrocroxanthine, paraxanthine, and methylated hypoxanthines. Oct 7.

23,227. J. Y. Johnson.—From C. F. Boehringer and Soehne, Germany. Improvements in the production of oxypurines and their alkyl derivatives from trichloropurine. Oct. 9.

23,355. P. E. M. Jamain. Improvements in apparatus for compressing pharmaceutical and other powders for the manufacture of tabloids. Date applied for Aug. 9, 1897, being date of application in France. Oct. 11.

23,584. M. Otto. Improvements in the production of iodoform. Oct 13.

23,719. E. de Laire. Manufacture of a new product from ionone or pseudo-ionone. Oct 14.

COMPLETE SPECIFICATIONS ACCEPTED.

1896.

24,799. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Production of a pharmaceutical compound. Sept. 29.

25,905. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier. Manufacture of a new salt of piperazine. Oct. 6.

26,350. A. J. Boult.—From F. Fritzsche and Co. Manufacture of artificial essence or essential oil of violets. Oct. 13.

27,655. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier. Manufacture of benzoic sulphinide. Oct. 13.

27,656. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier. Production of formic aldehyde vapours. Oct 13.

XXI.—PHOTOGRAPHY.

APPLICATIONS.

21,679. G. A. de Katow.—From A. Schwarz, Germany. Improvements in method of and apparatus for toning photographs. Complete Specification. Sept. 22.

23,739. H. W. Cottrell. An improved method of producing coloured photographs. Oct. 15.

30,161. M. Jolles and L. Lilienfeld. Improved photographic printing paper or the like. Oct. 13.

XXII.—EXPLOSIVES, MATCHES, Etc.

APPLICATIONS.

23,340. G. Beneké. Improvements in the manufacture of explosive compounds. Oct. 11.

23,353. G. A. Witt.—From P. Cornet, Belgium. Improvements in explosives. Oct. 11.

COMPLETE SPECIFICATIONS ACCEPTED.

1896.

22,717. G. Kynoch and Co., Ltd., and A. T. Cocking. *See* Class VII. Sept. 29.

26,253. Vickers, Sons, and Co., Ltd., A. F. Dawson, and G. T. Buckham. Primers for ordnance. Oct. 6.

28,889. G. Kynoch and Co., Ltd., and A. T. Cocking. Explosives. Oct. 20.

XXIII.—ANALYTICAL CHEMISTRY.

APPLICATION.

23,321. F. Hugershoff. Improvements in or relating to centrifugal machines for examining and testing milk, sediments of urine, and other fluid substances. Complete Specification. Oct. 11.

PATENTS UNCLASSIFIABLE.

APPLICATIONS.

23,126. A. J. Boult.—From A. O. Richter, Germany. Improved compound for use in extinguishing fires. Complete Specification. Oct. 8.

23,229. D. G. Dow. Improvements in and relating to fire-resisting material. Oct. 9.

23,330. T. Oddy and C. Heap. Improvements in and apparatus for the manufacture of certain carbonaceous compounds. Oct. 11.

23,874. E. Heusch. Improved manufacture of artificial mother-of-pearl. Oct. 16.

COMPLETE SPECIFICATION ACCEPTED.

1897.

17,610. J. Carnick. Preparing protonuclien. Oct. 6.

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In reply to numerous inquiries, the compilation of the Collective Index is in active progress, half of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

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B

Foreign and Colonial Members are reminded that the subscription of 25s. for 1898, payable on January 1st next, should be sent in good time to the Treasurer, in order to ensure continuity in the receipt of the Society's Journal. Any changes of address to appear in the new List of Members now in course of preparation, should reach the General Secretary not later than January 15th, 1898.

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SESSION 1897–98.

Dec. 6th.—H. L. Sulman and Dr. F. L. Teed. "The Sulman-Teed Process of Gold Extraction."

Meeting held on Monday, November 1st, 1897.

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THE ADULTERATION OF PORTLAND CEMENT.

BY W. HARRY STANGER, M. INST. C.E., AND BERTRAM BLOUNT, F.I.C.

(See also this Journal, 1894, 1236—1237.)

During the last few years, two notable tendencies have been apparent in the manufacture of Portland cement. The first, which is altogether admirable, is the tendency of many manufacturers to study the principles of manufacture more closely than heretofore, and, as a consequence, to turn out a product of ever improving quality. The second, which cannot be too strongly condemned, is the tendency of certain other manufacturers to mix with Portland cement foreign substances of the most diverse nature, having as a rule only one property in common, *viz.*, a price smaller than that of Portland cement, and to sell the product as Portland cement.

It is not our business, nor is it our present intention, to discuss matters of purely trade interest, but it is nevertheless necessary to say here at once that the former tendency is much the stronger, and that the number and importance of the makers who illustrate the second are relatively small. In a word, the Portland cement trade of this country, as far as it is known by us, is an honestly conducted industry, and the largest and best-known makers wish to keep it so. It is also but just to say that some makers who practise the addition of foreign substances to Portland cement, do so openly, and with the avowed belief that they improve the cement thereby; and although we hold them to be mistaken, we may accept the sincerity of their convictions. Others there be who adulterate grossly and secretly until detected and ignominiously exposed. It would be waste of time to discuss their merits.

On the 30th November 1894, we were requested by the London Chamber of Commerce to undertake the investigation of the effect on Portland cement of the admixture with it of various foreign substances, especially of Kentish ragstone. The immediate cause which made such an investigation necessary was that in the Thames and Medway district certain makers had adopted the practice of adding ragstone to Portland cement, and maintained that the cement was improved by this addition. We undertook the investigation, and at once initiated a series of experiments, which were designed to be as exhaustive as possible. The work involved was long and laborious, but we were greatly aided in its successful prosecution by many of the makers interested, who provided us with samples prepared according to our directions, and also carried out a large number of tests supplementary to our own, adopting a uniform method of procedure so as to facilitate comparison of the results. We are also indebted to the courtesy of a maker who considered that the use of ragstone was an improvement, and who allowed us free access to his works, and explained his method of operation with the utmost frankness and candour. Everyone concerned showed himself anxious above all things to get at the facts, whether he had a prepossession in favour of the use of ragstone or entertained an objection to its employment, and by this just and scientific attitude contributed in no small degree to the ultimate elucidation of the question.

The results of our labours, which occupied more than a year, were embodied in a series of reports to a Special Committee of the Cement Trade Section of the London Chamber of Commerce. Together with other evidence collected by that body, they formed the basis of a report by the Section, and of a resolution to the following effect:—

"That Portland cement be defined as a mixture of two or more suitable materials, intimately and artificially mixed in the requisite proportions, and afterwards properly calcined and ground, to which nothing has been added during or after calcination, excepting that an addition not exceeding 2 per cent. of gypsum is permissible for the purpose of regulating the setting."

So important did it appear to the members of the Section to secure Portland cement from adulteration that they have drawn up the following declaration, which all manufacturers of Portland cement in Great Britain and Ireland are to be invited to sign:—

"We, the undersigned, hereby agree to conform to and carry out the rule of the Cement Trade Section of the London Chamber of Commerce, as set forth in a report made by the Section, and adopted at a meeting held on Monday the 10th of May 1897":—

"That if any material whatever, excepting an amount not exceeding 2 per cent. of gypsum for the purpose of regulating the setting, be added to the Portland cement clinker, during or after calcination, the article so produced shall not be sold as Portland cement, but under some other distinctive name."

"And we further agree that if at any time any of the parties to this agreement shall—by resolution of a majority of those present at a meeting of such parties duly and properly convened in accordance with the practice of the London Chamber of Commerce, such resolution having been duly and properly confirmed by a majority of those present at a subsequent meeting called at not less than 14 days' notice—be found to have failed to conform to and carry out the said rule, then in such case his or their name or names shall be struck off the list, and notice of the same made public in such manner as shall be resolved."

There is thus no doubt as to the attitude of the most considerable part of the cement trade in this country, and with this attitude we agree thoroughly. The technical and scientific reasons which justify this attitude are, we venture to think, largely drawn from the result of our own investigations. With the express permission of the London Chamber of Commerce—to which body and to its courteous Secretary, Mr. Kenric B. Murray, we tender our fullest acknowledgments—we are now in a position to lay before the London Section of the Society of Chemical Industry some of the chief facts and deductions which have been the fruit of our investigation.

The facts about to be recorded give but a slight indication of the vast quantity of data which was accumulated in the course of this investigation. Even the series of reports to the London Chamber of Commerce, which were necessarily long and minute, represent only an excerpt of the work which had to be carried out. The results at our disposal have been submitted to a severe process of compression in order to bring them within the limits of a paper.

Outline of the Investigation.

On the question arising as to the effect of an addition of ragstone to Portland cement, it would appear fairly obvious,

—	"A."	"D."	"F."	"L."	"J."
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Silica (SiO_2)	0·45	0·60	0·08	0·60	0·61
Insoluble residue (sand and clay)	13·53	15·06	22·16	15·60	15·61
Alumina (Al_2O_3)	0·44	0·80	0·14	0·54	0·44
Ferric oxide (Fe_2O_3)	1·22	1·20	1·40	1·30	1·12
Lime (CaO)*	46·84	45·60	41·80	45·42	44·80
Magnesia (MgO)	0·36	0·50	0·44	0·61	0·38
Sulphuric anhydride (SO_3)†	Nil	0·30	0·16	0·18	0·30
Matter volatile at a red heat . { Carbonic anhydride (CO_2)‡	35·96	34·30	33·10	33·64	35·30
Matter volatile at a red heat . { Water (OH_2)	0·65	1·14	0·46	1·98	0·92
Alkalies { Potash (K_2O) / Soda (Na_2O) } and loss	0·55	0·50	..	0·13	0·52
Total	100·00	100·00	100·00	100·00	100·00
* Value after deduction of that necessary for the formation of calcium sulphate.	..	45·39	41·69	45·29	44·59
Corresponding with calcium carbonate ($CaCO_3$)	81·73	77·95	75·23	76·45	80·23
† Calculated as calcium sulphate	..	0·51	0·27	0·31	0·51
Specific gravity	2·720	2·680	2·680	2·690	2·685

on chemical grounds, that the ragstone would be an inert substance, and could be regarded merely as a diluent. Kentish ragstone is a natural form of carbonate of lime mixed with silicious matter. It varies somewhat in composition, as may be seen from the above analyses, which are calculated on the samples free from moisture. But although thus varying, its composition is always such that when the stone is powdered and mixed with water, it is in no way cementitious, and shows no tendency to set.

Nevertheless it was stated, by those that had made the experiment, that this inert, silicious limestone could be added to Portland cement in considerable quantity (10—20 per cent.) with positive advantage to the cement. It was credibly asserted that a cement thus diluted was actually stronger than one in its normal unmixed state.

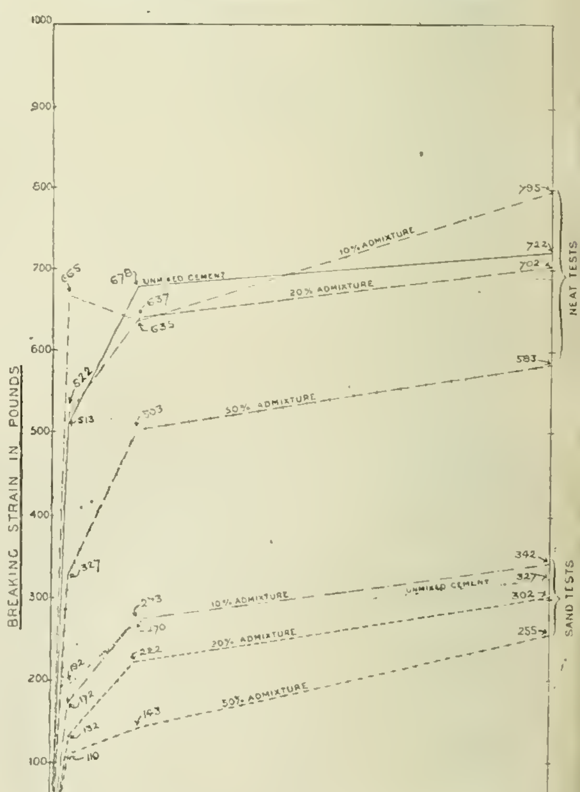
Rejecting both *a priori* views and the statements of other experimenters, we made our own tests. We may say here that in all cases our experiments have been made on cement and ragstone, ground together as in actual manufacture; separate grinding of the materials and subsequent admixture not only departs from the method practised by users of ragstone, but is likely to yield misleading results, because the intimacy of admixture is insufficiently great. Mixtures containing 10, 20, and 50 per cent. of ragstone were prepared in this manner, and tested at 7 and 28 days and at 6 months, neat and with sand, in tension and compression, and compared with corresponding tests made on the unmixed cement. The diagram below is typical of certain of the results obtained.

The composition of these samples is stated in the appended tables.

Many similar tests proved beyond doubt that some cements not only do not show a decrease in strength when mixed with 10 or 20 per cent. of ragstone, but are actually stronger when thus mixed. This is true whether the cement and mixtures are tested neat or with sand, in tension or in compression. Thus the upshot is in direct contradiction to what would certainly be expected by anyone endeavouring to solve the question on first principles. The result constitutes a useful example of the danger of making even what appear to be most reasonable assumptions, when it is open to one to dispense with assumptions altogether, and to substitute sound experimental data.

After close study, the reasons for this apparent anomaly were discerned. It became clear that when ragstone containing, as it commonly does, a small quantity of moisture, is mixed with cement clinker, and passed, together with that, through the stone-breaker and millstones or other grinding machinery, it is brought into the closest possible contact with the cement, and slakes any overlimed portions which

DIAGRAM OF TENSILE TESTS. SERIES "A."



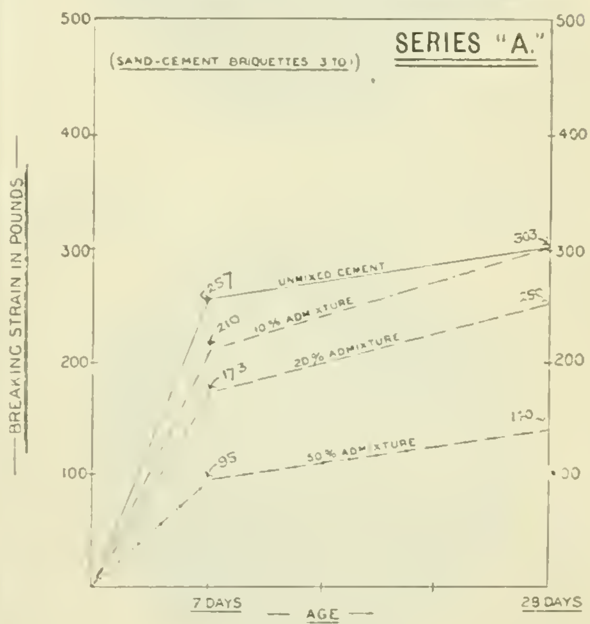
may be present. Thus, a cement containing ragstone as it comes from the mills is more slaked than one free from ragstone. Now it is known that cement which is not perfectly sound is improved by limited slaking; it is on account of this that air-slaking or aeration is insisted on

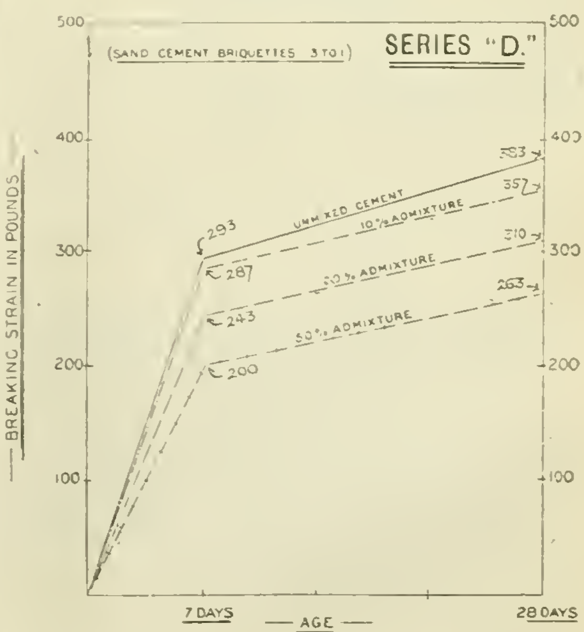
—	Unmixed Cement.	10 per Cent. Admixture.	20 per Cent. Admixture.	50 per Cent. Admixture.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Silica (SiO_2)............	20·32	18·36	15·54	10·98
Insoluble residue (sand and clay).	0·70	2·34	4·28	7·54
Alumina (Al_2O_3)........	8·36	7·16	6·22	4·72
Ferric oxide (Fe_2O_3)	4·34	4·18	3·80	2·80
Lime (CaO)...............	59·16*	58·72*	56·62*	53·10*
Magnesia (MgO)........	1·04	1·02	1·02	0·83
Sulphuric anhydride (SO_3).	1·37†	1·26†	1·11†	0·75†
Matter volatile at a red heat:—				
Carbonic anhydride (CO_2).	1·77	4·90	9·09	17·12
Water (OH_2)	1·81	1·64	1·81	1·34
Alkalis:—				
Potash (K_2O) } Soda (Na_2O) } and loss	0·83	0·42	0·51	0·52
	100·00	100·00	100·00	100·00
*Value after deduction of that necessary for the formation of calcium sulphate.	58·5	57·84	55·84	52·58
†Calculated as calcium sulphate.	2·33	2·14	1·89	1·27
Specific gravity	3·075	3·060	2·975	2·920
Fineness.				
Residue on 76 × 76 sieve.	8¼	9	8½	8
" " 50 × 50 " ..	4¼	4	4	4
" " 25 × 25 " ..	½	½	½	½

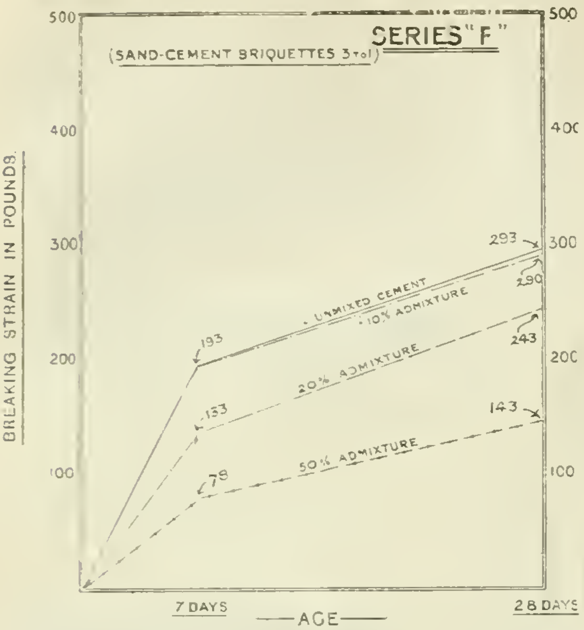
DIAGRAM OF COMPRESSIVE TESTS. SERIES "A."

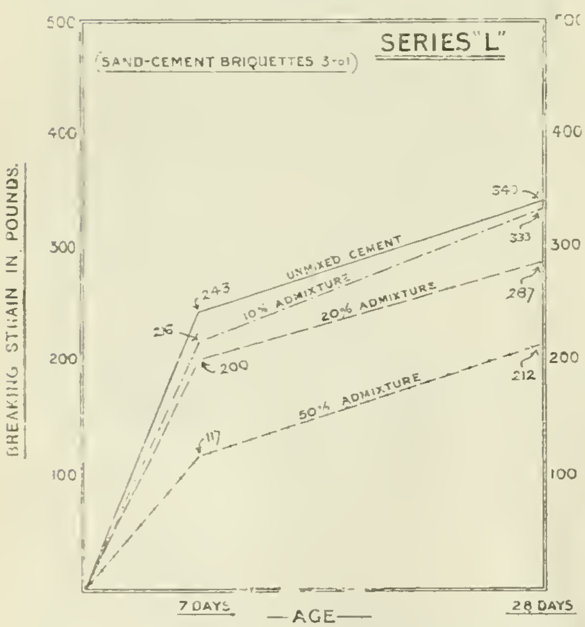
by many large users (notably by the Admiralty) in order to hydrate any uncombined lime or unstable lime compounds, and prevent their subsequent hydration and destructive expansion when the cement as a whole is gauged with water for preparing concrete or mortar. A moist ragstone, ground with such a cement, performs this function of limited hydration, and thus subjects the cement to what is, in effect, an artificial and rapid aeration. Cements which are not perfectly sound are improved by this procedure. But when a cement is rendered perfectly sound by aeration, this improvement disappears, as is evident from the following diagrams.

TENSILE TESTS AFTER AERATION.









These figures show that in an aerated, and therefore perfectly sound, cement, the ragstone acts precisely as would be expected, *viz.*, as a mere diluent. The effect is the more striking because these very samples before aeration showed a smaller strength unmixed than when mixed with ragstone. That this alteration of properties is caused merely by the increased soundness of the cement, which is induced by aeration, is proved by the following table:—

In this case the cement used was not aerated by long storage and yet gave tests which were considerably lower for the mixtures containing ragstone than for the unmixed cement. It was not a cement made in the laboratory, and therefore of better grade than can be readily manufactured on a large scale, but was simply an ordinary commercial cement of high quality and perfect soundness.

A word must be said as to what is meant by the word "soundness," which has been used in the previous paragraphs. A cement may appear perfectly sound to ordinary tests, and yet after setting may develop internal stresses which, though not sufficient to cause it to disintegrate or show any sign of actual failure, may diminish its strength appreciably. That this is not an uncommon condition is clear from the fact that many cements are considerably stronger after aeration than when fresh, as is evident from the following table:—

NEAT TESTS.

Breaking Weight in Pounds of Briquettes having 1″ × 1″ Section.

Unmixed Cement.		10 % Admixture.	
7 Days.	21 Days.	7 Days.	21 Days.
520 490 460	550 540 530	480 470 410	510 500 480
Average 490 lb.	Average 540 lb.	Average 453⅓ lb.	Average 496⅔ lb.

20 % Admixture.		50 % Admixture.	
7 Days.	21 Days.	7 Days.	21 Days.
410 390 380	410 410 390	240 220 220	300 290 250
Average 393⅓ lb.	Average 403⅓ lb.	Average 226⅔ lb.	Average 280 lb.

SAND TESTS.

Breaking Weight in Pounds of Briquettes having 1″ × 1″ Section.

Unmixed Cement.		10 % Admixture.	
7 Days.	21 Days.	7 Days.	21 Days.
300 260 250	330 320 280	250 240 ..	330 290 ..
Average 270 lb.	Average 310 lb.	Average 245 lb.	Average 310 lb.

20 % Admixture.		50 % Admixture.	
7 Days.	21 Days.	7 Days.	21 Days.
240 230 ..	300 270 ..	130 100 ..	200 160 ..
Average 235 lb.	Average 285 lb.	Average 115 lb.	Average 180 lb.

Breaking Weight in Pounds of Briquettes, 1″ × 1″ Section. Unmixed Cement and Sand (3 to 1).

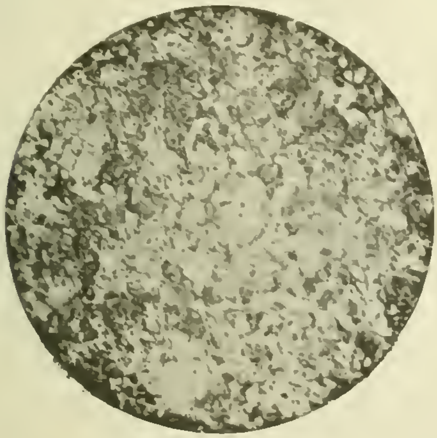
	7 Days.		28 Days.	
	Fresh.	Aerated.	Fresh.	Aerated.
Series "A"	191⅔	256⅔	270	303⅓
" "D"	259⅙	293⅓	276⅔	383⅓
" "L"	131⅔	243⅓	190	340

Incidentally, it may be remarked, that this great increase in strength is a strong argument in favour of systematic and thorough aeration of all cement that is to be used in heavy and important work. This has long been practised by many engineers to ensure safety; but if the cement be even slightly unsound, a large increase of strength will also accrue.

Reverting to the question of the difference of strength of unmixed cement and cement mixed with ragstone, it will be

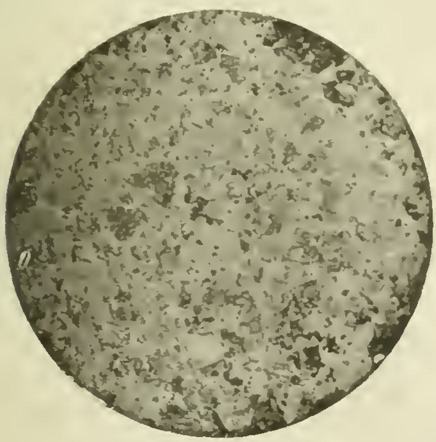
observed that although experiment has established that sound cement is not improved by the addition of ragstone, but is, on the contrary, deteriorated, yet the decrease of strength is smaller than that which corresponds with the percentage of diluent added. Thus a mixture of cement with 10 per cent. of ragstone is not necessarily 10 per cent weaker, although the ragstone is merely a chemically inert addition. The reason for this discrepancy becomes clear on studying the structure of set cement. For the purpose of this study, briquettes made from unmixed cement and from cement mixed with 10, 20, and 50 per cent. of ragstone were chosen, and very thin sections were prepared from them in the manner usual in petrological research. These were examined under the microscope, and a large difference in structure was at once apparent.*

The microscopic section of the unmixed cement consists of white, nearly opaque particles interspersed with dark patches fairly evenly distributed. Most of these dark patches are actual gaps, but some are merely plates of transparent crystalline substances, which, as they absorb little or no light, appear as gaps in the photograph.



Unmixed Cement.

A similar section of a briquette containing 10 per cent. of ragstone has a much closer structure. Very few true gaps appear in it, the interstices visible in the unmixed cement being filled with fine particles of ragstone.



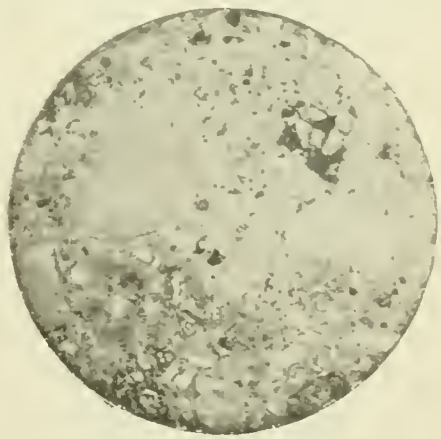
10 per Cent. Admixture.

With 20 per cent. of ragstone a similar filling is perceptible, but there is also visible a greater proportion of large crystals, either transparent or opaque.

With 50 per cent. of ragstone the filling of interstices is still noticeable, but the cement is, as it were, swamped with ragstone, and the section contains large inert crystalline masses which often exhibit cracks, and may be considered

20 per Cent. Admixture.

as reducing the strength of the material. The appended photo-micrographs illustrate these differences of structure.



50 per Cent. Admixture.

That the greater closeness of structure apparent in the microscopic sections of the mixtures has a physical existence, was proved by making actual measurements of the total cubic content of the cavities in the set cement and in the mixtures. A maximum density occurs with the 10 per cent. mixture, as is clear from the appended figures:—

Volume of Cavities in a Briquette, stated in per Cent. of the total apparent Volume of the Briquette.

——	Series 1.	Series 2.
	Per Cent.	Per Cent.
Unmixed cement	4·63	2·39
10 per cent. mixture	3·83	1·80
20 " "	6·32	3·79
50 " "	10·43	6·32

It will be seen that although the large quantity of ragstone in the 50 per cent. mixture communicates to it a loose and porous structure, yet the 10 per cent. mixture perceptibly exceeds the unmixed cement in closeness of texture, this fact serving to confirm the microscopic observations.

From both microscopical examination of the structure, and from measurement of the cavities in briquettes of unmixed cement and of cement mixed with 10, 20, and 50 per cent. of ragstone, it is evident that a mixture containing 10 per cent. of ragstone has a closer structure than any of the others, even than unmixed cement. The ragstone in

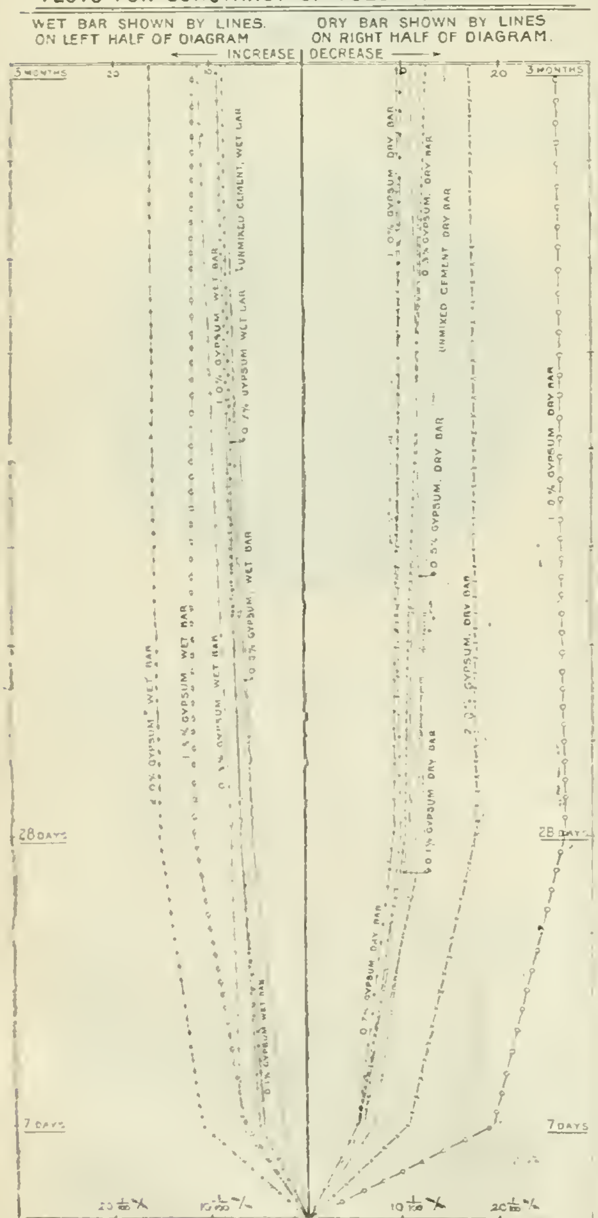
* The preparation of these photo-micrographs was kindly undertaken by Mr. Herbert Jackson, of King's College, London, to whose skill the beauty and accuracy of the original photographs are sufficient witness.

fact, during grinding, yields a quantity of fine powder which serves as a "filling" between the crystals formed when the cement sets. The absence of interstices thus caused appears to compensate in some measure for the fact that the ragstone is inert, and thus a cement containing 10 per cent. of what is simply a diluent is not necessarily 10 per cent. lower in strength than an unmixed cement. In fact, a briquette, even of neat cement, may be regarded as a concrete in which the strength depends not only on the true adhesive and cohesive quality of the cement, but also on the exact fit of the particles of cement and those of the inert matter, such as the coarse core. Any fine inert filling material may improve the closeness of texture of such a concrete if the average size of its particles be such as to occupy fully the spaces naturally present in set cement.

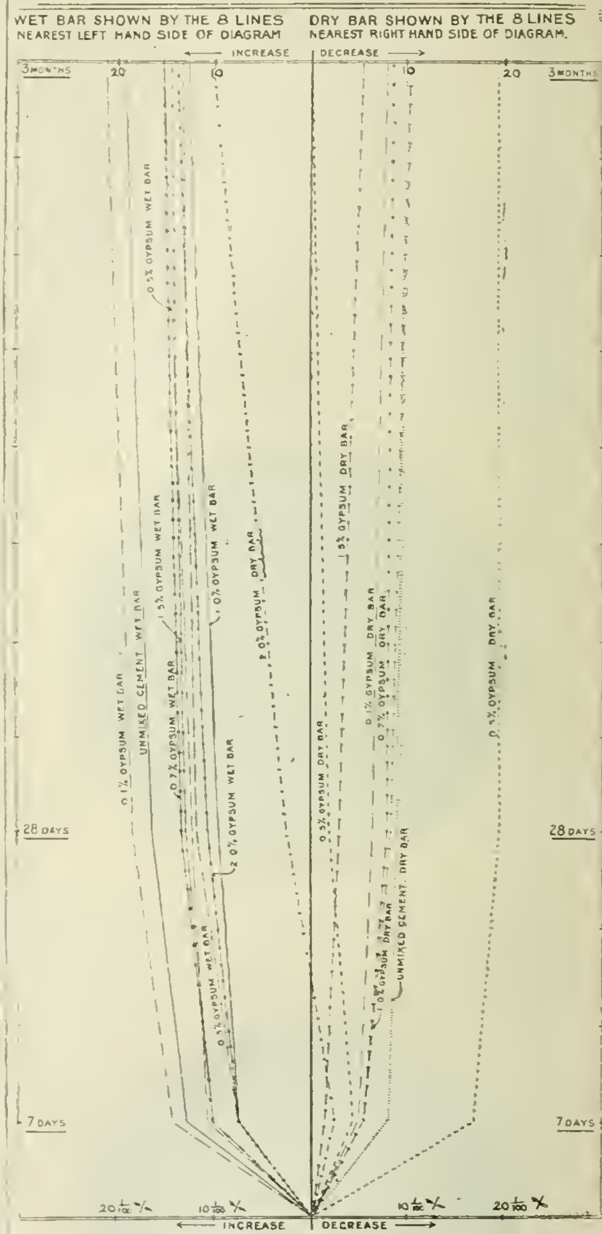
The whole question concerning the addition of ragstone may be summed up very briefly:—

1. Ragstone is not a cementitious substance, and its addition to cement is an adulteration.

TESTS FOR CONSTANCY OF VOLUME. SERIES "I."



TESTS FOR CONSTANCY OF VOLUME. SERIES "II."



2. Perfectly sound cement is weakened by the addition of ragstone.

3. This weakening is not fully proportional to the percentage of ragstone added, because the latter acts as a fine filling material and fills up the interstices naturally present in set cement.

4. Cement which is not perfectly sound, may be temporarily improved by the addition of ragstone. When the cement has become sound by aeration, this improvement disappears.

Many minor points were examined and determined in the course of the main investigation, but the most important results are embodied in the conclusions given above.

Additions to Cement other than Ragstone.

One of these, which particularly came within our purview in the course of our investigation for the London Chamber of Commerce, is gypsum. Gypsum is used largely in Germany, and to a considerable extent in this country,

in quantities not exceeding two per cent., and usually smaller than this, in order to lengthen the setting time of the cement. Regarding cement as a chemically finished product in the state in which it comes from the kilns, needing nothing but mechanical comminution to make it saleable, the addition of any substance to the finished clinker must be considered, in strictness, an adulteration. Thus gypsum becomes under this definition an adulterant. Nevertheless it is added for a distinct and useful purpose, and in quantities smaller than 2 per cent. does not affect the cement injuriously, as far as our own experiments indicate. The average results obtained with cement containing proportions of gypsum increasing from 0·1 to 2·0 cent. are given in the table below :—

Breaking Weight of Briquettes, 1″ × 1″ Section. (28 Days.)

—	Sample I.	Sample II.
	Lb.	Lb.
Unmixed cement	618	800
0·1 per cent. gypsum	587	693
0·3 " "	630	750
0·5 " "	623	777
0·7 " "	590	813
1·0 " "	620	823
1·5 " "	657	890
2·0 " "	613	860

It will be seen that no harmful effect on the strength of the cement can be detected.

The effect of the gypsum on the constancy of volume of the cement was also determined by the measurement of test bars of unmixed cement and of cement mixed with 0·1 to 2·0 per cent. of gypsum in a Bauschinger apparatus. The results are recorded in the appended curves. On the whole, a slightly greater tendency to expansion is perceptible when the proportion of gypsum exceeds 1 per cent., but the difference is not so great as to warrant the conclusion that gypsum causes any dangerous amount of expansion.

With these facts before us it is evident that though gypsum is an alien addition to Portland cement and therefore technically an adulterant, its use can be defended on the ground that it confers specific properties on the cement, and does not affect its strength or soundness unfavourably. The difficulty arising in the question whether cement containing a small quantity of gypsum, added for the purpose of regulating the setting time, can be legitimately sold as Portland cement, has been met by the Cement Trade Section of the London Chamber of Commerce, by the adoption of the rule which has been quoted above. This rule (page 853) expressly excepts gypsum in quantities up to 2 per cent. from the category of substances which, if added to Portland cement, constitute an adulteration. It will be noted that this convention is similar to that which has been in use for some years in Germany, where it appears to have worked satisfactorily.

The last and worst adulterant with which it is our purpose to deal, is blast-furnace slag. As far as our experience goes, this most objectionable addition to Portland cement is not employed on the Thames and Medway, but in certain other districts it is used in large quantities for the preparation of a grossly sophisticated product which is fraudulently sold as Portland cement.

We must not be understood as condemning true slag cement made by mixing granulated blast-furnace slag with slaked lime and sold under its proper title. This material is a perfectly legitimate product, and has its own uses; no one can reasonably object to its utilisation if it is not covertly substituted for Portland cement. But the addition of blast-furnace slag to Portland cement is another matter altogether. The general practice of the manufacturers who seek to increase their profits by the use of slag, appears to be to add to the clinker as it goes to the crushers as much crude blast-furnace slag as they consider can be mixed with Portland cement without risk of detection by the ordinary consumer, who buys cement in quantities so small that the cost of its analysis is too great for him to pay. The quantity added may be as much as 30 or 40 per cent.; and detection is not easy, or even always possible, for an unskilled observer. Apart from the fraudulent character of this addition, about which no doubt can well be entertained, there arises the question of its effect on the cement. And here it is necessary to make a small digression into the chemistry of the subject.

When Portland cement sets, a certain quantity of lime in the hydrated state is liberated. This lime in cement mortar, or concrete of fair closeness of structure remains distributed throughout the mass, and is there slowly converted into calcium carbonate. Now it is very possible that this lime could be utilised more effectively if it were provided with a certain quantity of silica, or an active silicate with which it could unite in manner similar to that of the lime of a puzzuolanic cement. Further, granulated blast-furnace slag will act as a puzzuolana and unite with slaked lime when the dry mixture of the two substances is gauged with water. It is therefore conceivable that granulated blast-furnace slag could be added to Portland cement in such quantity that its active silicates would unite with the lime set free in the normal setting of the cement. Whether this union would be advantageous to the strength of the cement is a matter for experiment. But whether it is or not, a cement thus dosed with granulated blast-furnace slag could not be legitimately termed Portland cement, and would have to be sold under a distinctive name. It may be mentioned incidentally that ordinary slag contains a good deal of sulphur (*e.g.*, 1 per cent.) in the form of calcium sulphide. This, slowly oxidising, would be likely to expand in the mass of the set cement and cause stresses, which could hardly fail to be injurious, and might be positively dangerous. Thus the burden of proof that the addition of granulated slag to cement is not actively harmful, rests upon the advocates for its use, and even if they prove their case they are confronted by the fact that the mixture is not and cannot be Portland cement.

But when the slag added to Portland cement is not granulated blast-furnace slag of the best composition for acting as a puzzuolana, but is the common stony material run out into trucks and allowed to cool spontaneously, instead of being rapidly chilled, the objection to the addition of slag is even stronger. In the first place this stony, slowly cooled slag is usually not of such a composition as to allow it to act as a puzzuolana. Next, even if it were, its condition—*i.e.*, annealed by slowly cooling instead of chilled by quenching—is most unfavourable to its puzzuolanic activity. Thus its tendency to unite with lime liberated by the setting of Portland cement would be likely to be small. It may be regarded for practical purposes as a diluent and makeweight. But this is not all; for much of this slag contains a notable proportion of sulphur, and the objectionable effect mentioned above, of the slow oxidation of this sulphur, holds equally in this case.

It is evident that the addition to Portland cement of blast-furnace slag, as usually practised, is not only an adulteration, but it is also an adulteration with a dangerous ingredient. Fortunately, this form of fraud is readily detected by analysis, although often escaping recognition by the usual mechanical tests.

In conclusion, we may reiterate our views as clearly and briefly as possible.

All materials added to Portland cement after the clinker comes from the kilns are adulterants, with the exception of gypsum, which is a recognised addition for a specific purpose in quantities not exceeding 2 per cent. Of the two adulterants which have been specially dealt with, *viz.*, ragstone and blast-furnace slag, the latter is by far the more objectionable, and it should be condemned and rejected by makers and users alike. In this view we believe we are supported by the great majority of engineers and manufacturers.

DISCUSSION.

Mr. H. K. BAMBER wrote as follows:—

Kentish ragstone could only be an adulterant in a good cement, but in a poor cement it might prevent the bad qualities having their full effect. But why make bad cement so as to require such addition? It was suggested that ragstone acted beneficially in some cements on account of the water in it, which had the same effect as aeration. Granted the presence of water in the ragstone, would this be taken up by the free lime only, or by some of the cement as well?

In several cases of good cements met with in the ordinary course of business, he found the gain by aeration in three days to be as nearly as possible $\frac{1}{32}$nd of the weight of the cement, except in a very dry atmosphere; and if exposure continued for a fortnight, no further increase of weight took place. While this increase of weight was taking place, the cement became a little lumpy, but nothing more than was easily removed by trowel or knife. The paper stated, "These figures show that in an aerated, and therefore perfectly sound cement," &c. Did that mean that an unsound and therefore bad cement became sound by mere aeration?

The results of breaking strains of cement and sand (pp. 854 and 855) were very high, and he would ask the authors if these were the results ordinary cements submitted to them in business gave, or was not this cement supplied for a special purpose by the manufacturer? Also what was its fineness? for the strength would depend on whether it contained all well-clinkered cement which had been finely ground. The authors were evidently struck with these results, for they made a kind of apology for them as follows:—"It was not a cement made in the laboratory, and therefore of better grade than can be readily manufactured on a large scale, but was simply an ordinary commercial cement of high quality and perfect soundness."

He found that aeration had but little effect, if any, on the strength of a genuine sound cement when mixed with sand. For a cement which, as before stated, gained $\frac{1}{32}$nd of its weight by aeration, the tensile strains averaged as follows:—

——	7 Days.	14 Days.	28 Days.	Cement.	Sand.
	Lb.	Lb.	Lb.		
Without aeration	146	157	198	1	3
After aeration........	133	168½	192⅓		

It was chiefly in a bad overlimed cement that the beneficial effect of aeration would be shown. There was no doubt that a cement having plenty of finely ground material to fill up the interstices of the briquettes was stronger than one where the air in the briquettes was not displaced by such material; but, that being so, why not grind the cement so finely that there might be plenty of very finely ground cement to do this work instead of using an extraneous inert substance? The results would be vastly increased tensile strength in sand and cement tests. Cements were much more finely ground now, with results that could not be formerly obtained even with genuine sound cements. Even genuine ground clinker above a certain size—say the residue left on 150 sieve (22,500 meshes per sq. inch)—would not set by itself when mixed with water. The presence of blast-furnace slag with cement could have none but a most injurious effect, and would often show bluish discoloration in the briquettes made from it, owing to sulphides. Gypsum could not be called an adulterant pure and simple, for it was added to give a special property to the cement, so much desired by engineers—"slow setting," which a genuine sound new cement could not possibly have. He was experimenting on this subject at present; and although an addition of 2 per cent. of gypsum to a quick-setting cement postponed the setting from 20 minutes to six hours and more, it had beneficial results as to strength as follows:—

——	7 Days' Average.	14 Days' Average.	28 Days' Average.
	Lb.	Lb.	Lb.
Cement and sand, without gypsum.	156	162½	189
Cement and sand, with 2 per cent. of gypsum.	176	186	230

These experiments repeated on the same cement—

	Lb.
Cement and sand, without gypsum	193
„ „ with 2 per cent. of gypsum	227½

In the latter case the other briquettes would be broken at two, three, and six months, to see if, in the long run, the gypsum had an injurious effect. That adding 2 per cent. of gypsum prolonged the time of setting from 20 minutes to six hours was a large result: How did it effect this? Evidently by in some way preventing the union of the cement with water, and delay of crystallisation. This was a good effect; but, as generally with every good there was an attendant evil, would this in time have an evil effect on the cement? This retarding of the setting was lost after a time—say a month or two. Probably the gypsum, by its water of crystallisation being gradually removed by the stronger affinity of cement for water, became anhydrous, and less soluble in the water with which the cement was gauged.

Mr. W. Matthews, as an engineer, felt greatly indebted to the authors for the information they had given in so clear and concise a manner. Generally speaking, he fully agreed with their views, but on one or two points he would have liked their experiments carried further. The utmost time covered with the ragstone experiments was only six months, whilst in the case of the gypsum tests it was only three months. Twenty years ago he made a series of experiments extending over eighteen months, and these showed that with good cement the increase of strength was continuous up to 12 months, or even more. He thought that if the experiments with gypsum had been continued for a longer period, they would have thrown an important light on the subject. The aeration of cement had proved so beneficial and important that it was his firm's intention in fresh contracts to provide for it being carried even further than hitherto. Some experiments made at Dover showed that the increase of bulk due to aeration resulting from six turnings was as much as 6 per cent. Taking the cost of cement at 26*s.* per ton, that increase of bulk was worth 1*s.* 6*d.*, which would be equal to 3*d.* per turn, or rather more than the actual cost of turning the cement.

Mr. Leedham White considered that ragstone owed its effects to its hygroscopic action; and he felt convinced as a cement manufacturer having some little knowledge of chemistry, that much more was due to its mechanical qualities than to its chemical qualities. He quite agreed with the authors that the improvement in certain cements by the addition of ragstone was simply due to the filling up of the interstices. He also agreed with Mr. Bamber that this effect might be arrived at with much greater advantage by grinding the cement itself finer, and if that course were followed, better results would be obtained from the usual admixtures with sand. As regards the report itself he would like to say a few words on the conclusions arrived at. As practical men of business the committee felt that the time had come when engineers should be reassured as to the quality of the cement supplied to them, and therefore, without any wish to harass trade, they felt bound to follow in this matter the example set by Germany 20 years ago, and reduce the manufacture of Portland cement to a matter of rule and definition. No doubt there might be good admixtures of cement with other bodies, and it was conceivable that chemical science might yet discover better methods of manufacture; at the same time the committee felt that they would not be hindering the march of science if they simply defined Portland cement as "a mixture of two or more suitable materials, intimately and artificially mixed in the requisite proportions, and afterwards properly calcined and ground, to which nothing has been added during or after calcination excepting that an addition not exceeding 2 per cent. of gypsum is permissible for the purpose of regulating the setting." That would not preclude any gentleman who discovered a better article or an admixture of articles suitable for the same purpose from selling it under its proper name and description, but not as Portland cement. With regard to the gypsum, the committee, after careful consideration, had concluded that it was best dealt with in the way mentioned in the paper, since the reasons for using it were so well known. There was a difference of opinion among chemists and engineers as to its advantages or disadvantages; and the committee preferred to abide by the opinion of such authorities as the authors of this paper, knowing at the same time that they were simply going over the same ground and arriving at the same conclusions as had been adopted in Germany some 20 years ago. Any engineers who objected to the use of gypsum had the remedy in their own hands, since they had nothing to do but specify that it should not

be added to the proper components in the cement contracted for. Again, some engineers attached great value to a slow-setting cement produced in the natural way. They would probably be surprised to learn that recent experiments made by Mr. Stiles showed that this was simply due to the fact that an excess of gypsum was present initially in the chalk used in the manufacture of that cement. He was sure that it was the earnest desire of the vast body of manufacturers to work heartily and honestly with the engineers and give them the best article obtainable, and in the production of it they would be guided by technical science and by the knowledge that as cement was important to engineers, engineeers were equally important to cement manufacturers.

Mr. OTTO HEHNER thought that the course which the London Chamber of Commerce had adopted in respect to the addition of gypsum to cement was liable to lead to friction and dispute. While it was easy to determine accurately the total amount of sulphuric acid contained in cement, and to calculate from this the corresponding amount of calcium sulphate, he knew of no analytical method whereby gypsum which had been intentionally added to cement could be quantitatively distinguished from the sulphates naturally present in fluctuating amounts. Thus a sample might contain naturally somewhat over 1 per cent. of sulphuric acid (SO_3), corresponding to almost 2 per cent. of gypsum Another might be almost free from sulphates, and would therefore stand the addition of 2 per cent. of gypsum. It would, in his opinion, have been better if the Chamber had laid down a maximum permissible limit of total sulphuric acid, than to draw a distinction between natural and added sulphates which eventually could not be enforced.

Mr. JOHN HUGHES agreed with Mr. Hehner's remarks, and said that his view was borne out by some analyses of very old mortars which he had made. For instance, the mortar from Tintern Abbey, which was of very superior quality, contained as much as 1·37 per cent. of sulphuric acid, equal to 2·33 per cent. of gypsum, and he therefore thought it was unwise to arbitrarily fix the limit of gypsum in the cement at 2 per cent. He also regretted that the authors' results had not been accompanied with chemical analyses of the cements tested, and also with grinding tests of the fineness of the ragstone and the cement respectively. Mechanical tests occupied a long time, but it was very easy to test the fineness of grinding by means of sieves, and the general quality of the cement by analysis. He thought the addition of such further results would greatly enhance the value of the paper.

Mr. A. G. BLOXAM thought it extraordinary that so small a proportion of gypsum should have so much effect on the setting of cement as had been stated. Referring to Mr. Hughes' remarks as to the large percentage of sulphuric acid found by him in ancient mortars, it was possible that that might be explained by the fact that the mortars had been exposed for centuries to a sulphurous atmosphere. On the other hand, in the case of cements, it was not improbable that much of the sulphuric acid added would be expelled by the silica during the process of burning, notwithstanding the generally basic character of cement. He would suggest that the sulphate found might be little more than that arising from the sulphur of the fuel used in kilning the cement.

Mr. D. B. BUTLER wished to heartily endorse the authors' remarks as to the general rectitude of the cement manufacturers in this country. A great many samples of cement passed through his hands for testing purposes, and the results fully bore out that opinion. Only one or two small makers used Kentish ragstone, and he believed that their product was used almost entirely for home consumption. He therefore wished particularly to emphasise this fact, because a report had been spread abroad, especially in our Australian colonies, that London manufacturers used adulterants, and such statements had most injuriously damaged the English trade in those colonies. He thought that the question of the effects of additions of gypsum had not yet been sufficiently thrashed out to be accepted without reserve. The authors' results were based on two samples, and at 28 days only, which was hardly sufficient to be conclusive. Undoubtedly the general effect of gypsum was to make a cement slower setting, and as it was the growing practice of engineers to demand a very finely-ground and, at the same time, slow-setting cement—two qualities which were antagonistic in themselves—hence the necessity of gypsum. He had himself made experiments on the ragstone question, the results of which were embodied in a paper read before the Society of Engineers some 12 months ago, and, with some modifications, confirmed the statements of the authors. He found that an admixture of ragstone reduced the cementitious value of the cement in every instance, though sometimes when gauged neat the result was higher. He was disappointed to find that there had been no investigation as to the effect of the admixture of ragstone when used in sea-water and in air, especially as cement was used almost as much under those conditions as in fresh water. His own experiments showed that under such conditions the effect of the admixture was much more harmful. If, as the authors suggested, the only advantage of using ragstone was a certain slaking effect on the free lime in the cement, one might as well simply water the clinker. His own experiments as to the comparative effect of admixtures of ragstone and finely-ground sand proved that the advantageous action of the admixture was a purely mechanical one. In the table given by the authors on page 856 there was an average of only three briquettes at each date. That seemed to him a very small number on which to base an average; it would have been more satisfactory to have taken an average of five or six at least, especially in such an important investigation.

Mr. W. H. STANGER said that Mr. Butler was mistaken in supposing that the conclusions were based merely on the averages of three briquettes. Every result put forward represented two or three hundred tests, and thousands of briquettes had been tested in their series of experiments. It would have been impossible to have given the whole of the figures, so they simply selected typical examples. To those members who wished for further details he would recommend a reading of the full report of the Chamber of Commerce.

Mr. W. F. REID thought the Society was indebted alike to the authors for their contribution to this subject, and to the Chamber of Commerce for putting it at the disposal of the Society. With regard to the action of the Chamber, he noticed that two definitions of Portland cement were given, one at the beginning of the resolution and one later on, in the document which the manufacturers were invited to sign. He would like to know why these specifications differed. He, himself, regarded the addition of 2 per cent. of gypsum allowed by the second definition as being quite as much an adulteration as the addition of any *useful* substance to cement could be. He had met with cases in which coke containing a large percentage of ash was actually preferred by makers simply on the ground that it increased the output. With regard to the analyses, he would like to know whether the silica given at the beginning was soluble silica. Also whether the percentage of water had been taken into account. The addition of gypsum was not really necessary to render a cement slow-setting. Some German firms with which he was acquainted had abandoned that practice in favour of another process of manufacture which had the same effect. There were substances the addition of which to cement would undoubtedly prove beneficial, such as soluble silica, which would add to the hydraulic properties of the cement, and therefore enhance its value. The advantage of adding to the bulk by aeration was at least questionable; and he thought that cement should be used by weight alone, or if measured, then the weight of a given measure should be ascertained. The reason German makers had adopted a uniform sand was to obtain the most regular results—not the highest; and the same practice would be advantageous in England. It had been said that the beneficial action of ragstone—when it was beneficial—was due to the fact that it could be ground finer than the ordinary clinker. In that connection it would be important to know the composition of the finest portion of the powdered mixture of ragstone and cement, as well as the composition of the residue on the sieve. In Germany, it had been recently proposed to grind the sand and cement together, and so they got greater strength of mortar.

Mr. E. Wood regretted that no users of ragstone were present to contribute to the discussion. If Mr. Blount were one of the audience, he would probably inquire whether it was reasonable to expect a thoroughly sound cement ever to be turned out; for the recommendations of the Chamber of Commerce stated that a sound cement should have no ragstone in it, and therefore the natural corollary was that some might be added to a cement which was *not* perfectly sound. According to the diagram L, a 10 per cent. admixture bore a strain of 216 lb. at 7 days, and at 28 days a strain of 333 lb. But what would have happened if the test had gone on for ten days longer? Would not the 10 per cent. mixture have tested better than the undiluted cement?

The Chairman thought that it would be desirable that the authors should give analyses of the cements tested, for unless they knew their chemical composition it was difficult to judge the effect of the addition of ragstone to them. He would also like to have more exact definitions of the terms "sound cement" and "aeration." Did the former merely imply manufacture according to a standard specification? And could the advantage of increased bulk said to result from the latter be secured by mere mechanical means? If so, why let the cement lie for three months?

Mr. Bertram Blount said that in mercy to the audience he would endeavour to reply as briefly as possible. Dealing with the Chairman's questions first, the subject of aeration was very interesting chemically. It was not merely a question of taking up water and carbonic acid. Le Chatelier had shown that water added to cement underwent transference from particle to particle until it had satisfied such compounds as were most eager for it. Such action could not be effected rapidly, and thus all attempts to effect it artificially had failed, simply because time was a necessary function. The chemical aspect of the question was too wide to be dealt with in a reply. A sound cement might be described as one in which the internal stresses which arose after the cement set were reduced to a minimum so small as to exercise no material effect on its strength. It was with a view to determine such stresses that hot tests had been devised. With regard to the number of tests and analyses made, as Mr. Stanger had already stated, the results brought forward that night were but an epitome of the work actually done. Although they had permission to do so, they did not feel justified in transferring the work bodily from their Report to the London Chamber of Commerce; and, moreover, to do so would have been wearisome. They had therefore selected typical cases. But if it would conduce to the better understanding of the subject they would be happy to incorporate the analyses alluded to by several speakers. He need not take up time in referring to Mr. Bamber's remarks, since they were in close agreement with the paper. He agreed with Mr. Matthews that it would have been better to carry the experiments further; but time pressed, and they had to pronounce as far as their data permitted. An ordinary test extended to seven days; they had adopted six months, and could therefore hardly be said to have erred from want of deliberation. No doubt longer tests were desirable, and should it be possible to make them, he trusted the Society would accord a hearing to the results. As to the increase of bulk by aeration, that was a matter which concerned contractors, who would now no doubt proceed to make the large profits said to accrue from additional turning. Mr. White had hit the nail on the head when he said that the chief use of ragstone was to "chock up the chinks." It was a body eminently adapted for that purpose; and tests with added powdered sand were not comparable, because the latter was a tougher article and did not "chock up the chinks" to the same extent. It grieved him greatly to have to differ from Mr. Hehner when he said that the addition of gypsum could not be detected. Mr. Hehner's reputation as an analyst was unsurpassed; and if he studied the question a little he would find it could be done. At any rate, it was done every day at Broadway, Westminster. He heartily concurred with Mr. Bloxam's remarks. It was matter of knowledge that one might take a cement, a tolerably basic material, with 62 per cent. of lime, and ignite it at a temperature of 1,100° C., and expel almost the whole of the SO_3, thus arriving at an erroneous result for the true loss on ignition, *viz.*, CO_2 and H_2O; in fact, it was a matter of delicacy to adjust the temperature so as not to expel SO_3. Mr. Butler's reference to the number of briquettes had been dealt with by Mr. Stanger, and he could only add that if anybody had waded through the tests registered in three large folios, as he (the speaker) had done he would not think that too few had been made. With regard to the analyses of ragstone referred to by Mr. Reid, the composition was given in the dry state. The quantity of moisture had been determined, but was not recorded in the tables. It would vary a great deal. The water in the analyses was simply the combined water; and similarly the silica was combined silica after the ragstone had been decomposed by hydrochloric acid in the usual way. The quantity was insignificant, but it was necessary to determine it in order to ascertain if there existed in ragstone any silica capable of acting as a puzzuolana. The analyses completely refuted any such idea. Mr. Wood's remarks as to an apparent discrepancy in the diagram, Series L., were not quite audible to him. If Mr. Wood would communicate with him, he would endeavour to clear up the point.

Mr. Walter Hewitt, Managing Director of the Dartford Portland Cement Co., Ltd., sends the following remarks:—

The paper appears to place beyond doubt the fact that the action of Kentish rag or Portland cement is not in any way chemical, but purely physical and mechanical, and that this effect may be accounted for in two ways: firstly, by filling up the interstices between the particles of the cement it makes the mass denser, and by bringing into full effect the properties of each particle increases the strength of the cement. I am given to understand that other materials (such, for instance, as fine sand), if used in the same way (*i.e.*, added to the cement clinker at the crusher), would produce exactly the same effect. I do not know if the authors of the paper have experimented in this direction. If they have, it would be interesting to hear their experience. Should this be correct, it would be one more proof that there is nothing in Kentish rag to justify those who object to its being considered an adulterant. The second explanation advanced by the authors of the paper, of the apparent usefulness of the Kentish rag is, that the moisture it contains slakes any particles of free lime there may be in the cement, and that its addition has thus the effect of aeration. This opens up a very big and important question, *viz.*, Should it ever be necessary to aerate or air-slake cement? The practice is frequently adopted in the case of highly-limed cements, *i.e.*, in cements which are manufactured with a large proportion of lime in order to attain a high tensile strain. Should any pieces of under-burnt clinker—known in the trade as "green" or "yellow"—be ground up with the rest, the effect is the same as if so much free lime were added, and it is to obviate any risk of this that engineers so often insist on the cement being thoroughly aerated before use. But are they not seeking a remedy in the wrong direction? Prevention is always supposed to be better than cure; and it ought not to be impossible to devise some means of ensuring the proper calcination of the clinker, and thus doing away with the tedious process of aeration. The same question crops up with regard to gypsum. The advocates of its admixture with Portland cement appear to rely mainly on the fact that its addition (up to 2 per cent.) is allowed in Germany. But there are two facts tolerably well known about German cements. One is, that most of them, if not all, are manufactured from various and different kinds of raw materials from those in England; and the use of gypsum may be rendered necessary by conditions which do not obtain here. The other fact is, that German cements, as a rule, attain their ultimate strength at a comparatively early date, and instead of toughening with age, like English cements, they merely harden and become brittle. This is a very serious defect, and it may not be very far-fetched to attribute it in part to the use of gypsum. But in any case I contend that gypsum is not necessary even here. It is added to retard the setting of the cement. It may be taken as a fairly general rule that a quick-setting cement is not to be relied on for hard wear, and it is an undeniable fact that a cement manufactured of chalk and clay in proper proportions, and *thoroughly well burnt*, will not set quickly, and, consequently, neither gypsum nor anything else is required to make it slow-setting. This brings me back again to my

former statement, that it would be well if some means could be devised for ascertaining that proper calcination of the cement had taken place. If this could be done, and a standard of test set up and accepted by engineers and the trade generally, both aeration, with all its attendant inconveniences and loss of time, and the addition of gypsum would become things of the past.

Mr. H. K. G. Bamber (Messrs. Knight Bevan, and Sturge, Northfleet, Kent) wrote to ask if Messrs. Stanger and Blount had made any experiments in the direction of adding gypsum to the raw materials during the manufacture of Portland cement, as distinct from what appeared to be the usual custom of adding it to the calcined clinker; and which was the method adopted by them for their experiments for this paper; and whether they had found that gypsum thus added, in the earlier stages of manufacture, which would of course be subjected to the extreme heat of the cement kiln, had the same effect in retarding the setting of the finished product. Some years since, when making some experiments on this subject of the adulteration of Portland cement, he found, as Messrs. Stanger and Blount had now done, that Kentish rag added to a good and perfectly sound cement only acted as a diluent, but if added to an overlimed cement, or one in the manufacture of which the raw materials had been badly mixed, thus preventing perfect combination during calcination, it had the effect of increasing the tensile strain at early dates, and perhaps partially counteracting for the time being some of the dangerous qualities which such cement would be certain to develop, and which of course would never be made a sound cement by such additions. While on the point of soundness, what did Mr. Blount consider the extreme and crucial test of soundness in cement? To be perfectly sound must it be capable of passing the stringent test introduced by Deval, or would it be sufficient if the cement failed under this test but was of sufficiently good quality to pass the less severe test now in vogue? He would like to have seen the comparative tests with and without the addition of gypsum carried to a much longer period.

In reply to the above communications, Mr. B. Blount adds:—"It is certainly desirable that cement should be manufactured so as to require no aeration. This can be done if it be understood that cement-making is a chemical manufacture and needs skilled chemical supervision. The Deval test mentioned by Mr. H. K. G. Bamber is the best criterion at present known for the soundness of a cement, and those makers whose cements pass it may feel assured that their manufacture is more successfully conducted than that of makers whose cements fail to pass the test. The general adoption in this country of a test of admitted severity cannot fail to have the excellent result of putting our trade not only on a par but in advance of that of our commercial rivals abroad.

AN IMPROVED ADJUSTABLE DRIP-PROOF "BUNSEN."

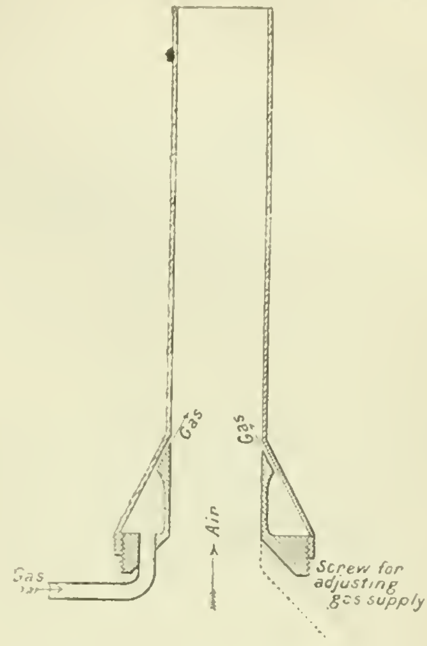
BY W. P. EVANS, M.A., PH.D.

The writer feels himself compelled, by a recent publication of Dr. Hugh Marshall (this Journal, 1897, 395), to publish an account of a burner with which he has been working for some time past, although it is still in the experimental stage.

The main idea of the burner is a *side gas* combined with a *central air* supply (as in the Marshall burner) *each* being made as *axially symmetrical* as possible. The base of the burner consists of two turned metal cones which screw one over the other, allowing the gas to enter by the annular opening between them. Over these cones is situated the ordinary mixing tube. The bore of this tube is also carried downwards completely through the inner cone. The gas supply may be readily and accurately adjusted by means of the screw threads of the cones. In the *smaller* sizes the ordinary side holes may *also* be used with advantage to gain an increased air supply.

Details of construction are evident from the accompanying figure.

If the gas supply is constant the burner can be easily and economically constructed out of sheet brass, and the cones fixed permanently in their right positions. The modification seems specially adapted for high-power burners, the short mixing tube necessary proving here a great



advantage. Instead of drip-proof it may fairly be called shower-proof, even a beaker full of water failing to put it out.

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SESSION 1897–98.

Meeting held on Wednesday, October 29th, 1897.

CHAIRMAN'S ADDRESS.

THE MANUFACTURE AND PROPERTIES OF CARBORUNDUM.

BY CHARLES A. KOHN, PH.D., B.SC.

The wide range of temperature through which reactions can be studied to-day, is one of the most marked advantages of the conditions of modern experimental investigation. Chemical changes formerly unknown have been realised between the temperatures of solid oxygen and gaseous silicon.

In chemical industries the dissociation temperatures of the products of combustion from furnaces, as well as the fusibility of porcelain and other refractory materials, have limited the temperatures obtainable to 1,700°—1,800° C. Platinum, which melts at 1,775° C., can be refined in the oxy-hydrogen flame, the temperature of which is about 2,000° C.—

the highest temperature obtainable before the introduction of the electric furnace. The temperature of the latter depends upon that of the electric arc. This Violle has estimated at 3,500° C.; and Moissan's experiments go to show that the actual temperature that can be attained in his various forms of electric furnace approaches this figure, and that it is a direct function of the intensity of the current employed.

The preparation of the metallic carbides, especially in a crystalline form, has been one of the most notable achievements of these new conditions of work.

It has been known for a long time that certain metals are able to take up varying and proportionately small quantities of carbon, but it has been reserved for Moissan to show, by means of his researches with the electric furnace, that the carbides form a well-defined group of compounds possessing very marked and novel properties.

Two of these carbides have already risen to the dignity of chemical industries—calcium carbide and carborundum; the energy of Niagara is now employed for both manufactures.

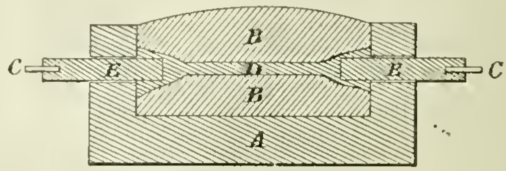
Owing to the kindness of the Carborundum Co., opportunity was given me to visit their works and to give you such details of the manufacture as might be of more than passing interest. For my information I am entirely indebted to Mr. E. G. Acheson, the discoverer of carborundum and the President of the Company, and to Mr. A. J. Fitzgerald, and I gladly avail myself of this opportunity of thanking them for the liberality with which they met all my inquiries, as well as for the care and trouble they have taken to enable me to illustrate this paper with specimens and photographs.

The experiments which led to the discovery of carborundum were undertaken by Acheson in March 1891. With the hope of obtaining carbon in a crystalline form, an electric current was passed through a mixture of carbon and clay placed in a carbon-lined iron bowl, the idea being that the clay, or the aluminium reduced from it, would dissolve the carbon and deposit it in crystals on cooling. The current employed was sufficient to fuse the mixture; a violent reaction took place, and a few hard blue crystals—not diamonds—were separated from the mass after cooling. A larger furnace made of refractory bricks was next tried, into opposite ends of which adjustable carbon rods were inserted as electrodes, and an alternating current of 100 to 200 ampères with an adjustable potential of 1 to 50 volts used. The crystals obtained were exceedingly brittle, but sufficiently hard when ground to a fine powder to abrade the diamond; from their colour and general properties, as well as from the materials used in their manufacture, they were regarded as composed of carbon and alumina (corundum), and named "Carborundum." This was in 1892 and before the product had been analysed. In the same year Schützenberger prepared amorphous silicon carbide (Comptes Rend. 1892, **114**, 1089) by strongly heating a mixture of carbon and silica in a bone-black crucible, and showed that the green amorphous product formed had the formula SiC. The temperature of the furnace was evidently insufficient to allow of the formation of the crystalline carbide.

Acheson's early experiments, already referred to, soon led him to recognise the fact that the silica present in the furnace played an important part in the production of the carborundum, and direct experiment, in which the alumina was entirely omitted, confirmed this. A mixture of equal proportions of carbon and sand, to which 20 per cent. of salt on the total mixture was added, was found to give a largely increased yield of carborundum. The salt was added to facilitate the fusion of the mass. The furnace employed was considerably modified, especially with the view of overcoming the serious difficulty of starting the fusion of the mixture and of avoiding the continual changes in its internal resistance. These two objectionable factors were simultaneously eliminated by building a solid core of carbon between the two electrodes along the length of the furnace, and thus dividing the single arc previously used into a very large number of arcs of varying size and intensity, at the same time providing a conductor for the current. It is this arrangement that forms the essential feature of the furnace now in use; it allows of the easy regulation of the current, and entirely avoids any possible electrolysis of the mixture.

A longitudinal section of the furnace is shown in Fig. 1. A represents the walls of the furnace, B the mixture of carbon, salt, and sand, D the core of granular carbon enlarged round the ends so as to surround the electrodes E, which are provided with copper connecting-rods C.

Fig. 1.



Longitudinal Section of Furnace.

The working of this furnace was a preliminary to the method now employed. It was sufficiently successful to establish the preparation of carborundum on a large scale. The details of the present conditions of manufacture can now claim our attention.

The Raw Materials.—These consist of coke, silica, salt, and sawdust. The coke is the ordinary beehive-oven product; the other ingredients call for no special comment. The salt acts partially as a flux, and also helps in the separation of the crusts of the different products that result in the manufacture. A considerable portion is volatilised. An addition of sawdust has been found advantageous, since the gases evolved during its carbonisation leave a porous coke, which serves to some extent as an exit for the carbon monoxide evolved. Formerly, sudden evolutions of gas occurred, forming "blow-holes" some distance below the surface of the mixture in the furnace, and often necessitating the removal of the overlying material; this is overcome by the addition of the sawdust.

These raw materials are delivered on the ground floor of the stock-house, a large building fitted with stages and elevators. The coke is roughly broken by a grinder, and then sorted by two cylindrical revolving screens. The coarsest portion is retained in the first screen, the remainder passing down a shoot to be ground to powder. The part kept back in this screen passes to a second similar screen, through the meshes of which the pieces, about $\frac{1}{2}$ to $\frac{3}{4}$ in. in length, pass, whilst the larger pieces are separated and ground with the finer portions from the first screen. The product which passed through the second screen is used for making the "core" of the furnace.

The ground coke, sand, salt, and sawdust, after being weighed out, are thoroughly mixed in a mechanical mixer. The following are the proportions used:—

Coke	34·2
Sand	54·2
Sawdust	9·9
Salt	1·7
	100·0

This mixture is now ready for the furnace. Two men can do the whole work of the stock-house required for 10 furnaces.

The Furnace House.—Ten furnaces can be accommodated in the furnace house; at present five are in use, which are worked successively. The sides of the furnaces are built up as each one is filled, and subsequently pulled down to allow of the removal of the carborundum, so that only the bed and ends of each furnace are permanent. This has been found advantageous, owing to the walls becoming incrusted and consequently to some extent conducting, when used repeatedly, thereby causing a decreased yield of carborundum. The furnaces, which are built of fire-brick, are 16 ft. long, 5 ft. wide, and about 5 ft. high. The ends are very solidly built, and about 2 ft. in thickness. In the centre of either end are the terminals, which convey the current through the central core of the mixture. These consist of 60 carbon rods, 30 ins. long and 3 ins. diameter, into the outer ends of which small pieces of $\frac{5}{8}$-in.

copper rod are fixed. A square copper plate, bored with 60 holes to receive the copper rods connected with the carbon electrodes, holds these latter in position. The carbons are placed in position from the inside of the furnace, starting with the bottom row, the spaces between the rods being packed tight with graphite as the fixing proceeds. The graphite packing prevents the oxidation of the carbons, and thus adds very materially to their durability. Each copper plate is provided with four projecting rods, to which the cables carrying the current are bolted.

Three rows of bricks are left permanently along the sides of the furnace, the remainder being built up as stated. At first the bricks are carried to a height of about 4 ft. Before putting in the mixture, iron screens are put round the inner ends of the furnace, about 4 ins. away from the carbon terminals, so as to keep the mixture from coming in contact with the latter. The mixture is then thrown into the furnace until it is rather more than half full, when a semicircular trench about 10½ ins. radius is made along the length of the furnace from end to end. Into this the weighed "core" is placed, a mixture of old and new core being generally used. The bottom of the trench reaches a little above the bottom row of carbons. The core is built up so as to complete the circle, forming thus a solid coke cylinder the length of the furnace and 21 ins. in diameter. To connect the core with the terminals, finely-ground coke is packed into the space between the carbons and the iron screens; next, the sides of the furnace are built up to the full 5 ft., the screens removed, and, finally, more mixture thrown in and heaped up to a height of about 8 ft. The furnace is then ready for use.

Fig. 2.

Carborundum Furnace, ready for Operation.

Fig. 3.

Carborundum Furnace at work, showing position of Carbon Terminals.

The Electric Current.—The favourable conditions for the supply of electrical energy at Niagara led to the erection of the present plant in 1895, the successful manufacture of carborundum having been previously worked out on a smaller scale at Monongahela, Pa.

The current as supplied by the Niagara Power Company has an electro-motive force of 2,200 volts. By means of a transformer this is reduced to 165 volts. The transformer has a maximum capacity of 830 kilowatts (1,100 horse-power); it is connected with a regulator, by means of which the current can be raised to 220 or lowered to 85 volts, and transmits an alternating current to the furnace. This reduction of the voltage involves a loss of about 4 per cent.; suitable cooling arrangements are adapted to the transformer and regulator to avoid overheating. Two copper conductors, each having a sectional area of 8 sq. ins., convey the current to the furnace room; these are bolted on to the outside copper plate of the furnace as described. The circuit is completed in the furnace room by means of a water rheostat, the ordinary methods of completing a circuit being too dangerous for such large currents as are employed in this manufacture. This rheostat consists of a circular iron vessel filled with salt water, into which a large iron plate is lowered by means of a pulley. To break the circuit, the plate is, of course, simply lifted out of the tank.

Working of the Furnace.—The charge of new mixture per furnace has gradually been raised, with improved conditions of work, to about 30,000 lb.; a considerable quantity of old mixture is also always used over again—sufficient to fill up the furnace. The core forms about 3 per cent. of the total charge; a mixture of new and old core is employed, the latter being added on account of its better conductivity.

After the cables have been bolted on to the plates of the furnace, the current is completed through the water rheostat in the transformer room, and the readings of the voltmeter and ammeter watched carefully. A pressure of about 190 volts is required in the start, but as the coke gets warmed the resistance decreases, and within an hour to an hour and a half becomes practically constant. As the current increases the regulator is run back, reducing the electromotive force, which is kept at about 125 volts till the end of the run. During this period the ampères increase from 1,700 to 6,000, and the kilowatts from 125 to 745. Very little regulating is subsequently required, but a very gradual decrease in resistance appears to be continuous, and is due to the formation of a layer of graphite immediately round the core, thus increasing the diameter and conductivity of the latter. This formation of graphite is regarded as due to the decomposition of silicon carbide after formation by the intense heat in this portion of the furnace.

The subjoined tables show the conditions of working now as compared with last year. In the early part of 1895 the energy expended per pound of carborundum made was about 7 kilowatt hours, whilst now it is only 3·9—a reduction of over 44 per cent.

Working of Carborundum Furnace.

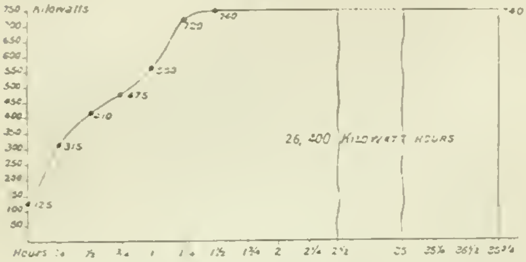
——	April 3rd, 1896.	June 28th, 1897.
Mixture (new)*	18,500 lb.	29,900 lb.
Core. Old	..	153 lb.
Core. New	800 lb.	833 lb.
Carborundum	3,446 lb.	6,735 lb.
Amorphous carbide	3,880 lb.	4,960 lb.
Ampères. At start	2,500	1,700
Ampères. Approximately constant.	6,000	6,000
Volts. At start	150	190
Volts. Approximately constant.	125	124
Length of run	23 hours 12 mins.	35 hours 45 mins.
Kilowatt hours	16,900	26,100
Kilowatt hours per pound	4·9	3·9
Pounds per hour	149·0	188·0

* A considerable amount of old mixture was also used.

No immediate change is apparent on starting the furnace, but within half an hour the decomposition of the sawdust in the mixture is rendered evident by the smell. Carbon

monoxide is gradually evolved, and after three or four hours it comes off in sufficient quantity to be lighted, when the

Fig. 4.



sides and top of the furnace become enveloped in the flames. No less than 6 tons of this gas are given off during the run of a furnace. The equation representing the decomposition is :—

$$SiO_2 + C_2 = 2CO + Si$$
$$Si + C = SiC$$

After 5 to 6 hours from the start, the progress of the working of the furnace is indicated by the appearance of the yellow sodium flames; fissures begin to form in the mass, and the upper portion sinks down as much as 3 ins. This decrease in bulk continues till the end of the run, but more mixture is thrown on as it proceeds, so that by the end of the run it reaches up to the top of the side walls of the furnace. From $\frac{1}{2}$ to 1 ton extra mixture is thus added. After 36 hours the heating is finished. The current is then broken and the furnace allowed to cool for 24 hours. The length of run is considerably longer now than formerly; both the energy expended, and the rate of production are favoured by this change.

Products obtained.—After the furnace has cooled, the side walls are partially pulled down and the loose unchanged mixture on the top of the furnace raked off. This is used over again. Its composition is given below, and is seen by comparison to be essentially that of the original mixture, except that the proportion of salt is very largely increased—due, of course, to volatilisation from the hotter parts of the furnace.

Analysis of Outside Mixture after Heating.

I. Salts soluble in water	11·19
II. Ash (insoluble in water)	55·85
III. Free carbon	32·96
	100·00

——	I.	II.
SiO_2	..	94·28
NaCl	96·57	..
F_2O_3	0·71	1·75
Al_2O_3	0·04	4·24
CaO	0·19	0·21
MgO	0·04	0·14
P_2O_5	0·02	..
SO_3	2·43	..
	100·00	100·62

Below this is a well-defined layer of amorphous carbide of silicon, with which varying quantities of unchanged mixture are mingled. This is the same product as was obtained by Schutzenberger, and, as the analyses show, it is practically pure carbide of silicon. The portions of this crust nearest the core are of a greenish tinge—due to the contained iron. On the outer portions of the crust the volatilised salt can always be seen. The amorphous carbide forms a readily friable mass of a dirty grey colour, and is easily removed, being first cut through with iron bars and then shovelled out. As yet, this product is entirely valueless—very unfortunately so, as its forms over 40 per cent. of the total carbide produced. Its amorphous nature is due to the relatively lower temperature of this portion of the furnace, and it is the sole product obtained when the furnace is only run for a short time. It has not been found possible to convert it into the crystalline variety.

The change from the amorphous carbide to the carborundum is well marked, the latter forming a layer from 10 to 12 ins. thick round the core.

The outside of this crust of carborundum is first scraped clean with spades, then loosened with bars, and the carborundum dug out. The appearance of the mass when this crust is opened is exceptionally striking, the whole forming a glittering mass of brilliant crystals measuring up to half an inch in diameter and all beautifully iridescent. The colour varies from a steel-grey to a brown-green, and many of the crystals are quite translucent.

Below the carborundum is a thin layer of beautifully crystalline graphite, and a similar series of crusts obtain on the other side of the core. This formation of graphite is most probably due to the carbide taking up additional carbon when hot and re-depositing it as graphite on cooling, since many of the carbides prepared by Moissan behave similarly. The carbon of the core is partially converted into graphite also; this, too, is probably due to its having been taken up by the carbide and subsequently separated, for the quantity is not found to be appreciably increased even when the same core is used several times over.

Fig. 5.

Carborundum Furnace opened, showing Carborundum and Carbon Electrodes.

Fig. 6.

End View of Furnace after Heating, showing Carborundum and Core. (End wall specially removed for this photograph.)

One other product of the furnace is worthy of note. Tiny feathery tufts are often found sticking to the carborundum crystals, and occasionally the same product is found in larger quantity in the inner parts of the furnace in the form of pale greenish-yellow masses, excessively

light and extraordinarily like a fungus in appearance. On one occasion a piece quite 18 ins. long, about 1 ins. broad, and 2 ins. thick was obtained. At first this was regarded as volatilised silica, but it is really carbide of silicon in an extremely fine state of division. Under the microscope it appears distinctly crystalline, and consists essentially of perfectly transparent prismatic needles, amongst which a number of equally transparent hexagonal crystals were observed. The product closely resembles that obtained by Moissan by the combination of the vapours of carbon and silicon, and in all probability it has a similar origin.

Working up of the Carborundum.—This consists simply in grinding, washing with dilute acid, and sorting into different grades of fineness. The carborundum, as taken from the furnace, consists of various-sized aggregates of crystals. It is taken to a crusher, which consists of a large iron pan rotated horizontally by means of a vertical shaft. Attached to a collar surrounding this vertical shaft is a horizontal shaft, which carries two heavy iron rollers; the grinding action is aided by water placed in the pan with the carborundum. The ground mass is then transferred to large lead-lined wooden tanks, where it is treated with dilute sulphuric acid (1:2) for three days, the acid being heated by steam to 100° C. The iron and alumina are the chief impurities removed by this washing. 10,000 lb. of carborundum can be washed in each tank at a time. After leaving the tanks the carbide is thoroughly washed with water in a large rectangular tank. The finer portions, which are washed away by the water, are separately collected, whilst the remainder are thrown on to a drainer and subsequently dried on the outside of a small kiln. This portion of the product is finally graded through a system of screens. The finer powder washed away by the water is collected and dried in a similar manner and is known as "flours"; it is chiefly used for making hones and for polishing glass. "Hand-washed powders" are made by stirring up a quantity of ungraded powder with water, allowing this to settle for a certain time, and then pouring off the supernatant liquid. "10-minute powder," for instance, is one which separates from the liquid poured off from the original powder after it has been stirred up with water and allowed to settle for 10 minutes—*i.e.*, a powder which remains in suspension for 10 minutes.

The Composition and Properties of Carborundum.—The carborundum as taken from the furnace and subsequently washed, still retains small quantities of iron and alumina, to which the varying colours, yellow-green and blue-green, are due. After treatment with acids and alkalis, heating in a current of oxygen to remove uncombined carbon, and finally treating with hydrofluoric acid, the carbide is obtained practically pure, as shown by analysis:—

Analyses of Carbide of Silicon.

—	Carborundum, Commercial Product.	Carborundum after Treatment with Acids, &c.	Amorphous Carbide.	Theory for SiC.
Si	62·70	69·10	65·42	70·00
C	36·26	30·20	27·93	30·00
Al_2O_3 / Fe_2O_3	0·93	0·49	5·09	..
CaO	..	0·15	0·33	..
MgO	0·11	..	0·21	..
	100·00	99·94	98·98	100·00

A purer carbide of silicon has been prepared by Moissan, who obtained it in several ways. He found that the temperature of a wind-furnace, 1,200°—1,400° C., was sufficient to produce well-defined crystals from carbon and silicon, the latter acting as a solvent and the excess being subsequently dissolved away. By heating the elements in the electric furnace, yellow and occasionally sapphire blue, crystals were got, many of them quite transparent, whilst by bringing the vapours of carbon and silicon together in a special form of furnace, beautiful almost colourless prismatic needles were formed. The following analytical results were obtained by Moissan (Compt. Rend. 1893, **114**, 1089):—

—	1.	2.	Theory for SiC.
C	69·70	69·85	70·00
Si	30·00	29·80	30·00

The carbon was determined by combustion with chromate of lead, the silicon by fusion with a mixture of potassium nitrate and carbonate.

Crystalline carbide of silicon has a specific gravity of 3·12. The faces of the crystals are usually smooth, with a brilliant adamantine lustre. The crystalline form is hexagonal, with the basal pinacoid predominant; exact measurements of the crystals have been made by Prof. B. W. Frazier, of the Lehigh University, who has confirmed the hexagonal symmetry of the crystals by optical examination.

The carbide is infusible and practically unaltered by prolonged ignition; heated to bright redness in a current of oxygen for one hour, the total loss in weight was only 0·41 per cent. Neither boiling hydrochloric nor sulphuric acid have any effect; even hydrofluoric acid alone or mixed with nitric acid does not attack it in the least. Sulphur is without action at 1,000° C. Chlorine at 600° C. acts very slightly, but at 1,200° C. complete decomposition occurs. When fused with caustic alkalis, the carbide is decomposed, silicate and carbonate of the alkali being formed. Lead chromate at a high temperature effects complete oxidation. The most valuable property of silicon carbide is, of course, its hardness. This lies between 9 and 10 in the ordinary scale, between the sapphire and the diamond, and certainly nearer 10 than 9, because the diamond itself can be undoubtedly polished by carborundum powder. This approach to the purest form of carbon by a combination of two elements so closely allied as carbon and silicon, neighbours in the periodic law, is exceptionally interesting. The synthesis of the diamond has only proved possible by allowing melted carbon to cool under increased pressure, under the ordinary pressure graphite always results; (Moissan). It appears, therefore, that the energy required for the formation of what is probably the more complex molecule is decreased when a portion of the carbon is replaced by a dissimilar element. This is not a function of the atomic weight, for carbon boride which has been obtained by Moissan (Compt. Rend. **117**, 423), appears to be even more stable and harder than carbide of silicon; it forms brilliant black crystals, has a density of 2·51, and the composition B_6C. Boron, like silicon, adjoins carbon in the periodic system of the elements. The properties and conditions of formation of a boride of silicon will therefore be of very considerable interest. In the carbon group of the elements the stability of the carbides decreases with an increase in atomic weight. Titanium carbide, TiC, which is not attacked by water at 700°, takes fire when heated to redness in oxygen; zirconium carbide, ZrC, is not decomposed by water at 100° C., but is readily attacked by hot concentrated nitric acid; whilst thorium carbide, ThC_2, is decomposed by cold water, forming a mixture of acetylene, methane, ethylene, and hydrogen. Such considerations show, as Moissan has pointed out, that these carbides may play an important *rôle* in the classification of the elements.

Uses of Carborundum.—The practical value of carborundum is essentially as an abrasive, either in the form of powder or as wheels. For the latter the carbide is mixed in suitable proportions with clay and felspar, the mixture moistened, pressed into moulds, and then dried in kilns. Wheels from 36 ins. diameter down to the small dental wheels are thus prepared, various grades of carborundum being chosen according to the requirements of the work. For fine hones, rub-stones, and the like, other binding material is employed, whilst papers are prepared similarly to emery paper.

In these various forms carborundum is employed in the iron and steel industries, in the manufacture of pottery and

porcelain for smoothing up the "biscuit ware" and for polishing granite. It is stated to be specially good for brass valve grinding, and is extensively used in the form of paper in the United States by shoe manufacturers for finishing the soles of shoes. The experiments made on the use of carborundum for polishing plate-glass have not been entirely successful, as, owing to its extreme hardness, the glass is scratched; but I am informed that a new method for polishing plate-glass allows of its advantageous use. A very interesting application is the use of barrel-shaped wheels for finishing hides and putting on the "nap."

In all these applications carborundum has to compete with emery, and for it to replace the latter its higher price—it is from two to five times as dear—necessitates very marked advantages. These advantages appear to be twofold. On the one hand it can work more quickly, and on the other it can do more work. Very many comparative tests have been made, and I believe I am well within the margin of possible error in saying that carborundum can do twice the amount of work of emery with a saving of 25 per cent. of the labour. In addition, in many instances, the work done is better.

In each application, however, the absolute advantages can only be judged by individual experience; so far, a continually increasing demand for carborundum has proved beyond a doubt that such advantages exist.

Uses other than mechanical may also arise; for instance, attempts have already been made to employ carborundum in the manufacture of ferro-silicon, but I have not heard with what success.

Our knowledge of the carbides of the metals is still in its infancy, but I believe there is hardly an instance in which a laboratory curiosity has risen so rapidly to an important product of manufacture as has been the case with carborundum. Carbide of silicon is only six years old and the accompanying figures showing the increase in the production of carborundum and the decrease in cost, the latter resulting entirely from improved conditions of manufacture, are ample evidence that Mr. Acheson's discovery has enriched chemical industry as well as chemical science.

Make of Carborundum.

	Lb.
1893	15,200
Six months ending June, 1896	549,036
" " December, 1896	658,783
" " June, 1897	741,297

Price of Carborundum.

	Per Lb. £ s. d.
March, 1892	2 0 0
August, 1892	0 16 0
September, 1892	0 8 0
1894	0 4 0
March, 1895	0 1 4
October, 1895	0 1 0
October, 1897	0 0 7½

Manchester Section.

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SESSION 1897-98.

Dec. 3rd:—
Dr. J. Grossmann. "Indigo Testing with Permanganate."
Dr. F. H. Bowman. "Steam, Gas, and Electricity as applied to Chemical Works."

Meeting held on Friday, November 5th, 1897.

CHAIRMAN'S ADDRESS.

BEFORE speaking to you on the subject I have chosen for this, our opening meeting of the Session, I have, with sorrow, to mention the loss this Section has sustained since we last met, by the death of its Vice-Chairman Mr. Peter Hart, and also, quite recently, by the death of Mr. Thos. Jackson, one of the old school of vitriol makers of Clayton. Both had been with us from the formation of the Section, and indeed had much to do with its establishment, but Peter Hart was better known amongst us as the contributor of many papers to the Society's Journal, and by the general and willing aid he always gave when appealed to for advice. Peter Hart was the link binding the old school of practical chemists with the new school in the Manchester district, and his death deprives us of the pleasure which we should have had in seeing him presiding over us—which I had hoped would have come about after my term of office was over. I am sure we all may express our sincere condolence with his family in their loss.

To turn now to the immediate subject of my address "Recent Developments in the Chlorine Industry." The theme I have chosen has been taken in consideration of the fact that all the world is waiting to see what will be the outcome of the various installations which have been set up to make chlorine, and therefrom bleaching powder, by means of the electrolysis of common salt.

As you are doubtless aware, the proposition is by no means a new one—Charles Watt, in 1851, obtained letters patent (No. 13755, 1851) for a process for the decomposition of saline and other substances and for separating their component parts from each other by means of electricity. His specification is more than usually complete and shows how carefully he had worked out detail. He used a vessel divided into two or more compartments separated from each other by means of partitions of porous materials; he employed a Daniell's constant battery and the cells were fitted with movable hoods to conduct away the gases to where they were to be utilised. His *modus operandi* in the case of the electrolysis of common salt was to keep the brine saturated by adding common salt from time to time and when carbonates were required in the place of caustic alkalis, a current of carbonic acid was passed into the compartments which contained the eliminated alkali.

A second process described in the specification was that of converting the chlorides of the alkalis into hypochlorites and chlorates of the bases. Here again he is very minute in his instructions, which goes to show that his specification was something more than a mere idea.

Watt's specification was filed nearly half a century ago, and we may pause to inquire why the consummation of his invention has taken so long a period. Watt lived before his day, the dynamo is but a machine of yesterday and it certainly was not practicable to contemplate the use of such batteries as Watt describes, for the production of, say, hundreds of tons of bleach per week. Then again, it is easy to say in the description of one's invention "separated from each other by partitions of porous materials"—but the real invention would have been the production of such a partition of practicable size—which has not been accomplished until quite recently, although the energy of the whole electrical world has been concentrated upon the problem for many years.

No one, I believe, has ever doubted the possibility of the production of chlorine and alkali by the electrolysis of the alkaline chlorides, but its success as a lasting commercial operation depends entirely upon the pecuniary result. In a paper of no very ancient date, Dr. Ludwig Mond expressed the opinion that in many attempted electrolyses the operation would be found more costly than the existing or modified pyro-chemical methods. This may be so in isolated cases, but I do not think that the electrolysis of common salt is one of them, as industrial electrolysis is much nearer consummation than it has ever been yet.

Some inventors have found so much difficulty in securing a suitable porous diaphragm that they have endeavoured to separate the two ions by other means and thus it comes

about that practical electro-chemists are split into two schools, those who use other means than the diaphragm for keeping the ions apart, and those whose energies have been mainly devoted to perfecting the diaphragm itself, for as a matter of fact, this difficulty having been overcome the rest of the work is fairly plain sailing. The cost of the mechanical energy and the transformation of the same into electrical energy is now so well established and understood for all possible conditions that no difficulty should arise in this connection, and the question of current efficiency in chemical work may now be said to have reached that point when only percentage improvements are possible.

Granted then that these quantities are known, is it not possible, you will say, to accurately forecast what will be the result of a new manufacture on these lines? I do not think we yet know sufficient to do so, as it is not only necessary to make a profit on the new manufacture at present, but the articles produced must be made at a less cost than by any known method. My impression is that the electrolysis of chlorides is more costly than the simple calculations would have us believe if we may judge by the comparatively slow development of the electrolytic method for the production of chlorate of potash. Had the electrical method been so much cheaper than the existing methods, there is no doubt it would have been much more extensively adopted, as the production of chlorate of potash by this means possesses manufacturing, physical, and monetary advantages which no one can well over-rate.

If we say that one horse-power on a large motor should not cost more than a farthing per hour, that only 10 per cent. of the mechanical energy is lost in transforming it into electrical energy and that the current efficiency is 90 per cent of the theoretical, then we may take it that, with any reasonable length of run with the diaphragm and with present selling prices, the result of the manufacturing operation should be a profit. The extent of that profit it is almost impossible to forecast, but I am very strongly of opinion that bleaching powder, the hydrochloric acid included, can be as cheaply made by existing processes as is possible by any electrolytic method.

The difference between the electro-decomposition of salt and its decomposition by sulphuric acid lies in the fact that in the former case two valuable products are formed, the alkali or its carbonate on the one hand and the chlorine on the other, while from its decomposition by sulphuric acid only one valuable product is produced, the chlorine—the resulting salt cake being actually a drag on the process seeing that it costs about 36s. to produce, and only realises 28s. in open market. If salt-cake should ever again rise to a remunerative figure, of course the position would be changed, but are there any hopes for such a change? I am afraid not, and the only salvation of the alkali trade is in the production of as little of it as possible. At the present moment at least two tons of salt cake are produced for every ton of bleach made by the manganese process which means a reduction of at least 36s. per ton from the revenue of the manufacture, whereas in the electrolytic method the by-product is a solution of caustic soda or its carbonate which adds something to the revenue.

As I have pointed out in a previous address to you, a solution of the foregoing evils has been attempted by the introduction of the Hasenclever improvements as an addendum to the Deacon chlorine process, but this is only a partial relief, as there is still the loss on 20 cwts. of salt cake per ton of bleach to be reckoned with, and, further, the nitric acid chlorine process which still further reduces the salt cake, has come to stay. In actual practice, this process turns out to be more easy of manipulation than was at first anticipated, but nevertheless it appears necessary to cast about for some chemical process by which the production of salt cake will be suppressed altogether. And why? Because the uses for salt cake are very limited, and should the price rise to make the manufacture *per se* a profitable one it would be brought into contact with ammonia soda which would be found to answer the consumer's purpose better, and the consumer would never revert to salt cake unless its price was substantially lowered again. Roughly, three parts of salt cake will only do the same chemical work in glass-making (now its principal use) as two parts of ammonia alkali, and as there is now a large quantity of ammonia soda selling in the States at 12 dols. per ton (2*l*. 10*s*.) the price of salt cake at 33*s*. 6*d*. per ton exceeds the economical ratio.

You will be saying, perhaps, what are these electrolytic processes and how near are they to financial success? The representative of the no-diaphragm school is the Castner Kellner Co. with its works at Weston Point, near Runcorn, and it is stated that the installation is decomposing from 45 to 50 tons of salt per week, but it is too early yet to speculate upon the financial success of the operation. The representative of the diaphragm school is the Hargreaves-Bird process which seems now to have established its *raison d'être*. A practical working cell capable of turning out one ton of bleaching powder per week and its equivalent of carbonate of soda is in operation at the company's works at Farnworth, but I am not able to give you any financial results, though it is very probable that a much more extensive installation will soon be completed. But as I have already pointed out, it will not be sufficient for these processes to demonstrate that it is possible to produce chlorine and alkali as a practical operation, or even to have a mean balance of revenue over expenditure. If they are to compete with the older processes, they will have to produce bleaching powder at less cost considerably than is being done now. As the markets now stand, the manganese process is scarcely in the running, though if the salt-cake difficulty should be overcome, it is a process difficult to beat with regard to cost.

The two processes which will compete closely with electrolysis, as far as bleaching powder is concerned, are first the Deacon-Hasenclever, and second the nitric acid chlorine processes, and we may now consider the cost price of bleach made by them both.

More than 50 years have passed away since Robert Oxland obtained letters patent for decomposing a mixture of hydrochloric acid gas and air by passing the mixture into a furnace filled with pieces of porous pumice-stone and kept at a bright red heat. Such a process, without the perfection of details as we know them to-day, could not possibly compete with the manganese process, and it is even now doubted by many whether, with the Hasenclever improvements, the method could hold its own with the Weldon process if salt cake were to rise in price.

The method now followed in the most recent works is to pass the hydrochloric acid gas (mixed with air) from two or more salt-cake pots, first through a drying tower down which sulphuric acid is made to flow, and then through a series of towers of peculiar construction, where the mixture comes in contact with heated cupreous material, and where decomposition takes place to a greater or less extent according to the chemical and physical conditions. The mixture of chlorine and hydrochloric acid is then washed with water, which dissolves out the hydrochloric acid, and the chlorine being dried by another sulphuric acid tower is passed into absorbing chambers of the shelf pattern. The liquid hydrochloric acid is then treated with sulphuric acid by the method described by Isham Baggs (British Specification 3269, 1866), mixed with air, and sent through decomposers, as was the pot acid. It must not be forgotten that it is only the pot acid that is decomposed directly; the roaster acid is condensed in water and treated with sulphuric acid to retain the water. It will thus be seen that the Deacon-Hasenclever process has nothing in simplicity of plant to recommend it; in fact, when compared with the Weldon process, it is a labyrinth of complications. Besides the ordinary vitriol and salt-cake plant, with the necessary muriatic towers, there is the vitriol-drying tower, the preliminary heater, the decomposing towers, the water-wash tower, and the vitriol-drying tower, before we come to the chambers where the chlorine is absorbed; then there are the Hasenclever cylinders and the decomposers relegated to them, with the pans required to concentrate the weak acid—a lively prospect when almost theoretical results are necessary to yield a profit.

An analysis of the operations necessary in this process do not impress one with its simplicity any more than the plant does. In a works making chlorine equivalent to 100 tons weekly of bleaching powder, certainly not less

than 150 tons of water have to be evaporated from the Hasenclever pans, and the whole of the water in the oil of vitriol used for salt-cake making is also collected in the vitriol from the drying tower, from which it has to be subsequently evaporated. I am not specially urging these facts as any great objection, but in coming later on to the cost you will be able to better appreciate the figures if a rough outline is given of the operations and their quantities. The 150 tons of water mentioned above, when added to oil of vitriol of 145° Tw., will produce nearly 1,200 tons of weak acid of 120° Tw. for evaporation weekly.

I think we may take it that 18 cwt. of salt are required to produce a ton of bleaching powder by the Deacon-Hasenclever process. It is true that short runs have been made at less than this, but, taking all in all, 18 cwt. will be found a very fair average, and this means the production of 20 cwt. of salt cake. Now, the salt cake made in the Deacon pots costs more to produce than ordinary salt cake, as the pots have to be humoured more according to the flow of gas required. It is nothing uncommon to produce 90 tons of salt cake per week in an ordinary pot and furnace, whereas with the one under consideration it is much less than this figure—probably between 40 tons and 50 tons, or perhaps even less.

From the latest statistics of the most modern works, I gather that the actual manufacturing cost of Hasenclever bleach for manufacturing wages (other than salt cake or O.V.) for lime, for fuel, for cupreous material, for casks, for loss of sulphuric acid in the process, for repairs which are very heavy, and for the loss on the manufacture of 20 cwts. of salt cake cannot be less than 4*l.* 6*s.* 9*d.* per ton. No allowance is here made for the general expenses of the establishment and no one but he who has gone through the mill can imagine how quickly but surely these tell up. There are the rent, rates, and taxes, miscellaneous labour that cannot well be charged directly, carting and waste deposit, forwarding charges, consul's certificates, telegrams, telephones, commission, sampling fees, gas and water, insurance both of works and material, office charges, laboratory expenses, law charges, auditing charges, expenses of management, estate charges, and last, and not least, there should be provided at least 5 per cent. interest on the capital invested, or the same amount as provision for those who have supplied the wherewithal. Taking all these items into consideration, I shall not be far out in considering that they will amount to twenty shillings per ton of bleach if bleach only is made, so that Deacon bleach should be sold at more than 5*l.* 6*s.* 9*d.* per ton before there is any chance of setting aside anything for depreciation. This is the cost of Deacon Hasenclever bleach, and it is this sum that the electrolysers will have to beat before their labours can be deemed successful.

With respect to the new comer—nitric acid bleach—I do not wish to enter into details at the present moment. It is my intention to offer you a paper upon the subject so soon as my term of office as your chairman is over, and I venture to think you will find the subject interesting. I can, however, tell you that a ton of nitric bleach can be made for much less than the 4*l.* 6*s.* 9*d.* mentioned in the foregoing example, including the loss on the manufacture of 16 cwts. of salt cake. Weldon bleach cannot be made for less than 4*l.* 16*s.* 7*d.* actual manufacturing charges, including loss on salt cake but without any charge for the general expenses of the establishment which would bring up the figure to 5*l.* 16*s.* 7*d.*

These figures show what a small margin is left to the maker of bleaching powder and the near future will show us how much more cheaply the electrolytic method can produce, if at all, but we must not lose sight of the fact that the two processes are not identical. While the electrolytic method is producing chlorine, it is at the same time liberating alkali. In the Castner-Kellner process it is worked as a caustic solution and made into caustic soda, while in the Hargreaves-Bird process it becomes carbonate of soda.

To show you the extent to which the electrolysis of salt is now carried on in England it may be received as correct, that the Castner-Kellner Company at Weston Point is turning out 70 tons of bleach and 30 tons of caustic soda weekly. The plant is being duplicated in order to produce 140 tons of bleach and 60 tons of caustic and all arrangements are made to follow on with another two sets which will bring the production to 280 tons of bleaching powder and 120 tons of caustic soda weekly by July next.

Before leaving this subject I would like to put before you the cost of bleach making by the Weldon process in 1875 at a works not many yards from where the electrolysis of salt is now being effected.

Memo. of the cost of making 2,480 tons of Bleaching Powder by Weldon's process from July 1st to December 31st, 1875.

	Per ton Bleach. £ s. d.		£ s. d.
Manganese, 70 per cent., 197 tons.	0 7 11	....	990 16 10
Freight on ditto at 1*s.* 4*d.*	0 0 1	....	13 2 8
Powder lime, 1,532 tons at 17*s.* 8*d.*	0 10 10	....	1,352 18 2
Pot lime, 1,480 tons at 17*s.*........	0 10 2	....	1,263 1 6
Limestone dust, 1,117 tons........	0 4 3	....	529 3 6
Slack, 3,224 tons at 6*s.* 2½*d.*........	0 8 1	....	1,000 15 8
Casks (hardwood) and cooperage wages........................	1 6 0	....	3,224 0 0
Process wages	1 3 3	....	2,880 19 11
Repairs wages	0 4 3	....	531 11 5
Materials for repairs	0 5 4	....	660 5 5
Sundries..........................	0 1 4	....	174 16 4
	5 1 6		

The figures show pretty well what was capable of being done 22 years ago, but these were not all the expenses. There were other charges over and above the foregoing and which amounted to no less than 17*s.* per ton of bleaching powder.

At this time (1875–76) salt cake which cost 46*s.* per ton to make was selling at 55*s.*, while bleaching powder stood at 7*l.* 10*s.*, and nitre cake, which in some localities can now be had for the asking, could not then be purchased under 35*s.* per ton There are two ways at least of looking at the present position of the alkali trade. The late Mr. Weldon, in a pamphlet published so late as 1883, said "The inevitable result of the rapidly increasing competition of the ammonia soda process must be that the English Le Blanc soda maker must either sell his bleaching powder at 7*l.* 16*s.* 5*d.* or close his works." Now the English Le Blanc maker of the present day does neither of these things. Why? Because not only have the manufacturing ratios been changed but economies have been realised which even Mr. Weldon declared impossible, and the prices of raw materials have been considerably cheapened. The position in 1883 when the Weldon pamphlet was written was thus stated: "To obtain the quantity of hydrochloric acid at present required for the production of a ton of bleaching powder one must manufacture 55 cwts. of sodium sulphate." The position to day is that one need not manufacture more than 44 cwts. even by the manganese process; 20 cwts. by the improved Deacon; or 16 cwts. by the nitric process.

By the Le Blanc process, the manufacture of vitriol salt cake and bleaching powder must be taken as meaning one manufacture and if the salt cake is sold rather than used, the selling price will enable one to readily discover the deficiency that accrues from the operations. One way to bring this deficiency into £ *s.* *d.* is to assume the production of so much liquid muriatic acid at the market price and to credit the salt-cake department with this amount. This method of reckoning out costs is more or less correct according to what is done with the products. If salt cake and liquid muriatic acid are sold we need not find fault with the process, though in many cases the cost of filling the carboys and consequent tear and wear are too often left out of consideration. At the present moment the average production of 30° Tw. muriatic acid is 36 carboys (10 gallons) per ton of 90 per cent. salt and its value 7½*d.* per carboy in buyers' packages.

The other way of reckoning is to take the difference between the manufacturing price and the selling price of salt cake and to add this to the manufacturing cost of the bleach; but both methods are unsatisfactory and tend to show the artificial state of the industry.

This is the present condition of things as found by electro-chemists on their entry into this field and moreover they find a depressed market—a market in which any extra quantity of bleach is disposed of with difficulty. Philanthropists will, however, be glad to know that the electrolytic works have already given to the consumer a substantial part of what might have been profit, as a keen passage of arms has been going on between them and the amalgamated companies of the old school and orders have been fought for with more zeal than discretion. How long this will go on no one can tell, but does it not recall to memory the old cartoon wherein the lawyer and the two litigants are dividing their property in an oyster. There may be some doubt as to whom the oyster will eventually fall, but there is no doubt at all as to who will be left with the shells.

For several years now, chlorine has been produced from the electrolysis of chloride of potassium, and bleach, as well as chlorate of potash and caustic potash, have come into the English market. In fact, electrolysis of this muriate threatens to completely overturn the caustic potash and perhaps the carbonate of potash industry. By the electrolysis of 98 per cent. muriates caustic potash of much greater purity than that made by the old process can now be obtained and at a price less than was formerly charged for the impure article.

As to the progress of electrolysis as a standing manufacturing process we must ever admit the possibility of improvement and it is possible that other chlorides than those of sodium and potassium may in time be made to produce a cheaper bleach. The metallic chlorides offer many advantages over the alkali chlorides as will probably be discovered ere long, and the competition for orders, especially in the home market will most probably give even electrolysers a taste of bad times similar to those which the Le Blanc alkali trade has passed through. No one now has any prescriptive right to the use of mercury and no one can claim sole proprietorship in the use of a diaphragm, and once the electrolytic fever has fairly set in we shall have new calls and modified methods of working by the score, and only those will benefit who manage to get a good round sum for their supposed inventions from credulous shareholders in search of a cent per cent dividend.

It is a great pity some of our old inventors and patentees are not with us now, a Weldon, a Gossage, a Deacon, or even a Tessié du Motay would have made the Patent Office fairly hum.

We have quite recently had a foretaste of the things that will happen, in the case of a newly devised electrolytic cell which was ushered into existence by a grand parade of viands and select after-dinner speeches, and there is no limit to the extent to which this process may be carried, but those will suffer least who weigh well all the advantages or otherwise of the newer forms of apparatus side by side with those of older pattern.

We may now turn to another phase of the question. How will the electrolysis of salt affect the soda industry? I have already suggested, though not stated in so many words, that the diaphragm method is not specially suited for the production of caustic soda, mainly on account of the short life of the diaphragm in caustic solutions of any reasonable strength. This fault is eliminated in the mercury method, so that, at present at least, we may infer that caustic soda will be made by the Castner-Kellner process, and carbonate of soda by that of Hargreaves and Bird. If caustic soda be a first product and of a greater value than carbonate there is no reason in converting it into a low-priced article while this fact may be a stimulus to the invention of those in charge of the diaphragm plants.

Le Blanc caustic soda is now being made in Widnes and elsewhere from salt cake, but the industry is slowly dying a natural death being only kept alive by reason of the abnormally low price of salt cake. Caustic soda is also being made by Löwig's process, viz., that of calcining carbonate of soda with ferric oxide, and it is further produced by the causticisation of ammonia soda. Of the three processes there is no doubt the third is the cheapest when properly performed, but can even it compete with the caustic soda from electrolysis, which is in reality a by-product? I have heard it stated on what should be good authority that caustic soda can be made by the electric method for less than 50s. per ton, but this, of course, will depend upon the selling price of bleach, and the knowledge of some such cost price, as this will not only stimulate the Le Blanc makers to exertion but even the ammonia soda makers will have to look to their laurels. Specially does this remark apply to the English makers for the markets of the world are no longer their special customers. Continental caustic and even ammonia soda has found its way into London as the continental makers produce more than is required for their home consumption, and America is now producing largely both ammonia soda and caustic soda prepared from it.

But even in the States ammonia soda and its products may suffer by competition with electric soda seeing that the Matheson Company hold the patent rights of the Castner-Kellner Company for that part of the world. This has not come about yet, though, seeing that the Michigan Alkali Company have at Wyandotte, near Detroit, one of the largest caustic soda works in the world in full operation, it will not be long before a trial of strength occurs between them. I ought to be careful to state that in all these probable changes there is much uncertainty; the selling prices of both bleach and caustic might not always be regulated by the ordinary laws of supply and demand, and should the large companies not be wholly satisfied with their insane policy of "war to the knife" the position would have to be discounted in the light of a probable coalition. Anyhow, I believe there is no bright outlook for anyone producing ammonia soda or any of its products. Once given caustic soda or its carbonate as a by-product, first soda crystals will suffer (always supposing the bleach to be the main object sought for and its manufacture *per se* profitable) and then bicarbonate, as there is no reason to suppose that so profitable a product as this will remain long unassailed.

We move rapidly now-a-days, if it takes but 10 years to make countries such as those of which Salisbury and Buluwayo are capitals we may expect a decade to see the rise and fall of many a new chemical industry. A year has scarcely passed since the first sod of the Michigan Alkali Company's works was cut, and to-day they are producing 100 tons of caustic soda weekly and the first installation will be quadrupled before the end of the present month. An output of 400 tons per week is not one to be despised, and will, I fear, play sad havoc amongst the manufacturers here.

Before concluding this address I would like to enter a protest against the statement that the manufacture of bleaching powder is a growing industry in England. It is no such thing judging from the number of factories out of use, some pulled down and others only working half time. I should say it is a declining industry, and every ounce that is made by any new process will have to be placed at the expense of an equal quantity made by older methods and consequently every ounce to be sold will be fought over with a zeal deserving of a better object. If I had to advise the investor I should say "don't" and seeing that chemicals, as a rule, are likely to be cheap, in fact below cost price generally, I ask would it not be better to seek investment in a consuming rather than a producing chemical business?

Newcastle Section.

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Saville Shaw, Durham College of Science, Newcastle-on-Tyne.

SESSION 1897–98.

Dec. 17th.—Exhibition of New or Improved Apparatus for Chemical Work.

Meeting held Thursday, November 18th, 1897.

PROF. HENRY LOUIS IN THE CHAIR.

FLOCCULATION AS AN AID IN LABORATORY WORK.

BY R. GREIG SMITH, M.SC.

The flocculation of clay and the sedimentation of mud have been treated by several writers in the last number of years, and it has been shown that many salts, bases, and even acids have the power of causing the coherence of small solid particles into aggregates, which comparatively quickly settle down in water. In the practice of agriculture, lime has long been used with success upon clay soils to increase the tilth. This it does by inducing the microscopic particles of clay to adhere closer together, and as a result, the soil water, which could only pass between the particles with difficulty, owing to the great friction produced, runs more freely through the channels between the aggregates; the soil becomes less plastic, drier, and better suited for the growth of crops.

This phenomenon has been shown (Proc. Chem. Soc. 1891, 176) to be practically identical with the coagulation of colloid bodies by electrolytes. In some excellent papers on "Solution and Pseudosolution" (Trans. Chem. Soc. 1892, 160; and 1895, 63), Messrs. Picton and Linder have demonstrated that all electrolytes have not the same coagulating power, and consequently, since coagulation is so closely allied to flocculation, it follows that some salts will act better as flocculating agents than others. From their experiments, Messrs. Picton and Linder conclude that the "tervalent metals have the highest coagulative power, bivalent metals about one-tenth of this power, and univalent metals, including hydrogen and ammonium, less than one five-hundredth"; ammonium salts are more powerful than potassium, and the latter are stronger than sodium.

In the discussion which followed the reading of the first paper, Mr. Herbert Jackson called attention to the fact that coagulation was similar to the phenomenon observed during the washing of many precipitates, such as lead hydrate and silver nitro-prusside, which furnish turbid solutions and refuse to settle when the liquid in which they are suspended approximates to pure water, but which flocculate when a drop of saline solution, as sodium acetate or potassium nitrate, is added.

The use of flocculating agents does not appear to be so generally known as it might be, for in the various methods of analysis one is sometimes advised to add this, that, or the other salt. The reason for so doing is sometimes given, but a choice of flocculating agents never is; and it is with the idea of altering this rule-of-thumb state of things that I have ventured to collect and bring before you a few examples of flocculation and of coagulation. At the same time, a few suggestions are made which I trust may be useful.

What is usually admitted to be one of the most troublesome substances to filter is barium sulphate precipitated from a solution of chlorides. It is frequently recommended to have the hydrochloric acid in considerable excess, and to boil the solution for some time. Instead of the hydrochloric acid being present in quantity, ammonium chloride may be used with precisely similar results; but surely it is only a little extension of the principle to suggest that ammonium chloride, or better, perhaps, ammonium nitrate, would be a good substitute for water in washing the precipitate on the filter. Since Messrs. Picton and Linder have shown that the precipitates carry down traces of the flocculating base which cannot be washed away by water, it follows that non-volatile salts cannot be employed where the precipitate has to be ignited and weighed, and consequently in such cases only salts of ammonium and the acids are permissible. But they have also shown that the firmly adhering base of the coagulant may be displaced by the mass action of other bases, and therefore, in cases where non-volatile salts have been used, the objectionable base may be eliminated by subsequent washing with solutions of acids or of ammonium salts. If precipitation, even, has taken place in presence of non-volatile bases, water is not sufficient for the elimination of the adhering traces of these, and recourse must then be had to solution and reprecipitation, or to the quicker washing with a volatile electrolyte. These fixations and replacements recall the fixation of bases by basic silicates and by double humates in the soil.

In the determination of phosphoric acid, the precipitated phosphomolybdate of ammonia is washed with diluted molybdate of ammonia solution, dilute nitric acid, or with a solution of ammonium nitrate, in order to prevent it passing through the filter paper. Of the three bodies, ammonium nitrate lends itself best for this purpose, since, owing to its neutral reaction, the yellow precipitate is sooner dissolved in ammonia, and, moreover, a smaller volume of filtrate is obtained. When, however, large amounts of iron are present, as in the nitric acid solution of soils or basic slags, the ammonium nitrate should be acidified with nitric acid, and it is well to have a little ammonium citrate dissolved in the dilute ammonia subsequently used in dissolving the phosphomolybdate. The addition of ammonium nitrate to the stock solution of molybdate, to the extent of 10 per cent., not only assists the precipitation of the ammonium phosphomolybdate, but also prevents the precipitation of yellow molybdenum trioxide, should the reagent be kept for any length of time.

Silver chloride is generally precipitated in presence of an excess of free nitric acid, which causes the precipitate to collect in large flocks, and any tendency which these may have to break down and pass through the filter paper can be checked by using water acidified with nitric acid for washing instead of ordinary distilled water. In the volumetric estimation of chlorine by direct precipitation, a few cubic centimetres of a solution of aluminium nitrate will be found to be more useful than nitric acid, since it causes a better coagulation, the flocks are denser, and the fluid becomes clearer, so that the end of the reaction is more easily seen.

There is rather an anomalous case in the estimation of manganese by precipitation with permanganate. Zinc salts are added and the liquid kept hot to "regulate" the precipitation of the dioxide. It seems to be a case of coagulation or flocculation, since iron salts may be substituted, but, curiously enough, neither aluminium nor barium salts are said to have the same desirable effect.

In the separation of nickel and cobalt qualitatively, by means of potassium ferricyanide and ammonia, the operation is greatly facilitated by the addition of an excess of ammonium chloride to the slightly acid solution of the two metals. The cobalt ferricyanide becomes much denser and does not run through the filter.

In the extraction of chlorides and nitrates from large quantities of soil, the exhaustion, with a dilute solution of a suitable flocculating agent, such as potassium sulphate or lime water, will not only counteract any tendency which the clay particles may have to pass into the extract, but also, by maintaining the flocculated condition, enable the extraction to be quicker and better made.

The appearances of small quantities of calcium oxalate, when precipitated in the presence and in the absence of excess of ammonium salts, are sufficient to show that these flocculating agents are decidedly advantageous, and should they be also used in the washing, the floccules will be maintained, and there will be little tendency for the oxalate to pass through the filter paper.

Thus far in these examples the flocculating or coagulating agents have been used in neutral or in acid solution. A considerable number of our precipitates, however, are effected in presence of alkaline hydrates, or of salts with an alkaline reaction. The hydrates or basic salts of the metals and the earths act as flocculating agents, while, on the other hand, the hydrates of potassium, sodium, and ammonium appear to prevent flocculation to a greater or less degree. Fortunately the metallic hydrates or basic salts, which are the precipitates generally obtained by the action of the stronger alkalis, are gelatinous, and to a certain extent self-contractile, especially in the heat.

The effect of alkalis is perhaps best seen in their action on soils, or on clay particles suspended in water. The

alkalis, alkaline carbonates, phosphates, and oxalates prevent sedimentation, while lime, as a type of the earthy hydrates, is one of the best flocculents which we have. The action of the neutral phosphates and oxalates of the alkalis in preventing flocculation can be explained by the production of an alkaline solution through the double decomposition which will ensue between these and the lime, magnesia, &c. present in combination with basic silicates and double humates.

Only in one place in Messrs. Picton and Linder's papers can I find any mention of the action of alkalis on flocculation, and that is in connection with Congo red. In alkaline solution it was, in contrast to its behaviour in neutral and acid solution, found to be filterable through porous earthenware. Evidently in alkaline solution Congo red does not consist of such large molecular aggregates as in neutral or acid solutions, and consequently is not so easily coagulated.

Examples of coagulation by means of metallic hydrates are to be seen in the coagulation of albuminoids by copper hydrate (Stutzer's reagent), of colloid organic matter by basic lead acetate, and by basic aluminium and iron salts in the formation of lakes and precipitation of sewage. It is true that this coagulation may be explained by the formation of insoluble double compounds, and so be made to appear quite distinct from flocculation, but a sharp line cannot be drawn between the two phenomena. Once the coagulating hydrate is removed, the organic matter, unless it has been altered as with the albuminoids, generally becomes again soluble, just as when the flocculating agent is washed away the floccules are destroyed and an emulsion-like condition produced.

Organic matter contained in some alkaline solutions—as, for example, the aqueous extract of farmyard manure—can be readily removed by the addition of a few drops of a solution of aluminium sulphate. The organic matter in the almost black solution coagulates at once, and the clear colourless liquid can be readily filtered and washed, or the whole solution made up to some volume, filtered through a dry filter, and an aliquot portion taken for the estimation of chlorides or nitrates.

The power of the alkalis in preventing flocculation may be minimised by the simple process of taking care not to add an excess of alkali or by adding a considerable excess of some flocculating agent.

As an instance of the former case, take the precipitation of zinc as sulphide by the method of Jean Meunier, lately abstracted in the Journal of the Chemical Society. The author adds ammonia carefully till the precipitate is just redissolved, and passes sulphuretted hydrogen through the solution till precipitation is complete, as indicated by testing the solution with ferrous sulphate. "The precipitate is readily filtered and washed, especially if the liquids are warm. The precipitate is not interfered with by relatively large quantities of ammonium salts." In this process, a minimum amount of alkali is employed; there is no excess of ammonium sulphide—the sulphides and especially the polysulphides retard flocculation strongly—and there may be large quantities of ammonium salts. With regard to the latter, it may be said that ammonium salts certainly do not interfere with precipitation; they rather assist. That they do so can be shown by possibly one of the best examples of their action in alkaline solution.

A dilute solution of a nickel salt is taken, saturated with sulphuretted hydrogen, and treated with an excess of ammonium hydrate. The sulphide so produced is filtered with difficulty, and the filtrate is generally coloured. Should, however, half of the unfiltered liquid be treated with an excess of ammonium chloride, flocculation is soon apparent, filtration can be easily effected, and a clear filtrate obtained. The use of ammonium chloride in this way has been found exceedingly useful in the laboratory, when separating the metals of the iron group qualitatively.

But the action of ammonium salts, which appear to be the best flocculating agents in alkaline solution, can be shown in a more commonly occurring case of precipitation, *viz.*, that of ferric hydrate. A solution of ferric chloride, when treated with an excess of caustic potash in the cold, gives a gelatinous precipitate. Should an ammonium salt, as ammonium nitrate, have been previously added, the precipitated hydrate is clearly less gelatinous; it settles better, contracts more on warming, and filtration, in consequence, is much more rapid. In continuation of this idea, one finds that washing the precipitate with ammonium nitrate in some cases causes a faster filtration. There is no doubt, however, that when the precipitation is effected in the presence of the flocculating agent, the results are far superior to any subsequent attempt at flocculation. Although by the washing with ammonium nitrate, filtration may not always be faster than when water is employed, yet the precipitate is always sooner freed from adhering alkali. Salts of potassium and sodium, so far as the coagulation of ferric hydrate is concerned, do not seem to have much effect in presence of their hydrates. This is at variance with what has been observed in the volumetric estimation of sugar. In using a Fehling's solution made with caustic soda, the reduced cuprous oxide can be made denser, and the end reaction can be better seen by the addition of two or more grammes of potassium sulphate per test. In preparing the reagent, however, caustic potash is to be recommended in place of caustic soda for giving a more compact precipitate. The heating and the relative density of the solutions will probably account for the observed difference in the action of potassium salts upon the condensation of ferric hydrate and of cuprous oxide.

A further example of the action of ammonium salts in alkaline solution is seen in the reprecipitation of aluminium hydrate from an alkaline solution of potassium or sodium aluminate.

The use of 140 grms. of ammonium chloride in the litre of magnesia mixture not only assists the rapid precipitation of ammonium-magnesium phosphate, but also prevents the possible precipitation of magnesium hydrate, which sometimes occurs when the whole quantity (70 grms. per litre) is employed.

I trust that these few examples which I have brought before you may be the means of showing the advantages that are to be obtained in the laboratory by the use of flocculating or coagulating agents.

Nottingham Section.

UNIVERSITY COLLEGE, NOTTINGHAM.

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Hon. Local Secretary:

J. T. Wood, 29, Musters Road, West Bridgeford, Nottingham.

ON A PURE CULTIVATION OF A BACILLUS FERMENTING BRAN INFUSIONS.

(*This Journal, June* 1897, 510.)

ERRATUM.

The title of Fig. 1, col. 2, should read as follows:— "Cultivation of *a* in Glucose Gelatin."

Scottish Section.

SESSION 1897-98.

Meeting held on Tuesday, October 26th, 1897.

CHAIRMAN'S ADDRESS.

The Scottish Section of the Society was formed at a meeting held in Glasgow on the 8th of January 1884; it has, therefore, nearly completed its fourteenth year. After an infancy and childhood of exceptional vigour and promise, it is now about to pass into the periods of youth and manhood, and I trust that my successor in this Chair, 14 years hence, will have the satisfaction of recording an equally successful passage through these later periods.

It was largely due to the wisdom and foresight of the founders of the Section 14 years ago, that these early periods were passed through so safely and successfully, and that the Section is in such a position to day that we can look backwards with pride and satisfaction, and forwards with confidence and hope. But if we are in this privileged position through the wisdom and loyalty of the founders of the Section, it behoves us to remember our own duties and responsibilities in order that we may put forth our best efforts to provide for the growth and development of the Section through the coming years.

In setting ourselves to enquire how we may best forward the interests of the Society of Chemical Industry, it is not amiss that we should try to arrive at a clear idea of what are the actual and what the possible functions of the Society. In the Byelaws the objects for which the Society was established were stated to be :—

a. To advance applied chemistry in all its branches.

b. To promote the acquisition and practice of that species of knowledge which constitutes the profession of a chemical engineer.

c. To enable all persons interested in applied chemistry to meet and to correspond, to facilitate the interchange of ideas respecting improvements in the various branches of chemical manufacture, and to publish and communicate information on such subjects.

The first of these declared objects is sufficiently wide to contain the two which follow, which may be taken as suggestions more in detail as to how the "Advancement of Applied Chemistry" was to be secured. The wording of *b.* (the second object) suggests that it was at first intended that the Society should interest itself directly in the education and certification of Chemical Engineers, to do in fact for that profession what the Institute of Chemistry has done for professional chemists, or the Institute of Civil Engineers has done for the members of that profession. So far as I know, nothing was ever seriously attempted in this direction. The times were certainly not ripe for any such attempt when the Society was founded, and it is very doubtful if it is now so constituted that it could move in this direction without sacrificing much of the broad catholicity in its membership which I venture to think is one of its very best characteristics. It ought immensely to strengthen the claims of the Society to the respect and consideration of all public and official bodies, that it is in no sense a close professional or trade corporation, but that it exists solely for the benefit of all of the industries in which chemistry is concerned, and for the benefit, therefore, of the very large proportion of our population who are connected with these industries, either as capitalists, organisers, or wage-earners. It is only necessary to glance through the lists of presidents, members of council, and of local committees past and present, to realise how widely representative the Society is, and to feel assured that it would be next to impossible to use it for any narrow or selfish professional or trade objects.

The Society, having from the outset abandoned this idea of taking upon itself educational and diploma-granting functions, has to a large extent refrained from exercising any directing influence over the technical education movement of the past decade. At the present moment, when public attention is being so generally directed to the question of the commercial competition of other nations, and the important bearing on it of technical education, I believe there is no single body or corporation which has so much right to speak with authority on the subject as the Society of Chemical Industry with its 3,000 members, drawn from every branch of chemical industry, as well as from all of the leading universities, colleges, and laboratories. And yet we are compelled to confess that the Society as such has never made any adequate deliverance on this great subject, nor has the council in its representative capacity ever been consulted by the Government or by municipal authorities upon educational subjects. Surely there is here a noble opportunity for the Society to fulfil one of the express objects with which it was founded. The wise and thoughtful utterances of the past presidents and chairmen of Sections, show how much latent strength there is within the Society to deal with this subject if the collective wisdom and energy were judiciously led into definite channels.

There is ground for much satisfaction in the conspicuous success which has attended the attainment of the other objects set forth in the Byelaws. Those of us who have regularly attended the meetings, general and sectional, and who possess the 15 volumes of the Journal, which form so noble a record of work done, cannot fail to realise the splendid impulse which has been given to the more scientific study of the chemical industries, as well as the lasting benefits which have followed to the members themselves through the freer intercourse which the Society set itself to establish.

The "Advancement of Applied Chemistry in all its Branches" appears to include the proper collective representation of chemical industry in its relations to the legislature and to the executive government, Imperial or local. It is evident that the Council has kept this function of the Society before it, and in London, at least, slow, but certain progress is being made towards the attainment of official recognition. In the provinces, on the other hand, little or nothing has been accomplished in this direction, partly, no doubt, because the Sections have never set themselves seriously to work to obtain such recognition. It would be gratifying to us all if it were to become a natural and ordinary thing for the Lord Provost, and corporation of a city like Glasgow, to invite the Society to organise a discussion on any matter of public interest before taking any important steps in dealing with questions of industry or health, in which applied chemistry or physics take a prominent place. I do not, for a moment, mean to suggest that the Society should in such cases take the place of the paid professional adviser; but it is certain that there are many large questions which might be profitably discussed before they reached the stage at which they could be judiciously placed in the hands of the professional expert. In such cases a free discussion by men trained for the study of technical problems would certainly help to clear the way for final settlement.

To take another example, how gladly we should have received and complied with a request from the recent Petroleum Committee that the Society as a whole should discuss the great question of safe and unsafe oils, and should then present to the Committee a carefully prepared report embodying the results of these discussions in which both sides should be fairly and clearly stated and argued.

With such a report before it the Committee would have been in the position of the Lords of the Appeal Court, who do not themselves hear evidence, but have the evidence and arguments brought before them in a clear concentrated form, and with the personal element practically eliminated.

We want the public to realise that the members of this Society have enough public spirit to be willing to devote some of their skill and experience to the service of the community. Such service is accepted quite naturally, if a man proposes to represent a constituency in Parliamant or on the Town Council. We do not propose to sacrifice so much of our time as either the member of Parliament or the Town Councillor; but we are sincere in our offer of service and we think it will have a value.

Your Committee feel that the time has come when it will be advantageous to introduce certain changes in the arrangement of the meetings for the Session. Hitherto it has been the custom for the Section to meet monthly during the Session, either in Edinburgh or Glasgow. At these meetings a large number of interesting and valuable papers have been read; but only occasionally has it been possible to deal at all exhaustively with the subjects discussed, as it has seldom been possible to secure a full attendance of members sufficiently interested in the special branch of industry dealt with. There are many reasons for this, some of the members live out of town, and for those who live in town the pressure of evening engagements is such that meetings like ours are apt to give place to others of a more attractive kind.

For the forthcoming Session it is proposed to hold some of the meetings at centres of special industry, and to devote the whole afternoon or evening to the reading of papers and the discussion of matters likely to be of interest to manufacturers and their employees. If this scheme can be efficiently carried out it is believed that benefits both direct and indirect will accrue. I believe that a great deal too much has been made of the supposed unwillingness of manufacturers to take part in such discussions, or to allow their employees to do so in case they may be expected to disclose the special secrets or the dearly bought experience of their manufacture. As a manufacturer who has had considerable experience in working new processes, both patented and unpatented, I have, as might be expected, the utmost sympathy with the right and natural desire to conserve the results of brains and experience for the primary use of their possessors, but I can personally testify to the great help which my own work has received from the stimulus of meeting workers in departments other than my own, as well as from the unique stores of information contained in the Journal. Having been the recipient of these benefits it has appeared to me a duty to try from time to time to give something of my own work and experience, on the chance that it might perhaps be helpful to some other member. To those who feel that any connection of themselves or their employees with the Society might endanger their special trade secrets, I should like to say that the Society does not want yours or any man's secrets, but it does urgently want you to join in the intelligent discussion of the scientific principles which underlie each and every manufacture, and it can promise you that if you give to the common fund some of your wisdom and experience you will receive in return the powerful stimulus of criticism, which may lead you to do your own work better. And further, that by contact with the active minds in other branches of industry your own mental horizon will be widened, and you may find that principles which have been worked out in some widely different branch of industry have a valuable application to your own special work.

It is one of the most important functions of the Society to aid in opening up cross tracks between the various departments of chemical industry. Some manufacture may be languishing for want of a new raw material, another may be at a standstill for want of some special kind of plant, which, all unknown to the manufacturer, is perhaps in regular use in another department of industry. The Society ought to be the instrument for bringing these interests into touch.

What the Committee most desire is, that the manufacturers and employees connected with special industries should be willing to utilise the Society by bringing up at its meetings or by correspondence, problems and difficulties which may be limiting or injuring their industry. These difficulties need not necessarily be of a purely technical or chemical kind they may be legislative or fiscal, or commercial. As I reminded you a little while ago, the first and chief object of the Society is "To Advance Applied Chemistry in all its Branches." Surely that is wide enough to cover every possible case that we may have to deal with.

There is a feeling in the Committee that something should be done to give the meetings more of a social character so that the members may have some opportunity for conversation. This would have many advantages, not the least of which would be, that the older members might take the opportunity of becoming acquainted with the younger men who have still their position to make. This would be good for both classes. The young men are on the outlook for new spheres of usefulness, and it is to the older men they must look to provide them with these. The older men want young hands and active brains to help them, and it ought to be worth their while to keep in touch with the coming men. If the new scheme for holding meetings at various places in the country is found to be workable, one of its advantages will be that the meetings will necessarily be much more social.

In connection with this proposed new departure allow me to illustrate my meaning by a reference to a trade with which you are all more or less familiar, I refer to the shale oil industry.

The history of this trade for nearly 20 years has been one long struggle against the results of excessive competition from abroad. No sooner has an improved yield or a reduced cost been evolved to meet a decline in selling price, than the trade has been faced by a further decline, and the hoped-for margin of profit has again disappeared. No one who knows the history of the industry can doubt that a splendid battle has been fought against very heavy odds. But the very fact of the great technical strength *within* the industry has perhaps kept it from gaining all the advantage which might have been derived from the resources of some other branches of industry, either through the adoption of entirely new processes or apparatus, or through the finding of new outlets for the products of the industry. The time appears to have arrived when, by a full presentation of the circumstances of the industry, such as can be made through the Society's meetings and Journal, opportunity may be given for some entirely new departure which may save a most important industry from the danger of extinction. In view of the great importance of the oil industry in Scotland, the Committee of the Section have arranged to hold the second meeting of the Session at Broxburn, when various subjects will be presented for discussion. In order to clear the way for a discussion of the special points which are to be brought before that meeting, it appeared desirable that a portion of the address at this opening meeting should be devoted to a general survey of the technical and commercial position of the shale industry. It is hoped that this general sketch may be the means of arousing such an interest in the industry as will ensure the presence and help at the Broxburn meeting of members, whose talents and abilities have not yet been directed to the problems of the shale industry. It is further hoped that the Broxburn meeting may be the means of introducing to the notice of technologists who are not familiar with them, the wide range of products which are so plentifully and cheaply produced in this industry.

During the past 14 years, papers dealing with various features of shale oil manufacture have from time to time been read before the Section, and printed in the Society's Journal, and in his opening address in 1894 Mr. Robert Irvine described certain new developments in connection with the visit of the Society to Broxburn Works, but no attempt has been made to give any general resumé of the progress all along the line in this interesting industry. It is quite natural that papers dealing with special inventions or details should treat of their subjects at so close a range that the resulting pictures are more or less "out of scale" when viewed in connection with the larger interests of which they only

form a part, so that it would seem as if an occasional corrective ought to be applied in the shape of a general survey of the whole subject from a broader point of view. On more than one occasion I have advocated the cultivation of such general surveys; but that it is one thing to preach and quite another thing to practice, I soon found out while preparing the following sketch, for the subject grew larger and larger while the time for preparation grew shorter; I feel impelled therefore to ask your indulgence in so far as my sketch has fallen short even of my own ideal.

THIRTY YEARS OF PROGRESS IN THE SHALE OIL INDUSTRY.

BY GEORGE BEILBY.

The consideration of this subject naturally begins with its raw material, the shale, but as the shales of Mid and West Lothian have been fully described by Mr Robert T. Moore, C.E., in a paper read before the Federated Institution of Mining Engineers in 1892, it is only necessary for our present purpose to refer to this part of the subject in very general terms. These shales occur below the coal measures, and taking them in their order from above downwards they are known as Fells, the Broxburn, the Dunnet, the Barracks, and the Pumpherston seams. Broadly speaking the working of these seams in point of time has followed the same order. The Addiewell Works were put up over 30 years ago to distil the Fells seam, Uphall, Oakbank, and Dalmeny followed up with the Broxburn seams. Ten years later the Broxburn Works came in with its splendid field of the same seams, and five years later the Pumpherston and Holmes Works took up the Pumpherston shales. The Dunnet and Barracks seams came into general use at about the same time as the Pumpherston. Fells seams yielded about 35 galls. of crude oil per ton, Broxburn seams about 32 galls., Dunnet seams about 25 galls., and Pumpherston about 20 galls. The lower seams, as a rule, yield a larger percentage of paraffin wax, and more ammonia than those above them, and, occurring as they do in thicker beds, they can be worked more cheaply, so that the difference in oil yield does not correctly represent the difference in their value. Thirty years ago the chief supplies were drawn from the Fells and Broxburn seams, while to-day the Pumpherston and Dunnet are at least equally important sources. These changes in the character of the shales worked during the past 30 years are so important that they must not be lost sight of in what follows:—

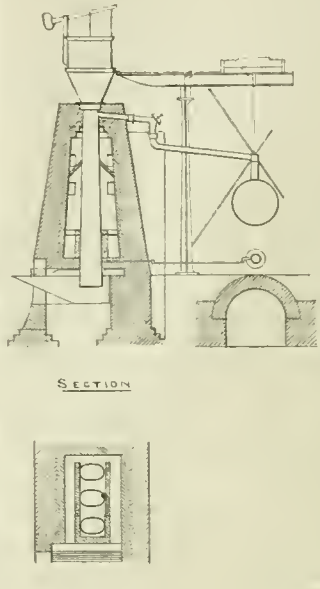
Retorting or Distilling the Shale.

When I joined the Oakbank Oil Company in 1869 a fierce controversy raged between the respective upholders of "Vertical and Horizontal Retorts," and I well remember my initiation into the mysteries of the subject by my early instructor, Mr. John Galletly, who, as chemist to Mr. James Young, founder of the industry, had acquired an immense fund of experience both valuable and unique.

The vertical retort was a cast-iron tube of oval or circular cross section, built vertically in an oven of brick work. At the top of the tube was fixed a charging hopper closed by a bell valve, while the bottom end dipped a few inches under water, contained in a shallow iron tray or pan. Every hour or so a small quantity of "spent shale" was raked out from the bottom through the water in the tray which served the double purpose of closing the retort and quenching the "spent shale." After every draw a corresponding quantity of fresh shale was introduced through the bell valve in the hopper so that the heated portion of the retort was kept filled with shale. The volatile products were removed by an exit pipe at the upper part of the retort. Fig. 1 shows this retort in vertical cross section.

The horizontal retort was a tube of cast iron of oval rectangular or D cross section closed at one end by an iron door and provided at the other end with an exit pipe through which the vapours and gases were led to condensers. These tubes were set horizontally in brick ovens heated by coal fires, in some cases the setting was similar to that of the ordinary gas retorts; but Fig. 2 shows a form of setting which was commonly used at the period referred to. They were usually filled with shale through the end door like gas retorts, but sometimes a charging hopper was provided on the top side of the retort; after being filled with shale the doors were luted with fireclay and screwed up tightly. The volatile products were driven off in from 16 to 24 hours, after which the "spent shale" was removed by the end door and a fresh charge was introduced. These horizontal retorts were called intermittent, and the vertical retorts continous working. Steam was regularly introduced into the lower end of the vertical retorts to help to sweep the oil vapours upwards to the exit pipe, but was seldom or never used in horizontal retorts. A great variety of both forms were to be found at work in the year 1869, each form had its partisans who believed that the true wisdom was only to be found in their particular retort benches.

Fig. 1.

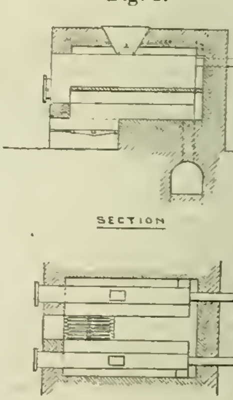


Old Vertical Retort.

In Oakbank Works, in 1869, the retort plant consisted of one bench of horizontal retorts and several benches of small vertical retorts of circular cross section. As these different retorts were worked side by side for several years, we had ample opportunity to test the one against the other. From shale of the Broxburn seam the horizontal bench yielded 24—25 galls. per ton of shale of a light clean crude oil, while the vertical benches yielded 32—33 galls. of a heavier and a darker oil, which lost more on refining than the horizontal oil, but yielded a larger percentage of paraffin wax, then, as now, the most valuable constituent of the crude oil. This, together with a larger yield of ammonia, turned the scale in favour of the vertical retort, and Oakbank became committed to that form.

The proper principles for setting and firing retorts had not yet been arrived at, and the waste of coals and of retorts was prodigious. The furnaces were relatively enormous, while the ovens in which the retorts stood were narrow and confined. The flame and hot gases from these powerful furnaces were led directly round the lower parts of the retorts, but to protect them from the keen action of the flame these parts were surrounded with fireclay covers moulded to the shape of the retorts. Coals were shovelled into the fires often at the rate of 6 cwts. for every ton of shale distilled, the combustion requiring a powerful chimney draught, which dragged large quantities of the powdery ash of the coal into the narrow channels surrounding the retorts, rapidly closing them as well as adhering to the hot surface of the retorts and forming a non-conducting coating to overcome, which involved more

Fig. 2.



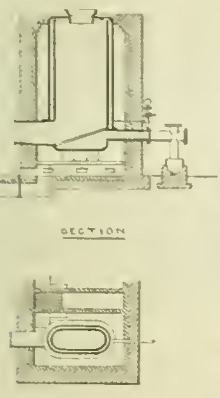
Old Horizontal Retort.

and more vigorous firing. Naturally the life of the retorts themselves was short, they generally bulged a little below the centre, and became hopelessly burned and cracked, sometimes even in 6, 9, or 12 months, especially if, at any time, the firemen had the run of a better class of coal than they had been accustomed to.

The crude oil, costing 4*d*. or more per gallon, suffered a loss of 40 per cent. on refining, and only contained about 7 per cent. of paraffin scale; an oil which to-day would be considered by any refiner too dear at a 1*d*. per gallon.

It was just at this time (1868–69) that Mr. William Young was busy re-discovering the virtues for paraffin making of that "low-red heat" which nearly 20 years before Mr. James Young had laid down as the proper temperature for the distillation of shale, but which the shale distiller had never been able to attain to practically. Mr. Young's 1868 retort, Fig. 3, was the outcome of these labours, and those of us who first saw and analysed the wonderful product of that gently-applied low-red heat, felt instinctively that a new era had dawned upon the shale distilling industry. But alas, for the complexity of the technical mind, instead of grasping the simple fact that the new retort was so excellent, mainly because the genuine low-red heat had at last been applied to the shale, thanks to the admirable setting and firing of the retort, we all wandered off into theories of "downward" and "upward" distillation, and a new controversy broke out which bid fair to be as prolonged and bitter as the horizontal *versus* vertical had been!

Fig. 3.



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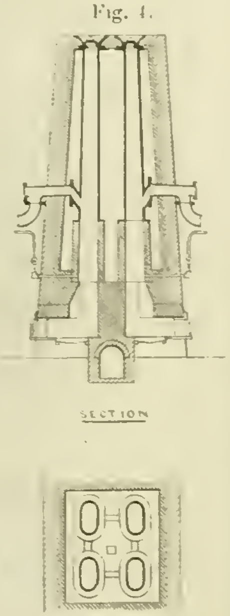
Young and Brash's Retort, 1867.

A bench of these retorts was erected at Oakbank in 1871, and continued to work for many years, producing an oil of fine quality; but a closer acquaintance with its practical working showed that the double casing, while adding considerably to the first cost, was also a source of weakness owing to the formation of carbon deposits between the outer and inner shells which prevented the free passage of heat to the shale and added to the consumption of fuel.

A few years previously, Macbeth had erected retorts in which the combustion of the shale in the lower parts supplied heat for the distillation in the upper parts of the retorts. This retort never advanced beyond the experimental stage.

Meantime, Mr. William Young was feeling round after a further advance in the same direction, by which the residual carbon of the "spent shale" was to supply the necessary fuel for the distillation. In 1872, the new retort, Fig. 4, was brought out (Eng. Pat. 2187, 1872), and we were fain to admire the ingenuity with which Mr. Young's "gas lute" separated the distilling zone of the retort from the fire chamber below, into which the spent shale passed for combustion! But this was too delicate an instrument to place in the hands of the unskilled labourers who work retorts. With skilled supervision it would do all that its inventor claimed for it; but unfortunately with benches containing hundreds of retorts the unskilled labourer had to be reckoned with, and often entailed much wreckage and misfortune before he became even moderately skilful in handling the retorts.

Fig. 4.



SECTION

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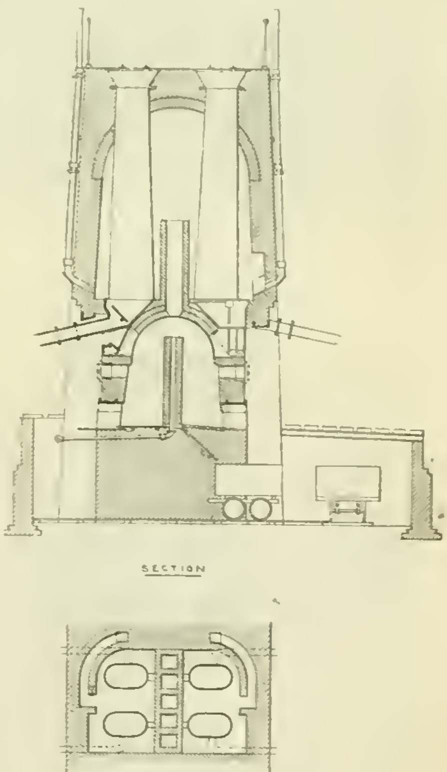
Young, Scott, and Stephens' Retort, 1872.

But Mr. Young had now shown us that the requisite uniform "low-red" heat could be obtained by the combustion of the residual carbon of the "spent shale" and that this heat could be perfectly well applied to the shale without the intervention of the costly double-casing of the 1867 retort.

The finishing touch was applied by Mr. N. M. Henderson in 1873 when, through his beautifully designed arrangement of retort and furnace chamber, the carbon of the spent shale was made available for supplying exactly the kind of heating required without making too many demands on the skill and attention of the labourer. This retort has been so often described (this Journal, 1889, 100), that it is only necessary to direct your attention to its general construction as shown in Fig. 5. (Eng. Pat. 1327, 1873.)

Fig. 5.



SECTION

PLAN

Henderson's Retort, 1873.

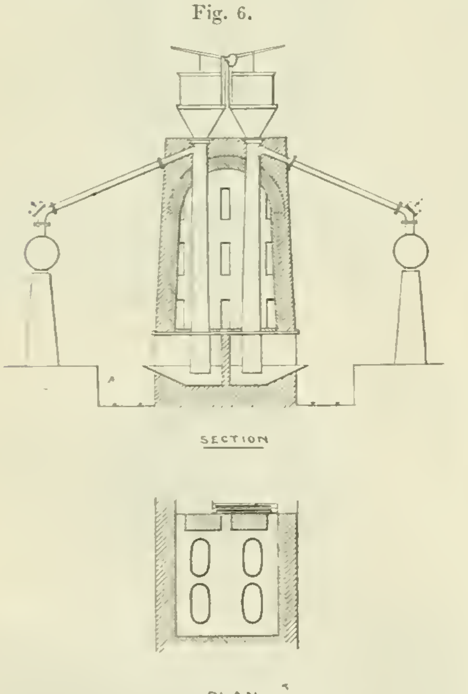
The first benches of Henderson's retort were erected in Oakbank Works in 1874, and these identical retorts continued to work without change or renewal for 12 years till 1886, when they were pulled down, not because they were worn out, but because the times had changed.

When the Broxburn Oil Company began its operations in 1878, Henderson's retorts alone were erected, and there can be no doubt that the brilliant success of that Company during its earlier years was largely due to their excellent qualities and perfect suitability to the shale of the Broxburn seam. Hitherto, retorts had been a perpetual source of weakness, and in the older works a large staff of bricklayers and fitters were continually at work repairing and renewing the retort benches, pulling down the worn out and rebuilding the new. Now, at last, it was felt that a kind of finality had been reached, and that the fortunate

manager who possessed these retorts would be able to leave them in charge of his subordinates, and devote his skill and energy to perfecting the other apparatus of the oil work.

The directors of the Oakbank Oil Company had not seen their way to pull down the vertical retorts and replace them by Henderson, so in 1878 we set to work to apply the best knowledge of the time to improve the conditions of the shale distillation in the vertical retorts. Mr. William Young's results had shown plainly that the moderate equal heating was best obtained by setting the retorts in large ovens, with as much free space round each retort as possible, these ovens being heated by furnaces burning under a very moderate draught. We therefore cleared the old settings of all superfluous flues and brickwork, reduced the size of the furnaces, and gave every inch of available space to the ovens in which the retorts stood. By reducing the amount of brickwork with which the retorts had been covered up, the available heating surface was considerably increased, so that, although the working temperature of the metal of the retorts was reduced to a very dull red, barely visible in daylight, the same through-put of shale was maintained, without any diminution in the yield of crude oil. The effect of this alteration on the quality of the crude oil obtained was very marked. Instead of the dark brown low-yielding oil of the old setting, we obtained a clean bright green oil, which contained 10 to 11 per cent. of paraffin scale, an improvement of fully 2 per cent., and valued at, at least, 20*d.* per ton of shale. The other products were improved in proportion. The coal consumption was reduced from 5 cwt. per ton of shale to $1\frac{1}{2}$ cwt., while the cost for repairs and renewals fell to a fraction of the former amount. Fig. 6 shows one of these improved

Fig. 6.



Oakbank Vertical Retort, 1880.

settings in vertical cross section. Retorts of this form, which were rebuilt in 1878, were practically as good as ever when they were pulled down in 1886. The chief drawback in these improved vertical retorts was that the yield of sulphate of ammonia was reduced from 15 lb. to about 12 lb., a loss in value equal to about $4\frac{1}{2}d.$ per ton of shale.

In the matter of yields and value of the products the Henderson retort had little, if any, advantage over the improved vertical, but in cost of working the advantage was quite decided. While the vertical retort was worked by the method of hourly withdrawals of a small portion of the charge, which was replaced by introducing a corresponding quantity of fresh shale through the hopper at the top, the Henderson was completely emptied and filled every 17 or 18 hours, and was practically left to itself for the whole of the intervening time. It is obvious that such a method of working can be carried out with fewer labourers and at a correspondingly lower cost. The firing by means of the carbon of the "spent shale" resulted in saving a portion of the coal used by the vertical retort.

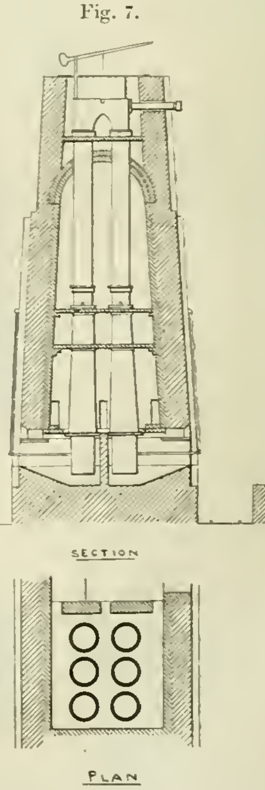
Between 1878 and 1880 Mr. McCutcheon at Addiewell, and Mr. Jones at Dalmeny, as well as other users of vertical retorts, also improved their settings so that the very low grade oil of the earlier years had become a thing of the past. Various attempts were made at Oakbank still further to improve the vertical retort. The diameter of the retort was increased, and a greater number of retorts were fired by a single fire, but the net result showed no improvement on those first erected.

Taking stock now of the position of retorting during the period 1878–83, we find that at Broxburn, Burntisland, and Linlithgow the Henderson retort was used exclusively, while at Addiewell and Uphall it had partially taken the place of the vertical retort. At Addiewell, Dalmeny, Oakbank, and Uphall there were still a number of more or less "improved" vertical retorts in use.

In 1880 the comparatively low yield of ammonia obtained from the improved vertical retort forced us to reconsider the whole subject of the ratio of the total nitrogen in the shale to the ammonia obtained, which I had first worked out in the years 1871–73. This matter was laid so fully before the Section in 1884 (this Journal, 1884, 216), that it will only be referred to now so far as is necessary to complete this historical sketch.

The leading idea of the new system which was simultaneously patented by Mr. W. Young and myself in 1881 was to subject the "spent shale" of the oil-making retort

Fig. 7.



Beilby's Retort, 1881.

to a further treatment with steam at a higher temperature than that suitable for oil-making, with the object of converting the residual nitrogen of the shale into ammonia.

The first retort for carrying out this double process was erected at Oakbank early in 1881. The drawing, Fig. 7, shows this retort in vertical cross section. It was intended to supply the answer to two vitally important questions, (1) was it possible economically to win an increased yield of ammonia by the application to the spent shale of a higher temperature? and (2) what influence would this supplementary treatment of the shale have upon the primary process of paraffin oil distillation which was to take place in the upper part of the apparatus?

The latter question had for us at that time a peculiar significance as we had only emerged from the "old vertical" period, and our recently acquired horror of high temperatures and local heating made us specially fearful as to the results to be expected from a retort which looked like the old vertical at its very worst. Fortunately these fears were groundless, and I shall not easily forget the delight with which we first saw a clean looking, bright green crude oil running from the condensers of the new retort, and when an assay of the oil showed that it contained to the full as much paraffin wax as the best existing crude oils while the liquor tank contained all the extra ammonia we had hoped to secure, we felt that our new system was to be the salvation of the trade! That the new system has indeed proved the salvation of the trade I suppose there can now be no doubt, but alas for the pleasant feeling of finality which we had begun to associate with the Henderson retort and its simple, almost automatic, distillation of our shale, it seemed as if we had again to begin the whole matter of retort construction afresh with certain new and formidable difficulties thrown in. It had taken years to arrive at the regulation of *one* satisfactory temperature for distillation, now we require to provide for *two*, and one of them much higher than anything we had had to deal with before. And this high temperature not only affected the retort itself but it also attacked and fused the shale ash which had been denuded of its carbon by the action of the steam. It is evident that the fusion of even a very small portion of shale occurring near the bottom of a long comparatively narrow tube could interfere most disastrously with the regularity of working and results.

The first benches of retorts under the new system were erected at Oakbank during the summer of 1881, and justified their existence by producing a doubled yield of ammonia along with an increased yield of paraffin scale. With careful supervision the working was quite regular and satisfactory, but so many points required to be watched especially during the education of the retortmen and the results of neglect or carelessness were so serious that this retort was not the final expression of the new system.

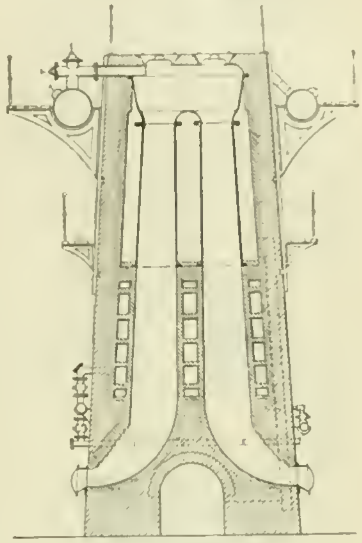
Meantime Mr. W. Young, after some preliminary trials, proceeded with the erection of some benches of retorts at Pentland Works of the form which has since become so well known as the Pentland or Young and Beilby retort. The drawing, Fig. 8, shows these retorts in vertical cross section. While still liable to the dangers and disadvantages which result from the employment of so wide a range of temperatures within the same apparatus, these were minimised by the larger size of the individual retorts by the easily accessible curved mouthpiece and door and by the use of a gas generator instead of an open fire. Large numbers of these retorts have been in constant and successful use since 1884 and it is an interesting fact that those benches of retorts which have been least altered or interfered with have continued to work most satisfactorily.

The monthly statement of the Scottish Mineral Oil Association for last April showed that the retorts in use in Scotland consisted of: Henderson, 896; Young and Beilby, 3,636; other kinds, 396.

No sooner were the general principles of this new system and its results published than numerous inventors set to work to improve upon the idea. It would be quite impossible, even if it were desirable, to detail all of these attempts, many of which were never carried out on a practical scale at all, but considerable interest attaches to a few of them.

Early in the course of our experiments, Mr. Young and I had tried the effect of mixing a proportion of air with the steam applied at the lower end of the retorts, with the object of burning a portion of the fixed carbon, so as to supply a part of the heat necessary to enable the remainder

Fig. 8.



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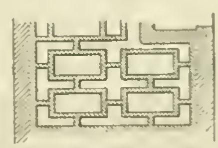


PLAN

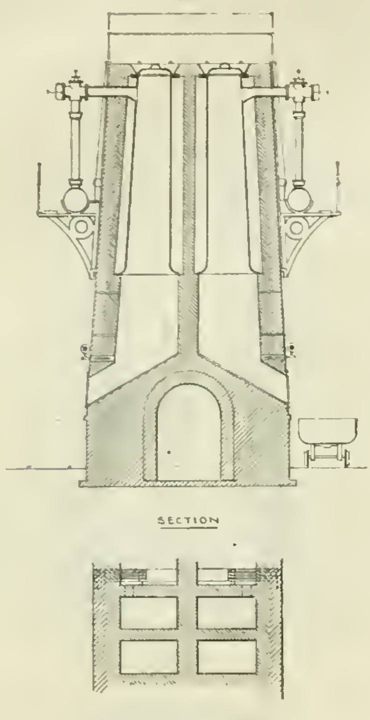
YOUNG AND BEILBY'S IRON AND BRICK RETORT.

of the carbon to decompose part of the steam, and to set free the fixed nitrogen as ammonia. The plan worked quite successfully so far, that is to say, the carbon of the "spent shale" was nearly all burned away, and a good yield of ammonia was obtained, although a lower heat was applied to the outside of the fireclay retort. As every volume of oxygen in the air took in with it four volumes of nitrogen, the quantity of air that could be economically employed was very small. Up to a certain point the extra volume of gas passing up through the red-hot spent shale acted usefully in carrying with it heat that was available for effecting the primary distillation of the oil from the shale in the upper part of the retort. It also helped to carry off, more rapidly, the oil vapours as they were formed, thus reducing the amount of secondary decomposition to which these vapours were subjected. But beyond this point the presence of the nitrogen was harmful, not beneficial. The volume of gas from a ton of shale had already been increased by the new system from 2,500 up to about 11,000 cb. ft. This increase had already rendered the condensation and scrubbing of the more volatile parts of the crude oil a matter of considerable difficulty, so that a further addition to the volume of gas through the admission of atmospheric nitrogen increased these difficulties, and was looked upon as not economical. Further, the addition of a quantity of inert nitrogen to the gas rendered it much less valuable as a fuel.

In 1884–85 an attempt was made, by means of what was known as the "Couper Rae retort," to effect the separation of the ammonia in the lower retort entirely by the agency of internal heat generated by a free mixture of atmospheric air with the steam introduced. These retorts shown in

Fig. 9. were built below as solid-walled combustion chambers entirely without flues or means of external

Fig. 9.



Couper and Rae's Retort, 1883.

heating. After a considerable amount of time and money had been spent in trying to get satisfactory results from them, these retorts were finally abandoned.

Later on, Messrs. A. Neilson and A. C. Thomson renewed the attempt to work with air combustion in the lower part of a retort of considerable size, built like a blast furnace; but I understand that the method was only successful with shales containing a large percentage of fixed carbon.

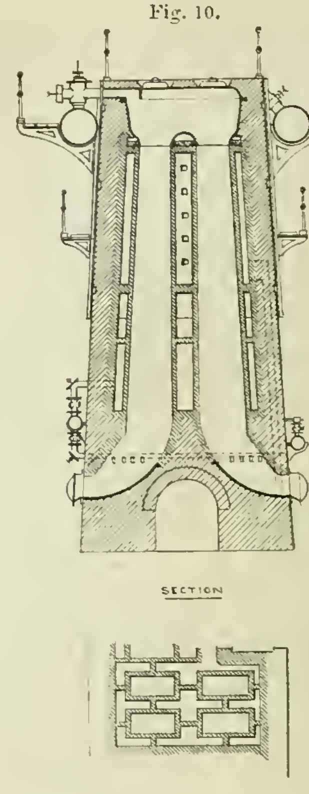
When it was found, in the case of the Pentland retorts, that the joint between the iron above and the firebrick below was troublesome and apt to leak, a number of benches were built (Fig. 10) in which the iron retorts were replaced by firebrick, that is to say, the firebrick retort was carried right up to the hopper, thus removing the principal joint between the iron and fireclay into a cooler region. These worked well until the pores of the bricks and the joints between them became loaded with carbon deposited by the oil, when the walls of the retorts swelled and cracked, becoming leaky at the joints.

In 1885 the Hermand Oil Company erected a bench of retorts at their Breich Works under licence from us. These, as shown in Fig. 11, were longer than the Pentland retorts, and differed from them further, in as far as each retort was provided with a separate hopper. They worked for several years and gave excellent results, the increased height being evidently an advantage, as it enabled the shale to be subjected to heat for a longer time. Subsequently the same Company erected several benches of retorts at the Hermand Works, in which the brickwork was carried up to the hoppers as in the Pentland retorts, already shown (Fig. 12). While these retorts began well, they soon became leaky and turned out inferior oil, thus confirming the experience which had been gained with the Pentland retorts.

All of the retorts referred to have been carefully drawn to scale, so as to show them in their true relative size.

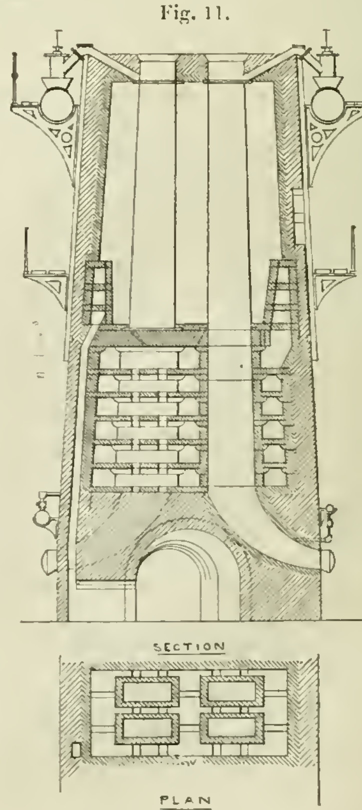
The experience of the past 16 years with the Young and Beilby system of distillation has shown that the original combination of iron and fireclay retorts is a practical

Fig. 10.



Pentland Retort, 1882.

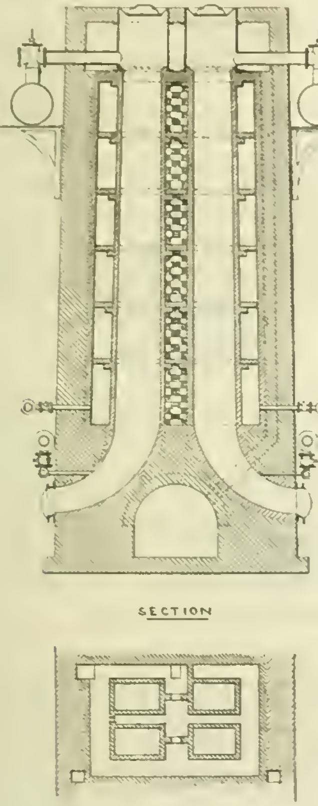
Fig. 11.



Breich Retort.

necessity, and that with proper care in its design and construction the combined retort can be maintained in good working order for years. It has also shown that the maintenance of regular heats on the upper and lower retorts is very difficult, except with a purely gaseous fuel, generated by the retorts themselves when the shale is suitable—otherwise by independent generators.

Fig. 12.



Hermand Retort, 1889.

The fluxing and clinkering of the shale ash is still the most serious difficulty in practical working, and during the past few years Mr. N. M. Henderson, Mr. Crichton, Messrs. Bryson, Fraser, and Jones, as well as some others, have designed and erected retorts in which the curved shoot of the Pentland retort is removed and replaced by various pieces of mechanism for the continuous withdrawal of the spent shale ash. It is undesirable that I should in any way anticipate what these gentlemen may have to tell us about their several inventions at the Broxburn meeting next month; we shall therefore leave the subject of retorting at this point.

Sulphate of Ammonia Manufacture.

Thirty years ago the method of dealing with the ammoniacal liquor from the retorts was crude in the extreme. The liquor was boiled off in ordinary egg-ended steam boilers, a very large proportion of the water having to be evaporated in order to exhaust the ammonia even moderately well. In working with a single boiler it was usual to feed slowly till the liquor was well up the glass, and then to shut off the feed and boil off till the liquor was down near the bottom of the glass. At this stage a portion of the spent liquor was blown off and the feeding started again. When several boilers were worked in series, the fresh liquor was fed into the first of the series and the spent liquor was blown off from the last.

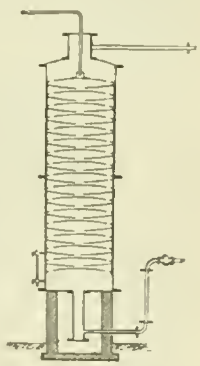
The shale liquor was very weak as compared with gas liquor, containing from 0·5 to 0·7 per cent. of ammonia, only a small proportion of which was present as fixed ammonia, so that lime was never used.

The general result of this system was far from satisfactory, the exhaustion of the liquor being, as a rule, very imperfect. It was not at all unusual for the spent-liquor still to contain a fifth of the whole ammonia.

Up till 1881 there was an impression that the Coffey still was not applicable to the liquor of the oil works; either the liquor was too dirty or too weak, or the quantity was too small to be economically dealt with by the large type of Coffey stills which had been designed for the spirit industry.

At Oakbank, in 1881, a series of experiments was carried out on the use of open steam in column stills, with the result that a simple form of still was designed and brought into practical use during that year. This column still is shown in section in Fig. 13. It was cheaply constructed, easily worked, and kept itself clean even when the liquor contained oil sludge. It dealt economically and effectively with even moderate quantities of liquor. This still seemed to meet the requirements of the time, for it was quickly adopted by a number of the oil companies.

Fig. 13.



Beilby's Ammonia Still, 1881.

Since that time the use of column stills has become so common in gas works that almost every maker of gas plant has his own pattern. Several of these designs are much more suitable for dealing with large quantities of liquor than the more simple form, whose interest is now chiefly historical.

The introduction of the column still effected a very great saving in fuel, while it reduced the loss of free ammonia in the spent liquor to a mere trifle.

The requirements of the Alkali Acts drew a good deal of attention to the "cracker boxes," in which the ammonia gas is converted into sulphate, and the necessity for working with close gas-tight boxes resulted in various improvements in their construction and working, which further cheapened the cost of producing sulphate.

This cost, which 30 years ago was sometimes as much as 6*l.* to 7*l.* per ton, has been reduced now to under 3*l.*

An important economy, first proposed by Mr. W. Young in 1880, was the returning of the waste steam from the cracker boxes into the retorts in place of a corresponding quantity of fresh steam. The practice has since become universal; the whole of the exhaust steam from engines and pumps, as well as from the cracker boxes, is taken into a common system of piping and led to the retorts, so that very little fresh steam is used. The saving thus effected cannot be less than 4*d.* per ton of shale.

Refining of the Crude Oil.

In the chemistry of oil refining there has been practically no change for the past 30 years. Oil of vitriol is still used for the removal of basic tars and resinous bodies, and caustic soda solution for the removal of the creosote tars and sulphur compounds. The different oils are separated by fractional distillation, and the paraffin by cooling, crystallisation, and filtration. While these general principles remain the same, the details of their application have been altered in the direction of increased precision and economy. Fuel, labour, and chemicals are all used more sparingly and with better effect. In one respect the refining of 1869 to 1876 was very imperfect; that was in the making of lubricating oils. Burning oils and paraffin wax of the finest quality were made in the works of Young's Paraffin Light Company more than 30 years ago, but the lubricating oils of 20 to 25 years ago left much to be desired. Neither in colour, in setting point nor in

viscosity were they at all up to the standards which have come into force since 1877.

The colour difficulty was overcome by distilling the heavy oils off caustic soda, the viscosity was raised by the plentiful use of superheated steam in the distillation, and the setting point was lowered by improved freezing machines and filter presses.

A great step forward was made when Mr. N. M. Henderson perfected his apparatus for the continuous distillation of the oils. This apparatus was fully described in 1889 by Mr. D. R. Steuart (this Journal, 1889, 100) in a paper read before this Section. The use of this system in Broxburn Works at once reduced the refining loss from 30 per cent. down to $26\frac{1}{2}$ per cent., the quantity of chemicals used being also reduced. The entire cost of refining fell from over 1*d.* per gallon down to $\frac{3}{4}$*d.* or less.

The cooling of the heavy oils and the extraction of paraffin scale received a great deal of attention. As a rule each work had its own methods and apparatus for carrying out these operations.

An apparatus erected at Oakbank in 1885, and described in papers read before this Section in the same year (this Journal, 1885, 321 and 653) has continued to work satisfactorily, and has been considerably extended on the original lines.

In paraffin-wax refining the "sweating" process, which was brought out by Price's Patent Candle Company, has almost completely superseded the old naphtha process, greatly to the advantage of economy and safety. In this process the crude paraffin scale is melted and cast into cakes. These cakes are placed on drainers in ovens heated by steam pipes to a temperature just a little short of the melting point of the scale. The low-melting portions of the scale are sweated out of the cake, taking with them the oil and colouring matter, and leaving a small, but a purer and harder cake. One or two repetitions of this process are sufficient to bring the colour and hardness of the wax up to the required standards.

This process has been improved by Mr. W. M. Henderson (this Journal, 1894, 1043), so that the cost of handling the wax is greatly reduced.

The general effect of all of the improvements of the past 30 years has been to reduce the cost of refining a gallon of crude oil from 2·2*d.* down to 0·7*d.*, the latter figure including the refining of the scale into wax—an operation which was formerly carried out separately and at great expense.

Costs and Profits.

Thirty years ago market prices were still so high that it was not necessary to scan the works cost sheets very narrowly; indeed, many companies did not even trouble to make out cost sheets at all. It is, therefore, not possible to speak of the average costs of that time with the precision and certainty of to-day, when, in every works, costs are made out to the $\frac{1}{100}$ of a penny.

Cost of Shale.

I have not the experience which would justify me in speaking of the various factors which influence the cost of shale; in what follows, therefore, I simply take it as a fact that in 1868–69, as well as in 1897, shale of such a standard quality as the Broxburn 30-gallon seam could, in the more favourably situated fields, be mined and delivered to the retorts for 5*s.* per ton; and further, that the lower shales of the Dunnet Barracks and Pumpherston seams of 20-gallon quality could be worked for 1*s.* less, or say 4*s.* per ton. While passing thus lightly over the subject of shale costs, it is necessary to guard against any misapprehension as to the vital importance of the shale supply in determining the profitable working of an oil works. In the past history of the oil companies far more has turned on the quality and cost of their shale than has appeared on the surface; and for the future it is clear to those who know the trade best, that it is this question which will determine which companies shall survive in the struggle for existence. With these qualifications we may take it that the costs of 1868–69 were approximately:—

		Per Ton. s. d.
Shale	say	5 0
Retorting	"	5 0
Refining	"	5 6
A total cost of	"	15 6

The produce obtained from a ton of shale was:—

Burning oil	12·0 gals.
Lubricating oils	3·9 "
Paraffin scale	2·1 "
Sulphate of ammonia	12·0 lb.

The net prices of these products in the works were:—

	d.
Burning oil	15·0 per gal.
Lubricating oil	12·0 "
Paraffin scale	30·0 "
Sulphate of ammonia (cost of making being deducted)	$\frac{3}{4}$ per lb.

Putting these figures into the form of a profit-and-loss statement we have:—

I.—Yields and Costs of 1869, at the Prices of 1869 (by Old Vertical Retorts), with a Thirty-Gallon Shale.

Costs per Ton.

	s. d.		d.
Shale	5 0	=	60
Retorting	5 1	=	61
Refining	5 6	=	66
			187

Yields per Ton.

		d.		d.	
Burning oil	12·0 gals. at	15	=	180	
Lubricating oil	3·9 "	12	=	47	
Paraffin scale	2·1 "	30	=	63	
Sulphate of ammonia, 12 lb. at		$\frac{3}{4}$	=	9	299
Gross profit per ton of shale					112

Subject to deduction of commercial charges and interest.

The rapid development of the petroleum industry of the United States caused the price of burning oil to fall steadily from year to year, and by 1877 it had reached $9\frac{1}{4}$*d.* per gallon, while lubricating oil was still in the region of 11*d.* to 1*s.* For those earlier years there was comparatively little lubricating oil or paraffin exported from America, but from 1877 onwards the exports went up rapidly. Five years later, in 1882, we find that burning oil had fallen to 4·3*d.*, lubricating to 7·5*d.*, and paraffin scale to 19·2*d.* per gallon, showing that the competition from America was telling now on all three products.

If we apply these prices to the costs and yields of 1869 we find:—

II.—Yields and Costs of 1869, at the Prices of 1882 (by Old Vertical Retorts), with a Thirty-Gallon Shale.

Costs per Ton.

	s. d.		d.
Shale	5 0	=	60
Retorting	5 1	=	61
Refining	5 6	=	66
			187

Yields per Ton.

		d.		d.	
Burning oil	12·0 gals. at	4·3	=	52	
Lubricating oil	3·9 "	7·5	=	29	
Paraffin scale	2·1 "	19·2	=	38	
Sulphate of ammonia, 12 lb. at		$1\frac{1}{2}$	=	18	137
Loss					50

Subject to addition of commercial and interest charges.

that instead of a profit of 9*s.* 4*d.* per ton there would have been a loss of 4*s.* 2*d.* But the improvements effected in retorting and refining had been sufficient to turn the scale on the right side; for the cost of retorting had been reduced

from 5s. 1d. to 2s. 6d. per ton, and the cost of refining from 5s. 6d. to 3s. 6d. per ton, making the total cost 11s., as compared with 15s. 7d. in 1869, while the yield of refined products obtained from 30 gallons of crude oil had been increased from 18 gallons up to 21·3.

III.—Yields and Costs of 1882, at the Prices of 1882 (by Henderson's Retorts), with a Thirty-Gallon Shale.

Costs per Ton.

	s.	d.		d.
Shale........................	5	0	=	60
Retorting....................	2	6	=	30
Refining	3	6	=	42
				132

Yields per Ton.

		d.		d.	
Burning oil.........	12·6 gals. at	4·3	=	54	
Lubricating oil.....	5·4 „	7·5	=	40	
Paraffin scale.......	3·3 „	19·2	=	63	
Sulphate of ammonia, 12 lb. at		$1\frac{1}{2}$	=	18	175
Gross profit per ton of shale					43

Subject to commercial charges and interest.

The profit-and-loss statement now shows a gross profit of 3s. 7d. per ton of shale.

In 1881–82 the introduction of the new system of retorting turned the attention of the trade to ammonia as a means of compensating for the falling price of oil products.

The price of sulphate of ammonia, which had been about 14l. per ton in 1869, had risen to 21l. in 1882, but the fall from this point was very rapid. The subjoined table, which was referred to by the Chief Inspector under the Alkali Acts in his report for 1896, shows the increase of production from 1882 to 1895 inclusive :—

Year.	Total Shale mined in the United Kingdom.	Total Sulphate of Ammonia recovered from the Shale.	Yield per Ton of Shale mined.	Price per Ton.
	Tons.	Tons.	Lb.	£ s.
1882	1,030,900	5,900	12	20 5
1883	1,167,900	6,400	12	16 10
1884	1,518,800	9,500	14	14 10
1885	1,770,400	16,200	20	11 15
1886	1,728,500	18,000	23	11 10
1887	1,411,000	21,100	..	12 5
1888	2,076,400	22,100	24	12 0
1889	2,014,000	24,000	26	12 5
1890	2,212,200	24,700	25	11 10
1891	2,361,100	26,000	25	11 0
1892	2,089,900	25,100	25	10 5
1893	1,956,500	28,500	32	12 10
1894	1,986,300	33,000	37	13 0
1895	2,212,000	38,300	39	10 0

Mr. Forbes Carpenter has pointed out that the quantity of shale in the second column does not exactly represent the quantity retorted, which on the average would be less than the quantity mined, as a certain quantity is used in gas works. The fifth column shows average yearly prices of sulphate per ton.

In 1887 the prices for oil products had dropped to the lowest recorded point. Burning oil was 3·2d., lubricating oil 2·2d., and paraffin scale 16·8d. per gal., while sulphate of ammonia had come down to 12l. 5s. per ton.

In 1897 we find that prices had fallen to 3·41d. for burning oil, 3·19d. for lubricating, 14d. for paraffin, and 8l. per ton for sulphate of ammonia. The diagram (Fig. 14) shows the course of prices for 18 years.

The prices for ammonia are taken per pound, less cost of manufacture.

A profit-and-loss statement, with the costs and yields of 1882 at the prices of 1897, shows a loss of 2s. 3d. per ton of shale :—

IV.—Yields and Costs of 1882, at the Prices of 1897 (by Henderson's Retorts), with a Thirty-Gallon Shale.

Costs per Ton.

	s.	d.		d.
Shale........................	5	0	=	60
Retorting	2	6		30
Refining......................	3	6	=	42
				132

Yields per Ton.

		d.		d.	
Burning oil	12·6 gals. at	3·41	=	43	
Lubricating oil	5·4 „	3·19	=	17	
Paraffin scale......	3·3 „	12·00	=	39	
Sulphate of ammonia, 12 lb. at		$\frac{1}{2}$	=	6	105
Loss........................					27

Subject to addition of commercial charges and interest.

But since 1882, costs have been reduced, so that retorting can now be done for 2s. per ton and refining at 0·75d. per gallon of crude or 1s. 11d. per ton of shale. Yields have been increased to 12·9 gals. of burning oil, 5·4 gals. of lubricating oil, 4·3 gals. of paraffin scale, 36 lb. of sulphate of ammonia.

A profit-and-loss statement, with these costs and yields, shows a gross profit of 2s. per ton of shale :—

V.—Yields and Costs of 1897 (by Young and Beilby Retorts), with a Thirty-Gallon Shale.

Costs per Ton.

	d.
Shale..	60
Retorting	24
Refining......................................	23
	107

Yields per Ton.

		d.		d.	
Burning oil........	12·9 gals. at	3·41	=	44	
Lubricating oil	5·4 „	3·19	=	17	
Paraffin scale	4·3 „	12·00	=	52	
Sulphate of ammonia, 36 lb. at		$\frac{1}{2}$	=	18	131
Gross profit per ton of shale					24

Subject to commercial and interest charges.

The sum of the balances from the tables (Nos. IV. and V.) is 4s. 3d., which represents the result of the improvements of the past 15 years, valued at the extremely low prices of 1897.

If we enlarge the period of comparison back to 1869, a statement of costs and yields of 1869, at the prices of 1897, shows a loss per ton of 8s. 6d. :—

VI.—Yields and Costs of 1869, at the Prices of 1897 (by Old Vertical Retorts), with a Thirty-Gallon Shale.

Costs per Ton.

	d.
Shale..	60
Retorting	61
Refining......................................	66
	187

Yields per Ton.

		d.		d.	
Burning oil........	12·0 gals. at	3·41	=	41	
Lubricating oil	3·9 „	3·19	=	12	
Paraffin scale	2·1 „	12·00	=	25	
Sulphate of ammonia, 14 lb. at		$\frac{1}{2}$	=	7	85
Loss per ton.................					102

Subject to addition of commercial and interest charges.

Fig. 14.

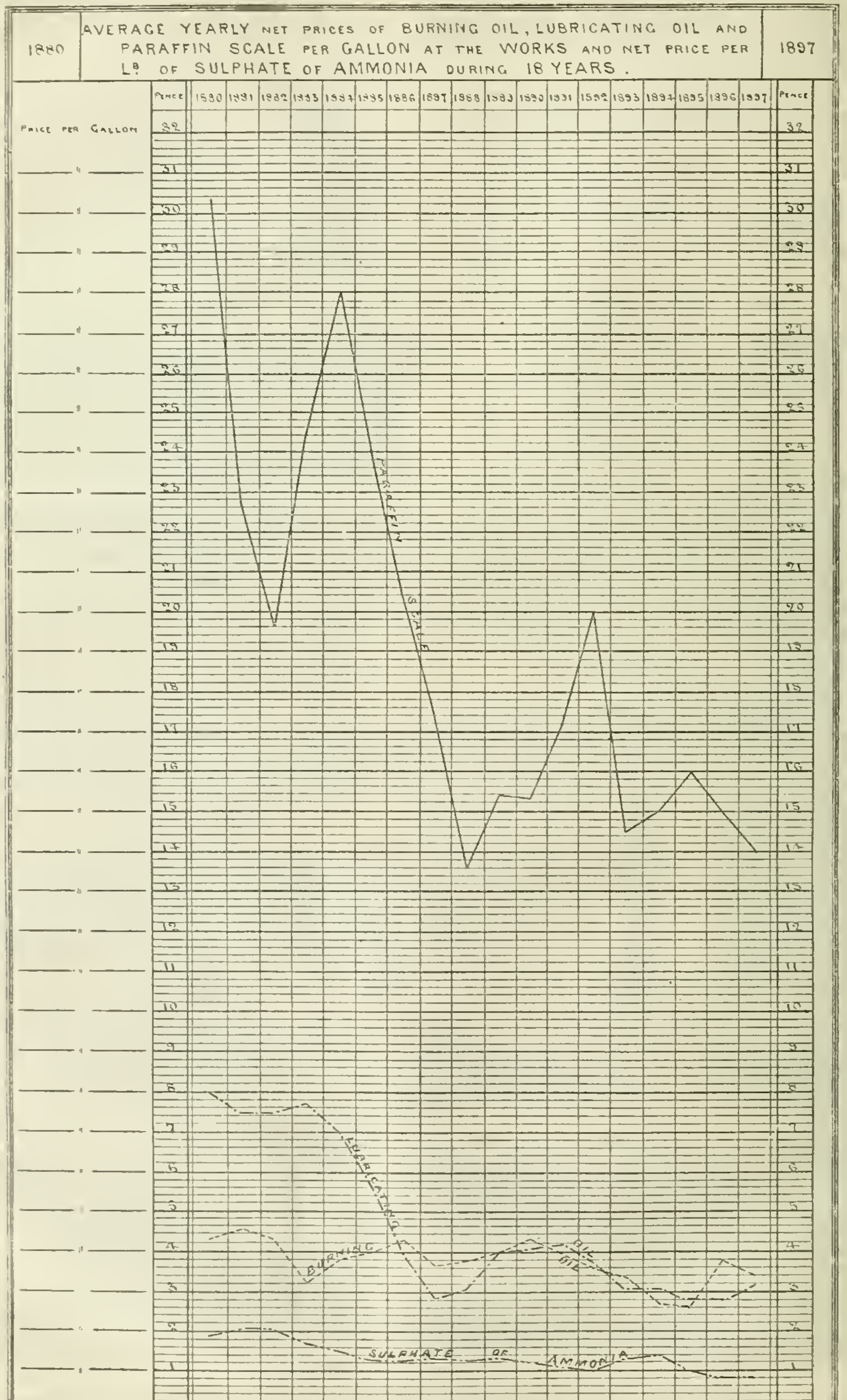
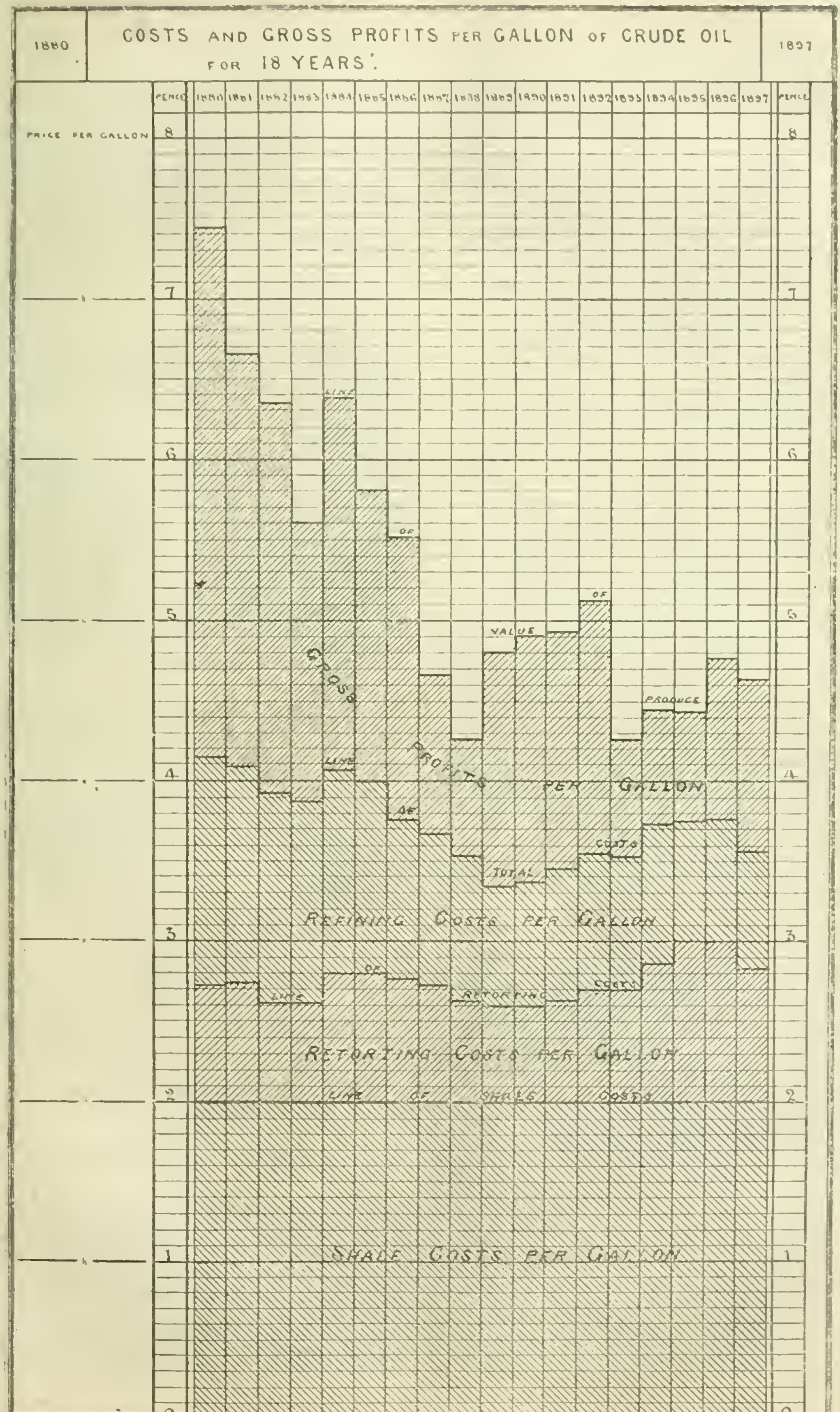

1880
AVERAGE YEARLY NET PRICES OF BURNING OIL, LUBRICATING OIL AND PARAFFIN SCALE PER GALLON AT THE WORKS AND NET PRICE PER L^{b} OF SULPHATE OF AMMONIA DURING 18 YEARS.
1897
PRICE PER GALLON
PENCE
PARAFFIN SCALE
LUBRICATING
BURNING OIL
SULPHATE OF AMMONIA

Fig. 15.


1880
COSTS AND GROSS PROFITS PER GALLON OF CRUDE OIL FOR 18 YEARS.
1897
PRICE PER GALLON
PENCE
1880 1881 1882 1883 1884 1885 1886 1887 1888 1889 1890 1891 1892 1893 1894 1895 1896 1897
8 7 6 5 4 3 2 1 0
LINE OF VALUE OF PRODUCE
GROSS PROFITS PER GALLON
LINE OF TOTAL COSTS
REFINING COSTS PER GALLON
LINE OF RETORTING COSTS
RETORTING COSTS PER GALLON
LINE OF SHALES COSTS
SHALE COSTS PER GALLON

This sum of the balance of Tables Nos. V. and VI. is 10*s.* 6*d.*, which represents the result of the improvements of the past 28 years, valued at the low prices of 1897. If the shale through-put of Scotland is taken at 2,400,000 tons, we find that the value of these savings on one year is 1,260,000*l.* Unfortunately for the shareholders in the oil companies, the greater part of this handsome sum has not gone into their pockets, but has been left in the pockets of the consumers of the products!

The diagram (Fig. 15) shows graphically the progress of the costs, prices, and profits for the past 18 years of one of the leading oil companies.

A supplementary profit-and-loss statement is given, showing the results of working a 20-gallon shale, such as the Pumpherston, at the costs, yields, and prices of 1897:—

VII.—Yields and Costs of 1897 (by Young and Beilby Retorts) with a Twenty-Gallon Shale.

Costs per Ton.

	d.
Shale	48
Retorting	22
Refining	14
	84

Yields per Ton.

			d.		*d.*	
Burning oil	8·4 gals. at		3·41	=	28	
Lubricating oil	3·4	„	3·19	=	11	
Paraffin wax	3·0	„	14·00	=	42	
Sulphate of ammonia, 60 lb. at			½	=	30	111
Gross profit						27

Subject to commercial and interest charges.

The most striking features disclosed by these later statements of profit and loss are, the extraordinary shrinkage in the value of the products from a ton of shale as well as in the costs for obtaining them. The significance of these shrinkages appears to be that nothing but an improvement of prices can raise profits to a satisfactory point. Costs have now been reduced to so low a point that there is small margin left for further reductions. I expect that we shall hear from Mr. Bryson next month something of what he has accomplished in the matter of retorting; but we cannot ignore the fact that each successive penny of reduction is more difficult to win than the last.

Like many others who have been interested in this industry, I have thought much and often on the possibilities which remain for developing new sources of profit. I shall conclude by referring to

Matters which Call for Further Investigation.

(*a*) In the distillation of the shale itself an excessive proportion of the volatile constituents goes off as uncondensed gas. I use the word "excessive" advisedly, for I have found that small quantities of shale, when distilled at a very gentle heat in a moderate vacuum, yield a much larger proportion of oil and a correspondingly smaller proportion of uncondensed gas. I have had this fact before me for more than 25 years, but have never been able to see how it is to be made available practically.

(*b*) At the oil stills a similar waste takes place. Papers on the subject were read before this Section in 1887 by Mr. J. B. McArthur and myself, but I am not aware that anything has been done practically towards the utilisation of this gas.

The total production of this gas cannot be much short of 60 million cubic feet per annum.

(*c*) The basic tars, which are removed from the oils by the action of oil of vitriol, may have unexhausted possibilities. Every ton of shale produces from 3 to 4 gals. of these tars, so that from 7 to 10 million gallons are produced per annum.

The present practice is to wash and steam the free acid out of the tar, and then to burn it as fuel under the oil stills. Its value for this purpose cannot well exceed 15*s.* to 20*s.* per ton.

These tars contain a high percentage of nitrogen, and many attempts have been made to convert this nitrogen into cyanogen or into ammonia. Both conversions are possible; but it is doubtful if either of them can be commercially successful.

It is to a deeper and more exhaustive chemical investigation of the less volatile portions of these tars that attention ought to be directed.

(*d*) The resinous bodies which come off during the coking of the residue stills also deserve fuller investigation.

Chemically, the hydrocarbons which compose the oils and paraffins are not very hopeful subjects to tackle; but perhaps the oil chemists of the next generation may be able to feed us and to supply us with soap as readily as those of the present generation have illuminated and warmed us!

In order that the forthcoming meeting at Broxburn may be practical and useful, it is desirable that all who have questions to propose for discussion should communicate them to the Secretary as soon as possible. It is intended to devote a part of the meeting to the consideration of the latest departure in retorting—the use of automatic drawing appliances; but it will be most gratifying to the Committee of the Section if those who are most heavily interested in the future of the industry will make the fullest possible use of the occasion for the discussion of matters which may influence that future.

In conclusion I have to express my indebtedness to the managing directors and officials of the leading oil companies for the very great frankness and courtesy with which they have placed their records and experience at my disposal during the preparation of this sketch. This is a most encouraging experience for an official of the Society of Chemical Industry, and ought to reassure those of our friends who fear that the objects of the Society may be defeated through the unwillingness of manufacturers to co-operate with the Sections. It is for the Society to show its appreciation of this generous policy by doing all in its power in order that this important industry may gain, not lose, by the mutual intercourse and association.

New York Section.

Chairman: Charles F. Chandler.
Vice-Chairman: T. J. Parker.

Committee:

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Virgil Coblentz.	Clifford Richardson.
H. Endemann.	Wm. Jay Schieffelin.
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E. J. Lederle.	

Hon. Treasurer: R. C. Woodcock.
Hon. Local Secretary:
Dr. H. Schweitzer, 77, William Street, New York, U.S.A.

SESSION 1897–98.

Meetings will be held on the third Friday after the first Monday in every month.

Meeting held at the College of Pharmacy on Friday, October 22nd, 1897.

MR. THOS. J. PARKER, VICE-CHAIRMAN, IN THE CHAIR.

The Secretary read a letter from Dr. Chandler in which he expressed his regret that an imperative engagement out of town made it impossible for him to be at the meeting. He extended a hearty invitation to the members to hold their November meeting in the new Havemeyer Hall, in the Columbia University, in their new building on Morningside Heights.

Dr. Endemann said it became his sad duty to announce to the Section the death of their friend and co-member, Dr. Alsberg. Towards the end of August he commenced to

feel unwell, and decided to visit his family in the Adirondacks. Here he was suddenly taken worse, and though he rallied for a time, he died early in the following month.

Dr. Alsberg was born in 1842, in Waldeck, in Germany, and studied until about his fourteenth year at the Gymnasium at Korbach. After that, his father put him to business, but this was so much against his inclination that his father sent him to the Polytechnic at Brunswick, where he studied and graduated, obtaining a prize, which induced his uncle to furnish the funds for his university education. He first studied with Prof. Woehler; then later on with Prof. Geuther in Jena, and after graduating in 1864, became the University laboratory assistant. He then came to the United States with recommendations from Prof. Woehler to Prof. Joy at Columbia College, and Prof. Chandler engaged him as his private assistant, with whom he stayed about two years. He then commenced the manufacture of "Lactine," an infants' food, well known in Europe, but apparently unknown in the United States at that time. Not being well supplied with funds for advertising, he soon had to give up this manufacture. He then started the manufacture of vermilion, of which he was really the only successful manufacturer in the United States, and he continued in that business until his death. His business has been merged in various firms, the firm at the time of his death being Alsberg and Pfeiffer. He introduced thereafter almost every colour which is manufactured on a large scale. Though essentially a manufacturer, and as such intimately connected with trade, at the same time he never forgot science. Whatever he could do to help on the struggles of scientific men he did, and no doubt many of the members of the New York Section had been benefited by his friendship, and would sadly mourn the sudden recall of their friend from this world.

THE CONSTITUTION OF HYDRAULIC CEMENTS.

BY SPENCER B. NEWBERRY AND W. B. NEWBERRY.

Hydraulic cements, as is well known, are compounds of lime with the constituents of clay, and are obtained by burning natural or artificial mixtures of clay and carbonate of lime at a high heat, and grinding the resulting "clinker" to powder. Portland cement is made by preparing an artificial mixture of the materials, of correct composition, relatively high in lime, and burning the mixture at a white heat. Natural-rock cements are made from a limestone containing a high proportion of clay, and usually much magnesia, by burning at a low heat. The composition of modern Portland cements is remarkably uniform, as may be seen from the following table, taken chiefly from the circulars of the various factories:—

—	Dyckerhoff. (German.)	Germania. (German.)	Porta. (German.)
Lime, CaO	63·75	66·04	62·28
Silica, SiO_2	19·35	21·14	22·69
Alumina, Al_2O_3	7·00	6·50	7·30
Iron oxide, Fe_2O_3	4·50	2·50	2·87
Magnesia, MgO	Not det.	1·11	1·08

—	Empire. (American.)	Saylor's. (American.)	Sandusky. (American.)
Lime, CaO	64·00	62·79	64·19
Silica, SiO_2	20·80	20·64	23·20
Alumina, Al_2O_3	7·39	6·93	7·03
Iron oxide, Fe_2O_3	2·61	5·41	2·41
Magnesia, MgO	Not det.	1·72	0·97

Much has been written in regard to the probable reactions which take place in the burning of cement, and the nature of the compounds of lime, silica, alumina, and iron oxide of which the finished cement is composed. Most of the earlier work in this field is unsupported by experimental evidence, and even a brief synopsis of it would cover many pages. It is generally held that cement consists of basic silicates and aluminates of lime, which form crystalline hydrated compounds with water, thus causing the hardening of cement. The part played by iron oxide and magnesia is obscure, and apparently of secondary importance.

The most complete study of the constitution of cements has been made by Le Chatelier (Ann. des Mines, 1887, 345). His experiments consisted of a careful microscopic examination of cement, both in the form of clinker and after hardening in water. He also made experiments on the artificial preparation of silicates and aluminates of lime, by heating mixtures of the pure materials. The most important of Le Chatelier's conclusions may be stated as follows:—

The monosilicate, $CaO.SiO_2$, found in nature as Wollastonite, is not acted upon by water, and therefore plays no part in the hardening of cement.

The disilicate, $2CaO.SiO_2$, formed by heating chalk and powdered quartz in proper proportions, in a crucible, falls to powder on cooling, in the same manner as over-clayed cement clinker. This spontaneous pulverisation is due to a change of crystalline form (dimorphism).

The trisilicate, $3CaO.SiO_2$, cannot be obtained by heating a mixture of silica with three molecules of lime, a mixture of lower silicates and free lime being always obtained. The mixture heats and slakes with water, but after slaking sets slowly. In Le Chatelier's opinion this trisilicate may be formed indirectly, by heating lime and fusible silicates, and is the chief active constituent of hydraulic cements.

The mono-aluminate, $CaO.Al_2O_3$ (lime spinel), is nearly infusible; sets rapidly with water.

The di-aluminate, $2CaO.Al_2O_3$, is rather fusible, sets quickly with water, like plaster; but the mass, after setting, falls to pieces on boiling with water.

The tri-aluminate, $3CaO.Al_2O_3$, is very fusible, and sets quickly with water.

The ferrites, made by heating mixtures of lime and iron oxide, all slake with water, and show no setting properties.

Hardened cement, according to Le Chatelier, consists of hexagonal plates of crystallised calcium hydrate, $Ca(OH)_2$, embedded in a white mass of interlaced needle-shaped crystals of hydrated calcium monosilicate, $CaOSiO_2.2\frac{1}{2}H_2O$. The chief reaction which takes place in the hardening of cement may therefore be represented as follows:—

$$3CaO.SiO_2 + XH_2O = 2Ca(OH)_2 + CaO.SiO_2.2\tfrac{1}{2}H_2O.$$

Assuming that three equivalents of lime or magnesia, and no more, can enter into combination in cement with one equivalent of silica or alumina, Le Chatelier states that the proportion of lime and magnesia should not be less than the minimum—

$$\frac{CaO.MgO}{SiO_2.Al_2O_3} = 3,$$

nor greater than the maximum of—

$$\frac{CaO.MgO}{SiO_2.Al_2O_3.Fe_2O_3} = 3.$$

In these formulæ, equivalents, and not weights, are indicated.

The conclusions of Le Chatelier are reasonable, and agree closely with the practical results of cement manufacture. Le Chatelier's work certainly deserves the highest praise, and throws much light on some very obscure questions connected with the constitution of cements. Taking this work as a basis, it appeared to the authors very desirable to carry the experiments further, and ascertain, if possible, what proportions of lime, silica, and alumina will yield cement of the highest possible quality. It will be observed that Le Chatelier's formulæ indicate the correct amount of lime to be used with a given quantity of silica *plus* alumina. It does not appear from these formulæ, however, whether a highly silicious clay requires a different proportion of lime from one rich in alumina. It would also be of great interest to determine whether silicious or aluminous clays give the best practical result, the correct proportion of lime being employed in each case

The questions which the authors have attempted to solve are as follows:—

1st. What proportions of lime must be employed for given percentages of silica and alumina in a clay?

2nd. Can a general formula be stated, applicable to all clays, which shall indicate the proportion of lime which will give the best result with each?

3rd. What effect has the presence of oxide of iron in the clay used on the proportion of lime required and on the quality of the resulting cement?

4th. Is the presence of alkalis in the raw materials necessary to the production of good cement?

5th. Is magnesia capable of replacing lime in cement mixtures, or is it inert, as has often been stated; and how does magnesia affect the quality of the cement obtained?

The plan followed in the efforts to solve these questions was that of synthesis with pure materials. These materials were as follows:—

1st. Silica; ground quartz, in impalpable powder, containing 99·19 per cent. silica.

2nd. Alumina, C.P. precipitated, containing 99·50 per cent. alumina, 0·20 per cent. soda, 0·05 per cent. iron oxide, and 0·25 per cent. silica.

3rd. Carbonate of lime, precipitated chalk, containing 99·7 per cent. calcium carbonate.

4th. Iron oxide, red, German C.P.

5th. Magnesia, German, C.P. containing only traces of silica, alumina, and lime.

All these materials were in the form of a perfectly impalpable powder, and none of them left any residue on a sieve of 180 meshes to the linear inch.

Mixtures of these materials, in calculated proportions, were made by rolling backward and forward on a large sheet of paper, then sifting three times through a 60-mesh sieve. The mixtures were then moistened with water, made into a cake, dried, and broken into small pieces for burning.

The burning was done in a Fletcher gas-furnace, using gasoline gas as fuel. To avoid contaminating the charge by the corrosion of the furnace, the fireclay cylinder was replaced by one of exactly similar shape, composed of basic material. This was made by mixing 21 oz. Portland cement clinker (passing a 20-mesh screen), 11 oz. Portland cement, 3 oz. plaster of Paris, and 1 oz. soda ash. The mixture was moistened with water and rammed into a suitable mould. After setting about a week under a damp cloth the cylinders were dried by gentle heat, and were then ready for use. Such cylinders stand repeated heating to a white heat, and are not in the least attacked by the charge. After use, they show, however, a tendency to air-slake, and crumble if left standing some weeks in the air. Good results were also obtained with cylinders cut from commercial magnesia brick.

The amount of material used in each experiment was about 300 grms. This was broken into small pieces and loosely piled into the furnace, thus being exposed to direct contact with the flame. The heat was varied to suit the material, an intense white heat being easily obtained with sufficient blast pressure. The burning of each charge occupied about an hour, or about one half hour after an intense and constant heat was obtained. On cooling, most of the material was found to be well sintered; the light-burned pieces on top were rejected.

It is generally agreed that cements of the highest strength and most vigorous hardening properties are those which contain the highest proportion of lime consistent with constancy of volume, *i.e.*, freedom from tendency to expand and crack. In these experiments the "high-lime limit" was determined by means of the *hot test*, by making a small pat of the cement, allowing it to set over night, and exposing it for four or five hours to steam at about 98° C. (208° F.). Tests were also made by placing the pats in steam as soon as made; this proved less severe and less distinct than the plan of allowing the pats to set over night before exposure to steam. The results obtained were very distinct and striking, cements containing a very slight excess of lime showing marked expansion and cracking. In this manner the limit of lime which could be brought into combination could be fixed within 1 per cent. In most cases the pats were kept in boiling water for several hours after exposure to steam, but no additional information was thus obtained, since pats which remained sound for five hours in steam invariably continued sound in boiling water.

In addition to the hot and cold pat tests, tensile strength tests were also made in many cases. As the amount of material available for these tests was very small, briquettes $\frac{1}{2}$ in. square or $\frac{1}{4}$ sq. in. in section were employed. It was found that these gave approximately one-fourth the tensile strength shown by the same cement in briquettes of 1 sq. in. section. A German Portland cement gave the following results at seven days:—

	Lb.
Average of four $\frac{1}{4}$ sq. in. briquettes, 149 lb. × 4 =	596
" " 1 " "	582

It is not claimed that the results of these tensile strength tests are of any especial significance; they were made simply in order to determine whether the artificial mixtures yield cements which compare favourably with commercial products. As the materials used were practically free from alkalis, which are supposed to play an important part in bringing about a good combination, this question appeared to be an interesting one. The results of these experiments will now be briefly stated.

I.—Proportion of Lime required for given Amounts of Silica and Alumina.

A. Calcium Silicates.—Mixtures of pure precipitated chalk and impalpable ground quartz were made in the proportion of 2, $2\frac{1}{2}$, 3, and $3\frac{1}{2}$ molecules of lime to 1 of silica. These mixtures were burned at an intense white heat.

$2CaO.SiO_2$.—Chalk 200, silica 60. Clinker shrunken and sintered, translucent and crystalline. On cooling, fell entirely to powder, as described by Le Chatelier. Some of the pieces were quenched with water, by which the dusting was prevented. Dried and ground, these pieces yielded a white powder. A pat of this set slowly, showing good hardness at one day; very hard, on glass, at seven days. Hot test, sound; on glass, very hard.

$2\frac{1}{2}CaO.SiO_2$.—Chalk 250, silica 60. Clinker white, somewhat sintered, much shrunken. No sign of dusting on cooling. Pat showed very little setting quality, but hardened slowly in water; fairly hard at seven days, decidedly hard after six weeks. Pat placed in steam (after setting one day), hardened well; very hard, sound, and on glass after six hours.

$3CaO.SiO_2$.—Chalk 250, silica 50. Clinker white, shrunken, slightly sintered, no dusting. Powder showed *no warming* on mixing with water. Pat set fairly at one day, still rather soft at seven days, *hard* at six weeks. Hot test, sound; on glass, very hard.

$3\frac{1}{2}CaO.SiO_2$.—Chalk 256, silica 44. Clinker very little sintered, soft. No dusting. Pat set *hard*, but left glass curved slightly, and cracked before placing in water; became very hard after several weeks in water. Hot test, on glass, sound, hard.

These results may be most conveniently compared in the following tabular arrangement:—

Silicates.

Formula.	Ratio CaO to SiO_2.	Per Cent. CaO.	Per Cent. SiO_2.	Pat Test.	Hot Test.
$2CaO.SiO_2$	1:85	65·11	34·89	Set hard, hard 7 days, hard 6 weeks..	Sound; on glass, hard.
$2\frac{1}{2}CaO.SiO_2$	2:33	70·00	30·00	Set soft, fair 7 days, hard 6 weeks....	" " "
$3CaO.SiO_2$	2:80	73·68	26·32	" " "	" " "
$3\frac{1}{2}CaO.SiO_2$	3:27	76·56	23·44	Cracked, soft; hard 6 weeks	" " "

These results are certainly very different from those obtained by Le Chatelier and described above. There appears to be no difficulty in obtaining a good combination of pure lime and silica up to the proportion of 3 mols. of lime and 1 of silica, provided the temperature is high enough, and the materials finely divided. Le Chatelier does not describe his methods of mixing and burning, and it is probable that his materials were heated in a crucible, and not in direct contact with the flame; in this case the temperature was doubtless much below that obtained in the authors' experiments.

While the combination of silica and lime appears to be complete, as shown by the constant volume of the product, both in cold water and in steam, the setting qualities of the higher calcium silicates are very slow. None of the products obtained, except the first, showed any decided *set* at one day, and the gradual hardening properties are more like those of hydraulic lime than Portland cement. According to Hauenschild (Thonind. Zeit. 1893, 418) the hardest burned portions, called "Grappiers," of the celebrated Teil hydraulic lime have the following composition:—Silica, 23·6; lime, 64·7; alumina, 1·4; iron oxide, 0·8; magnesia, 1·4; sulphuric anhydride, 0·5; water, 7·6. These, when ground, form a white cement, largely used by cement-ware makers. The above analysis corresponds to a ratio of lime to silica of 2·74, or approximately the same as $3CaO.SiO_2$. It will be seen later, however, that the presence of even 1·4 per cent. of alumina is sufficient to greatly modify the character of the silicate.

B. Calcium Aluminates.—Mixtures of pure chalk and alumina were made in the proportion of 2, 2½, and 3 mols. of lime to 1 of alumina. As these mixtures proved to be very fusible, the charges were burned in a large platinum crucible placed in the Fletcher furnace. An intense heat was employed—about the same as in burning the silicates.

$2CaO.Al_2O_6$.—Chalk, 200; alumina, 102. Burned by direct heat, fused to a white slag. Burned in crucible, strongly sintered, semi-fused. Powder white. With water, heated strongly, set in a few seconds, like plaster. Pat, set hard; hard, on glass, after six weeks. Hot test, on glass, fairly hard, rather soft inside.

$2\frac{1}{2}CaO.Al_2O_6$.—Chalk, 250; alumina, 102. Burned by direct heat, fused. In crucible, semi-fused. Powder heated strongly with water, set quick. Pat, off glass at one day; curved and cracked at three days; soft, cracked at six weeks. Hot test, curved and cracked, soft.

$3CaO.Al_2O_6$.—Chalk, 300; alumina, 102. Burned in crucible, semi-fused. Powder heated strongly with water, set quick. Pat, curved and cracked before placing in water, disintegrated entirely in water. Hot test, curved and cracked, soft.

These results may be more easily compared by reference to the following table:—

Aluminates.

Formula.	Ratio CaO to Al_2O_3.	Per Cent. CaO.	Per Cent. Al_2O_3.	Pat Test.	Hot Test.
$2CaO.Al_2O_3$	1·10	52·38	47·62	Set quick, hard, sound 6 weeks	Sound; on glass, fairly hard.
$2\frac{1}{2}CaO.Al_2O_3$	1·37	57·85	42·15	Set quick, curved and cracked	Curved and cracked, soft.
$3CaO.Al_2O_3$	1·65	62·23	37·77	" " "	" " "

The above experiments lead to the following conclusions:—

1st. Lime may be combined with silica in the proportion of three molecules to one, and still give a product of practically constant volume and good hardening properties, though hardening very slowly. With 3½ molecules of lime to one of silica the product is not sound, and cracks in water.

2nd. Lime may be combined with alumina in the proportion of two molecules to one, giving a product which sets quickly, but shows constant volume and good hardening properties. With 2½ molecules of lime to one of alumina the product is not sound.

II.—General Formula for Cements.

Assuming that the trisilicate and di-aluminate are the most basic compounds which can exist in good cements, we arrive at the following formula:—

$$X(3CaO.SiO_2) + Y(2CaO.Al_2O_3),$$

in which X and Y are variable quantities, having different values according to the relative proportions of silica and alumina present in the clay employed.

The formula $3CaO.SiO_2$ corresponds to 2·8 parts of lime by weight, to 1 part of silica.

The formula $2CaO.Al_2O_3$ corresponds to 1·1 parts of lime, by weight, to 1 part of alumina.

Substituting weights for equivalents, we have the following formula, representing the maximum of lime which should be present in a correctly-balanced cement:—

$$\%\ \text{Lime} = \%\ \text{silica} \times 2{\cdot}8 + \%\ \text{alumina} \times 1{\cdot}1.$$

This formula may be used to calculate the proportion of lime which should be used with any clay of known composition, as follows:—

Multiply the percentage of silica by 2·8, and the percentage of alumina by 1·1; add the products; the sum will be the number of parts of lime required for 100 parts of clay.

As 2·8 parts of lime correspond to 5·0 parts of carbonate of lime, and 1·1 parts of lime correspond to 2·0 parts of carbonate of lime, the calculation may take the following simple form:—

Five times the percentage of silica, *plus* twice the percentage of alumina = the number of parts of carbonate of lime required for 100 parts of clay.

As a practical example of the use of this formula, let us suppose a clay of the following composition:—

	Per Cent.
Silica	65·4
Alumina	16·5
Iron oxide	6·1
Lime	2·2
Magnesia	1·9
Moisture: combined water, &c.	7·9
	100·0

Let us now calculate the amount of lime (or carbonate of lime) which must be added to this clay to produce a correct cement mixture:—

		Lime.
% Silica	= 65·4 × 2·8 =	183·12
% Alumina	= 16·5 × 1·1 =	18·15
Total		201·27
Less lime contained in clay		2·20
		199·07 parts of lime required for 100 parts of clay.

As 56 parts of lime correspond to 100 of carbonate of lime, we have—

$$\frac{199{\cdot}07}{56} \times 100 = 355{\cdot}5 \text{ parts of carbonate of lime required for 100 parts of clay.}$$

The correct mixture would then be—

	Parts.
Clay	100·0
Pure carbonate of lime	355·5

The percentage of carbonate of lime in this mixture would be 78·0. On burning this, a cement of high quality will result, provided the materials are finely ground and perfectly mixed.

If we refer to the analyses of cements given at the beginning of this article, we shall find by calculation that the first two (German) cements correspond almost exactly to the proposed formula. The others show a slight deficiency of lime from the theoretical. It will of course be understood that the proposed formula represents the *maximum* of lime which can be used with safety. This maximum can be reached in practice only by most thorough grinding and mixing of the raw materials. Given these conditions, the more nearly the amount of lime approaches that called for by the formula, the higher will be the quality of the resulting cement.

It will be observed that the formula—

$$X(3CaO.SiO_2) + Y(2CaO.Al_2O_3)$$

differs only from the minimum formula proposed by Le Chatelier in the number of molecules of lime assumed to be combined with one molecule of alumina.

Le Chatelier's formula may be written—

$$X(3CaO.SiO_2) + Y(3CaO.Al_2O_3).$$

The difference in composition of cements made according to these two formulas is, of course, slight, especially when using a clay low in alumina. With high percentages of alumina, however, the difference is much more marked. If either of these formulæ is correct, it should give the best results obtainable, when applied to either highly silicious or highly aluminous clays. It may here be mentioned that the common practice of expressing the composition of cements by the *ratio of lime to silicates* $\left(\frac{CaO}{SiO_2 + Al_2O_3 + Fe_2O_3}\right)$ is very indefinite. With pure silica, according to the above formula, this ratio will be 2·8, while with pure alumina it will be only 1·1, and with clays of different relative proportions of silica and alumina this ratio will vary between wide limits. For example, applying the above formula, disregarding the iron oxide, to a kaolin and a silicious clay, we obtain the following :—

	Correct Ratio.
Kaolin (silica, 45 : alumina, 40)	2·0
Silicious clay (silica, 70 ; alumina, 15)........	2·5

This variation in the correct ratio of lime to silicates may be clearly seen in the table given below, in which the composition of cements prepared according to the proposed formula, with different relative proportions of silica and alumina, is given in full.

To determine whether the proposed formula—

$$X(3CaO.SiO_2) + Y(2CaO.Al_2O_3),$$

or the first formula of Le Chatelier—

$$X(3CaO.SiO_2) + Y(3CaO.Al_2O_3)$$

represents the correct proportion of lime in cements, a series of mixtures of chalk, silica, and alumina were prepared according to both formulæ, burned, ground, and tested for constancy of volume and tensile strength. In these mixtures the composition was calculated so that the resulting cements should contain 2, 4, 7, and 12 per cent. of alumina respectively. It was found that the presence of even 4·2 per cent. of aluminate, $2CaO.Al_2O_3$ (2 per cent. alumina), was sufficient to give the tricalcium silicate excellent hardening properties, equal to those of the best cements. With increasing proportions of aluminate the clinker became much more fusible, the mixture containing 12 per cent. of alumina fusing to a semi-transparent slag, and yielding a quick-setting cement. This corresponds exactly with the results obtained in practice with aluminous clays, such mixtures having been found difficult to burn correctly, owing to their fusibility.

The clinkers obtained from these mixtures were faintly bluish-white in colour, and about like good Portland cement clinker in degree of sintering and hardness, except in the case of the mixture with 12 per cent. of alumina, which gave a hard, semi-fused clinker, difficult to grind. The cements obtained were perfectly white, and slow-setting except in the case of that with 12 per cent. of alumina. They set sharply and hardened well, showing qualities fully equal to those of the best cements. The cements containing 2 and 4 per cent. of alumina showed no noticeable heating on mixing with water. That containing 7 per cent. of alumina heated slightly, and that with 12 per cent. strongly. This behaviour, and also the increased quick-setting qualities with rising percentages of alumina, indicate that the aluminate, in presence of large amounts of silicate, behaves in the same manner as when free, and make it probable that the silicate and aluminate of lime in cements are simply mixed and not combined together.

The following tables show the results obtained with the two formulæ under consideration. The tensile strengths given represent the average of two ½-in. briquettes of neat cement, gauged with 22 per cent. of water, in each case. As already explained, the tensile-strength tests are not considered of any importance except as showing that the products are generally at least equal in strength to commercial Portland cements :—

Formula, $X(3CaO.SiO_2) + Y(2CaO.Al_2O_3)$.

——	R. Lime to Silicates.	CaO.	SiO_2.	Al_2O_3.	Pat Test.	Hot Test.	Tensile Strength.	
							7 Days.	28 Days.
Silicate ... 95·8 / Aluminate 4·2	2·67	72·79	25·21	2·00	Set hard, sound, on glass 28d	Sound, off glass, hard	154	173
Silicate ... 91·6 / Aluminate 8·4	2·57	71·90	24·10	4·00	„ „ „	„ „ „	148	227
Silicate ... 85·3 / Aluminate 14·7	2·39	70·55	22·45	7·00	Set hard, sound, off glass 28d	Sound, on glass, hard	180	205
Silicate ... 74·8 / Aluminate 25·2	2·15	68·31	19·69	12·00	Set quick, sound, off glass 28d	„ „ „	105	84

Formula, $X(3CaO.SiO_2) + Y(3CaO.Al_2O_3)$, Le Chatelier I.

——	R. Lime to Silicates.	CaO.	SiO_2.	Al_2O_3.	Pat Test.	Hot Test.	Tensile Strength.	
							7 Days.	28 Days.
Silicate ... 94·7 / Aluminate 5·3	2·71	73·07	24·93	2·00	Not tested	Not tested	..	..
Silicate ... 89·4 / Aluminate 10·6	2·63	72·47	23·53	4·00	Set hard, sound, on glass 28d	Cracked, off glass, hard	196	240
Silicate ... 81·5 / Aluminate 18·5	2·51	71·56	21·44	7·00	„ „ „	Cracked, off glass, soft	155	234
Silicate ... 68·2 / Aluminate 31·8	2·32	69·92	18·08	12·00	Set hard, off glass, cracked	„ „ „	113	152

The above tables show that the formula—

$$X(3CaO.SiO_2) + Y(2CaO.Al_2O_3)$$

gives uniform and satisfactory results in point of hardening quality and constant volume with percentages of alumina ranging from 2 to 12 per cent. The product containing 12 per cent. of alumina is, however, quick-setting and low in tensile strength. A kaolin containing 45 per cent. silica and 40 per cent. alumina would, according to this formula, yield a cement containing 15·7 per cent. of alumina. The writers' experiments with kaolins from various sources show that they yield a very fusible clinker, difficult to burn uniformly and extremely hard to grind, while the resulting cements are quick setting and inferior in strength.

Turning now to the table showing the results yielded by the 1st formula of Le Chatelier—

$$X(3CaO.SiO_2) + Y(3CaO.Al_2O_3)$$

we see that though the difference in composition as compared with the cements in the first table is slight, the high-lime limit has here been clearly passed, as shown by the bad results obtained in the hot test. The defects are more apparent as the percentage of alumina increases, the cement containing 12 per cent. of alumina proving unsound even in the cold pat test. From this it is evident that the allowance of 3 mols. of lime to one of alumina is excessive, and that the proposed formula, in which 2 mols. of lime are assigned to 1 of alumina, is more nearly correct.

III.—Effect of Iron Oxide.

According to Le Chatelier, mixtures of iron oxide and calcium carbonate yield on burning products which slake with water and possess no hydraulic properties.

Dr. Schott (Dingl. Polyt. J. **202**, 434 and 513) found that the alumina in cement could be completely replaced by iron oxide without injury to the hydraulic properties, and prepared cement containing only lime, silica, and iron oxide, which showed excellent hardening qualities.

In the hope of throwing some light on the rôle played by iron oxide in cement, a mixture of pure iron oxide, (Fe_2O_3), and calcium carbonate was prepared according to the formula $2CaO.Fe_2O_3$. On burning, the material fused to a black slag, which yielded a brown powder on grinding. Mixed with water to a paste, this powder showed no heating, and did not set or harden in air or cold water. A pat placed in steam, however, after setting one day in air, hardened rapidly, and after several hours in boiling water showed no cracking and appeared very hard.

From this experiment it appears that lime and iron oxide readily combine, yielding a product which is constant in volume, though it shows no hardening properties in the cold.

A mixture was then prepared according to the formula $3CaO.SiO_2 + 2CaO.Fe_2O_3$, containing 7 per cent. iron oxide. The result was as follows: $3CaO.SiO_2$; $2CaO.Fe_2O_3$. Fusible, clinker black; powder, dark gray; pat, slow-setting, set hard; hard 28 days. Hot test, sound, hard, on glass.

It will be observed that the above cement contains only silica, iron oxide, and lime, no alumina being present. Except in colour, the resulting clinker and cement closely resembled that obtained from silica, alumina, and lime. It thus appears that iron oxide and alumina act in a similar manner in promoting the combination of silica and lime.

A number of mixtures were prepared according to the formulæ—

(1.) $X(3CaO.SiO_2) + Y(2CaO.Al_2O_3) + Z(Fe_2O_3)$ and

(2.) $X(3CaO.SiO_2) + Y(2CaO.Al_2O_3) + Z(2CaO.Fe_2O_3)$.

In the first formula no lime is assigned to the iron oxide; in the second formula the iron oxide appears with 2 mols. of lime. The percentages of alumina and iron in the mixture were 4 and 3 respectively. Both these mixtures gave perfectly sound and satisfactory cements, and no noticeable difference could be observed between them. In tensile strength, up to 28 days, formula No. 1 gave the better result.

Another mixture was prepared according to the maximum formula of Le Chatelier—

3. $X(3CaO.SiO_2) + Y(3CaO.Al_2O_3) + Z(3CaO.Fe_2O_3)$.

In this the percentages of alumina and iron oxide were also taken as 4 and 3 respectively. The resulting cement showed high tensile strength, but failed in the hot test. The pat placed in cold water also left the glass at 2 days, showing that the proportion of lime present was too high.

The percentage composition, and ratio of lime to silicates of the cements prepared according to the above formulæ, is shown in the following table:—

—	Lime CaO.	Silica SiO_2.	Alumina Al_2O_3.	Iron Oxide Fe_2O_3.	Ratio Lime to Silicates.
1............	69·68	23·32	4·00	3·00	2·29
2............	70·23	22·77	4·00	3·00	2·36
3............	71·09	21·91	4·00	3·00	2·46

As above stated, Nos. 1 and 2 gave cements which stood the boiling test well, while No. 3 failed.

From the close similarity in the composition of cements No. 1 and No. 2, it is evident that it is quite unnecessary, in working with ordinary clays, to take the iron oxide into consideration in calculating the amount of lime required. Iron oxide evidently combines with lime in the same manner as alumina. The amount of iron oxide present in ordinary clays is, however, so small, and the amount of lime with which it will combine so insignificant, that the effect of ordinary proportions of iron oxide in cements is extremely difficult to trace.

IV.—Effect of Alkalis.

Most writers on the cement industry have assumed that alkalis, potash, and soda play an important part in bringing about the combination of lime with silica and alumina, and the artificial addition of alkalis to materials deficient in them is often recommended. The writers' experience has been, however, that no advantage results from this addition, and Feichtinger states the same. The foregoing experiments show that pure silica, alumina, and lime, free from alkalis, combine readily to produce well-sintered clinker, and that the fusibility of the clinker depends chiefly on the proportion of alumina or iron oxide present. It thus appears unlikely that alkalis have any important action.

In order to test the effect of soda on the combination of silica and lime, two mixtures were prepared according to the following formulæ:—

(1.) $3CaO.SiO_2 + Na_2O$.

(2.) $3CaO.SiO_2 + Na_2SiO_3$.

5 per cent. soda was taken in both cases. The results were as follows:—

1. Soda largely volatilised. Clinker soft, light, infusible. Powder heated strongly with water. Pat swelled and cracked before placing in water. Hot test swelled, soft.

2. Clinker soft, light, infusible. Powder did not heat with water. Pat set fairly; swelled and cracked after two days in water.

It is interesting and rather surprising to note that while the tricalcium silicate, $3CaO.SiO_2$, proved to be of constant volume, though of feeble hardening properties, the same with the addition of sodium silicate gave a product which swelled and cracked.

It appears from the above experiments that soda is incapable of assisting in the combination of silica with high proportions of lime.

V.—Effect of Magnesia.

The question of the influence of magnesia in Portland cement has been studied by many chemists, and is now undergoing careful investigation by the Association of German Portland Cement Manufacturers. Several eminent authorities hold that the presence of considerable percentages of magnesia causes cement to expand and crack after long periods. The most thorough work on this subject is

doubtless that of R. Dyckerhoff, presented to the German Association in 1895 (this Journal, 1896, 913). According to Dyckerhoff's experiments, more than 4 per cent. of magnesia, whether added to a normal mixture or substituted for an equivalent amount of lime, causes a steady falling off in the strength of the resulting cement. Actual cracking was observed only with 8 per cent. or more of magnesia. As stated above, this question is now being most exhaustively studied by a committee of the German Association, who have laid out a series of experiments and observations, which will require five years to complete. In view of this, the writers have not attempted to investigate the injurious practical effects of magnesia.

There is another aspect of the question, however, which is of great importance to cement chemists, and that is, the influence of the presence of magnesia on the proportions of lime and clay required to produce a correct mixture. If magnesia replaces lime and combines with clay, as indicated by the formulæ of Le Chatelier, it must, of course, be allowed for in the calculation of the mixture. If, however, magnesia is inert, as some have maintained, it may be disregarded, and the clay used may be proportioned to the lime alone. A number of experiments were tried in the hope of throwing light on this question, and though the work is incomplete and not yet conclusive, some interesting facts were brought out which may well be briefly stated.

The first question to be studied is, whether magnesia forms with silica and alumina any compounds possessing hydraulic properties. It is well known that pure magnesia, calcined at high heat, sets with water and hardens like cement.

Pure magnesia (German C. P.) was moulded into cakes with water, dried, and burned one hour at a white heat. The resulting clinker was much shrunken, brittle, and hard to grind. The powder, made into a pat with water, set slowly, hard at one day; very hard but slightly cracked at the edges after several days in water. Under hot test the pat curved and cracked.

The same material, pure magnesia, burned in a crucible at bright yellow heat, showed only slight hydraulic properties and hardened fairly. The hot test remained soft.

It thus appears that pure magnesia, calcined at a high temperature, possesses decided hydraulic properties, but is not constant in volume.

Magnesium Silicates and Aluminates.—Several compounds of magnesia with silica and with alumina occur in nature as minerals, and therefore would not be expected to show any hydraulic properties. Among these may be mentioned: Spinel, $MgO.Al_2O_3$; Enstatite, $MgO.SiO_2$; Forsterite, $2MgO.SiO_2$.

It will be remembered that the mono-aluminate and disilicate of lime show decided hydraulic properties. It is therefore evident that magnesium compounds differ decidedly from those of lime in their behaviour towards water.

Artificial compounds of magnesia and alumina were prepared by mixing pure magnesia and alumina in the proportions: $MgO.Al_2O_3$, $2MgO.Al_2O_3$, $3MgO.Al_2O_3$, and burning at a white heat. None of the mixtures showed any sign of fusing or sintering, and the calcined products were in all cases light, porous, and soft. On pulverising and mixing with water none of the pats showed any tendency to set, and after several days in moist air all were still soft, and turned to mud when placed in water. Neither of the magnesium aluminates showed any tendency to harden in steam.

Artificial magnesium silicates were prepared from pure magnesium and silica, and burned at the highest white heat of the gas-furnace. The results were as follows:—

$MgO.SiO_2$. Semifused, clinker hard, like porcelain. Powder showed no setting or hardening qualities in air, water, or steam.

$2MgO.SiO_2$. Well sintered, clinker porous, rather hard. Powder showed no setting or hardening properties in air, water, or steam.

$3MgO.SiO_2$. Infusible, slightly sintered, clinker porous, soft. Pat seemed to harden slowly in moist air, but after several days in air turned to mud on placing in water. No setting in steam.

From the above experiments it is evident that the compounds of magnesia with alumina and silica have no hydraulic properties, and it is quite unlikely that they play any part in the hardening of cements. Whether the tribasic silicate and aluminate exist as chemical compounds is very doubtful.

Action of Magnesia on Clay.—At the suggestion of Dr. Aug. Dyckerhoff, experiments were undertaken to determine whether magnesia has the power of decomposing clay, when calcined with it, and rendering the silica of the clay soluble. The action of lime on various forms of silica has been studied in a most interesting way by Lunge (Thonind. Zeit. 1894, 655), who found that the silica of kaolin is rendered entirely soluble by calcining with lime, while precipitated and crystalline silica are less completely acted upon, the extent of the action increasing rapidly, however, with increasing fineness of division of the silica. Lunge's method consists in repeatedly digesting the product of the calcination with dilute hydrochloric acid (20 per cent.), and a 20 per cent. solution of sodium carbonate. By this treatment the lime and the silica which has been rendered soluble are dissolved out, while the uncombined silica remains unattacked (this Journal, 1895, 37).

For the experiments with magnesia, a clay of the following composition was taken:—

Loss on ignition	7·46
Silica	65·41
Alumina	16·54
Iron oxide	6·06
Lime	2·22
Magnesia	1·88
	99·57

A sample of this clay was ignited at a red heat, finely pulverised, and 1 grm. treated with hydrochloric acid and sodium carbonate, according to Lunge's method. The residue was 96·24 per cent., showing a loss on digestion of only 3·76 per cent.

A mixture of clay and carbonate of lime was then prepared, corresponding with the formula—

$$X(3CaO.SiO_2) + Y(2CaO.Al_2O_3).$$

This consisted of 100 parts of clay and 356 parts of calcium carbonate. Burned at a high heat, a grey-green, well sintered clinker was obtained. This was ground and treated with hydrochloric acid and sodium carbonate. The residue was only 0·4 per cent., showing that practically all the silica of the clay was rendered soluble by calcination with lime.

A mixture of clay and magnesia was then prepared, in the proportion required by the formula—

$$X(3MgO.SiO_2) + Y(2MgO.Al_2O_3),$$

the lime in the last experiment being replaced by an equivalent quantity of magnesia. The mixture consisted of 100 parts of clay and 144 parts of magnesia. Burned at a high heat, a reddish clinker, partially sintered, was obtained, which yielded a pink powder on pulverising. This showed no hydraulic properties on mixing with water.

Allowing for the loss on ignition of the clay, the composition of this mixture, after calcination, should be—

Magnesia	60·87
Clay	39·13

One grm. of the calcined mixture was then treated with hydrochloric acid and sodium carbonate solution. The residue was 10·14 per cent. or about one-fourth of the clay present. Analysis of the solution and residue gave the following:—

Solution—	
Silica	27·12
Iron oxide and alumina	15·26
Magnesia	48·36
Residue—	
Silica	6·63
Iron oxide and alumina	2·23
Magnesia	1·43
	101·03

From the above experiment it appears that magnesia decomposes clay when calcined with it, but that the action

is far less complete than in the case of lime. The product of the calcination of magnesia and clay has, however, no hydraulic properties, and cannot take part in the hardening of cement.

Proportion of Clay required with Magnesian Material. — In order to determine conclusively whether magnesia should be allowed for in calculating the proportion of clay required for use with magnesian materials, experiments were tried with a dolomite lime (air-slaked), of the following composition:—

	Per Cent.
Lime, CaO	43·5
Magnesia, MgO	31·0
Loss on ignition	25·0
Total	99·5

By calculation it is found that to convert all the lime into cement according to the formula stated, leaving the magnesia free, 21·6 parts of clay (analysis given on page 892 or preceding) will be required for 100 parts of dolomite lime.

A mixture was prepared in these proportions, and burned. The clinker was dark green and well sintered. The cement was yellowish in colour, slow-setting, and hardened well in water, appearing sound at 28 days. The hot test was perfectly sound, the pat remaining on the glass.

In this cement the ratio of lime *plus* magnesia to silica *plus* alumina is 4·46, an immense excess over that called for by the formula of Le Chatelier, in which the magnesia is assumed to act like lime. It is probable, as indicated by the experiments of Dyckerhoff and others, that this cement will show expansion and cracking after some time, and its behaviour in this respect will be carefully watched. This defect, however, as shown by the hot test, will be due to presence of a high proportion of magnesia, and not to excess of lime or deficiency of clay. (See also this Journal 1896, 1037.)

Another mixture, consisting of 100 of dolomite lime with 18 of clay, gave pats which curved and cracked, both in steam and in cold water.

It thus appears that the proposed formula is applicable to magnesian materials, as well as those free from magnesia, also that the magnesia should not be considered in calculating the amount of clay required.

Summary.

The conclusions to which the writers are led by the above experiments may be briefly stated as follows:—

1. The essential constituents of Portland cement are tri-calcium silicate with varying proportions of di-calcium aluminate. This composition may therefore be expressed by the formula $X(3CaO.SiO_2) + Y(2CaO.Al_2O_3)$. From this formula it may be calculated that the correct proportion of lime, by weight, in Portland cement, is 2·8 times the silica *plus* 1·1 times the alumina.

2. Iron oxide combines with lime at a high heat, and acts like alumina in promoting the combination of silica and lime. For practical purposes, however, the presence of iron oxide in a clay need not be considered in calculating the proportion of lime required.

3. Alkalis, so far as indicated by the behaviour of soda, are of no value in promoting the combination of lime and silica, and probably play no part in the formation of cement.

4. Magnesia, though possessing marked hydraulic properties when ignited alone, yields no hydrochloric products when heated with silica, alumina, or clay, and probably plays no part in the formation of cement. It is incapable of replacing lime in cement mixtures, the composition of which should be calculated on the basis of the lime only, without regard to the magnesia present.

Discussion.

The Chairman stated that a number of years ago it had devolved on him to look up the literature on cement, and the analysis more especially. The literature then at his command was not very extensive. He eventually appealed to the Astor Library, but in almost all the analyses he saw of the hydraulic cement none seemed to go above 90 per cent. The remaining 10 per cent. were left to the choice of the cement maker.

Dr. Newberry said he would like to add one thing which he had forgotten to mention. He had put on the board Le Chatelier's formula for the hardening of cements. It was a little hard to believe that those effects took place, and he would merely point out that they were engaged in a series of experiments on that subject which he hoped would soon throw some light on it. They should all acknowledge their obligations to Le Chatelier, who had done the first and most complete work in this field, but that what they hoped to do was to carry that still further and to apply Le Chatelier's microscopic study of the cement clinker and the hardened cement to materials of absolutely known composition; to take the dry calcium silicate without the presence of the aluminium and magnesium silicates, and all the things which confuse it, and submit it to microscopic study during its hardening. They were already carrying on some work in that direction, and when finished they hoped to be able to contribute something to the knowledge of how cement hardened. Their work had previously been devoted entirely to how cement combined.

Dr. F. Vanderpoel asked the reason for having the amount of water in Le Chatelier's formula an unknown quantity, when it appeared in the second member of the equation as a known quantity.

Dr. McKenna said that the review of Le Chatelier's work just presented by Dr. Newberry was a lucid and valuable one and the experiments with which he had followed it up and those which he had in view were really of great value. There was considerable work of that kind needed. It was viewing the subject from a very high plane; the behaviour of these ideal mixtures was very instructive. He thought that in practice the composition of good cements could not be placed within narrow limits. Dr. Newberry had mentioned that the lime should be from 62 to 65, the silica from 20 to 23, and the alumina 6 to 7; but it was found that some good cements varied. Thus, they could run higher in silica, changing the proportions of the other elements and yet not suffer. Silica could also play the part of a beneficial agent and actually combine with the lime and harden thus and not by hydration. This was the action which took place in puzzuolanic cements, but, of course, although a good cement, it would not be one of these ideal Portlands. Perhaps thermo-chemistry would yet tell us more as to the most probable combinations from cement of a given composition.

Very little was known of a certainty regarding magnesia, particularly in the case of cement immersed in water—sea-water. There were many situations in which cements were placed; but the experiments of the German Manufacturers' Association were almost solely on the subject of its influence when immersed in sea-water. This was, of course, its most important application. Cases of cements with high percentages of magnesia were recorded, which were still very sound after a long time. This was only another instance of the fact that in practice they had much to learn.

Dr. Wm. Jay Schieffelin asked Dr. Newberry whether in the last experiments mentioned with the dolomite he had also tried whether magnesia would require the silica in calcining.

Dr. L. N. Adler said he had seen it stated that magnesia when heated still had the capacity of taking up water, and asked whether there was any truth in that statement, and whether that might not account in a measure for the fact that a cement containing 32 per cent. of magnesia still had the necessary properties possessed by cements containing only 8.

Mr. Clifford Richardson said he would like to express his great appreciation of the value of Prof. Newberry's paper. It seemed to him to mark an epoch in the history of the cement industry, and it was encouraging to see that this work had been done in connection with the establishment of the industry in the United States. It was particularly important, in that it enabled us to judge accurately of the value of a clay for the purpose of cement making, and to determine the highest amount of lime which it could combine with, thus putting the industry on a more rational footing than it ever has had before.

Mr. S. A. Tucker said it seemed to him that at the very high temperature necessary, to bring out the effects of the hydraulic properties of magnesia, the electrical furnace product might be very variable.

Dr. Endemann, in reply to a statement that dolomite would give very excellent hydraulic cement, said that he had made experiments in connection with the manufacture of liquid carbonic acid gas from dolomite found in Westchester county, which could be had at a nominal price. The carbonic acid was obtained by passing steam over the dolomite in retorts while the retorts were heated to redness. In that way he found that just one-half the carbonic acid would be driven off and one-half remain behind with the lime. He made experiments with the product thus obtained to find whether it had really hydraulic properties, and had found it absolutely valueless for the purpose.

Mr. M. Toch said that when cement was used in sandstone and limestone buildings, a great many free soluble salts found their way to the surface of these stones and in the course of time defaced the surface of the buildings. These soluble salts had come to his notice in some very peculiar combinations. Several sandstones he examined showed calcium oxalate as the result of the use of cement. It took him a long time to find out how calcium oxalate was produced. He could at once account for magnesium sulphate, which was caused by the presence of pyrites in the coal used for burning the cement. Calcium oxalate appeared to be produced in cements through the action of a certain fungus—the *penecillium glaucum.* Carbonic acid from the air combined with lime and moisture at a favourable temperature to form calcium oxalate, CaC_2O_4, showing that certain micro-organisms possessed synthetic powers as well as analytic powers. Thus calcium oxalate was produced on the surface of limestone indirectly through the cement. Again, calcium oxide, which had worked its way to the surface and formed a white florescence, was treated by a builder with hydrochloric acid, forming calcium chloride, on which thrived the green alga "*Protococcus viride*," known as the green fungus, found on the north side of trees and on brown stone and sometimes on buildings after a rainy spell. When this micro-organic plant rooted itself on calcium chloride there was no way that he has ever heard of in which it could be destroyed. These two peculiar instances were directly traceable to cement.

Dr. S. B. Newberry, in answer to Dr. Vanderpoel, replied that he thought that Le Chatelier meant to imply that there should be an excess of water. In answer to Dr. Schieffelin, the author replied that they had tried that experiment, and the result was a failure. The material was no good as a cement. In answer to Dr. Adler, the author replied that Dyckerhoff stated that magnesia calcined at a high temperature was not constant in volume but was very dense, and when it took up its water, as it ultimately did, it expanded, but did so very slowly; but that magnesia calcined at a lower heat would readily combine with water, and did not swell; that is, it took up water before it set and hardened. Dr. Adler presumably wished to show that injury might arise where cement was calcined at a high temperature, whereas if calcined at a lower temperature it would not show itself. It well known that most common American cements, such as those made from natural rock, containing lime and from 15 to 20 per cent. of magnesia, did not perceptibly crack or go to pieces. Dyckerhoff explained that by saying if Portland cement were calcined at a white heat the magnesia would be very injurious. As to the effect of calcining pure magnesia, they did not find (although he had seen it stated in text-books) that if calcined at a moderate heat it would have hydraulic properties. Calcined at a cherry heat it had very feeble hydraulic properties. After calcining at a white heat, although it hardened very slowly, it became eventually quite hard. There was another reason that must be noted, viz., if the temperature were too high it would fuse down and probably fail to test; it would have no hydraulic properties.

In answer to Mr. Tucker, the author replied that he had never seen the product of an electrical furnace. It would be very interesting to see whether that extremely high temperature had modified these hydraulic properties at all.

ON THE ESTIMATION OF SMALL AMOUNTS OF α-NAPHTHOL IN COMMERCIAL β-NAPHTHOL.

BY JOHN PROCHAZKA AND H. N. HERMAN.

The following method for the determination of α-naphthol in β-naphthol was discovered in the works of the Heller and Merz Company more than two years ago, because a shipment of β-naphthol received at that time, being worked up for colouring matter in the usual manner, failed to give a product of the usual quality. Nor was the true cause of the trouble at once suspected. It was only by a logical analysis of all the conditions of manufacturing that the true cause of trouble was traced to the presence of certain impurities in the β-naphthol employed, impurities which were found to be absent in the lots of β-naphthol that had been used before.

A method for the determination of α-naphthol in β-naphthol has been described by Dr. A. Liebmann before the Manchester Section of this Society, and is published in the April number of the current year.

The principle there applied is identical with the principle underlying our method, and consists in the property of α-naphthol combining more readily with diazo bodies than β-naphthol, forming bodies generally more soluble.

The body recommended by Dr. Liebmann for the purpose of determining α-naphthol in β-naphthol is the diazo compound of *p*-nitraniline, the body we have found expedient to use is the diazo derivative of the 1-4-naphthylamine sulphonic acid, generally called naphthionic acid.

The diazo compound needed for this test should be carefully prepared as follows:—

$27\frac{1}{2}$ parts by weight of 90 per cent. of sodium naphthionate are dissolved in 185 parts of water. 6·2 parts by volume of 66° of oil of vitriol are diluted to 30 times their bulk and run slowly into the naphthionate solution, which is continuously stirred to secure a uniform paste. Then cool off the mixture to below 5° C. and add slowly, stirring carefully, a 10 per cent. solution of sodium nitrite, containing 7 parts of the commercial 98 per cent. product. The resulting paste is made up to a volume of 750 parts of water and is now ready for use.

The solution of β-naphthol to be tested is prepared as follows:—

15 parts by weight of β-naphthol;

200 parts by weight of 25 per cent. of caustic soda solution;

140 parts by volume of 10 per cent. of soda ash solution; are heated together until the β-naphthol is completely dissolved. The solution is then made up to 250 parts by volume, and cooled off to a temperature below 5° C. before use.

The diazo compound is now run slowly and carefully into the β-naphthol solution, which is continuously stirred during the process of mixing.

If the β-naphthol is pure, the resulting azo compound will precipitate completely in the shape of a fine crystalline powder, and hardly any colour will pass into solution, while the first portions of the diazo compound are being added. On the contrary, if α-naphthol be present in any appreciable quantity a coloured solution is formed beside the crystalline powder above mentioned. Even where the percentage is less than $\frac{1}{10}$th the presence of α-naphthol will reveal itself by the peculiar bluishness of the tinge imparted to the solution. If the rest of the diazo compound is added, the presence of the α-naphthol compound is obscured through the formation of a thick colour paste. However, by boiling the product the paste is made to settle in the shape of a layer of fine crystals, and the supernatant liquid is more or less coloured with the α-naphthol compound. From the phenomena previously mentioned, the following short method was finally deduced:—Instead of combining the whole of the β-naphthol, only a part, say, 5 per cent. of the whole, is combined with diazonaphthalene sulphonic acid. The diazo paste prepared as above is well shaken and $37\frac{1}{2}$ parts by volume run into the β-naphthol solution, the same quantity as above, and with the same precautions as already described. It is then not necessary to boil the product, as the insoluble colour formed settles readily, and by dipping strips of filtering paper into the supernatant liquid, and

comparing the tint with that obtained similarly from mixtures of pure β-naphthol with a known percentage of α-naphthol, a fairly accurate idea of the amount of α-naphthol present can be obtained. The method can be further simplified by using colour solutions of known strength and same shade as that of the above coloured filtrate, for the purpose of comparison. The method is more reliable, if moderately large samples are used, but with proper caution can be successfully applied to quantities less than 1 grm.

It is a gratifying sign of the progress of chemical industry that β-naphthol, almost entirely free of α-naphthol, can be had in commerce at a price only slightly above that of the impure article.

Discussion.

Dr. H. Endemann said that he had used the method just described on known mixtures and had found it work well. Did the author know the nature of the substance which is dissolved in an alkaline solution with blue colour?

Dr. Endemann said that the colour obtained in that way from α-naphthol was a brown, and that the solution of the brown did not become blue in the presence of an alkali—at least not according to the table, which gave the reactions. He thought it could not be that substance, but might perhaps be another derivative.

Dr. Schweitzer inquired whether the author claimed that the employment of naphthionic acid for paranitraniline had any advantage? He considered the use of alcohol and toluene in Liebmann's method rather objectionable. He thought, however, that the use of paranitraniline for naphthionic acid must be an advantage, because in the one case they had a soluble diazo compound and in the other the insoluble diazonaphthionic acid. He only called attention to the insolubility of diazonaphthionic acid, and theoretically would be of opinion that a combination of the Liebmann and Prochazka test might be of advantage.

Mr. J. Prochazka, in answer to Dr. Endemann, replied that he believed it to be the compound of the α-naphthol, which was soluble and very bluish, and perhaps it might be a diazo-derivative. The method described by Dr. Liebmann prescribed the use of alcohol and toluene, so that under certain circumstances this method might be more convenient. His method would not be more advantageous for everybody, but having conveniently at hand large quantities of diazonaphthalene sulphonic acid, it was more convenient, and he thought it might prove so for a great many.

A NEW CHROME MORDANT ON ANIMAL FIBRE.

BY O. P. AMEND.

Inasmuch as all persons familiar with the art of dyeing will readily concede that the methods for mordanting now generally in practical use are subject to serious defects and objections, it is most gratifying that a process has recently been found by which all of these defects and objections are practically obviated. The injuries resulting from the old (boiling) mordant bath were in some cases so serious that, for many reasons, some dyers preferred to use colours which would combine with and develop on the fibre without the use of a mordant, although such colours are, as a rule, inferior to colours produced on mordanted material. For the benefit of those not practically experienced in the art of dyeing, a short description of the mordanting, under the old, as well as the new method, will no doubt be interesting.

By mordants we mean chemicals that will form combinations with the fibre, and these combinations of the fibre and chemical will again combine with, or form a lake with, the dyestuff or colours in the subsequent dye-bath. The colours so produced are, as a rule, very fast to light and washing, and do not crock. The salts of aluminium, chromium, copper, cobalt, iron, nickel, manganese, and other chemicals have been employed for this purpose, but the most important by far is chromium. It is usually employed in shape of a chromic salt, such as bichromate of potash or soda. Acetate and fluoride of chromium have been suggested, and are used to some extent. The above-mentioned bichromates, however, still seem to be in general use. Along with the bichromates a secondary acid salt or free acid is used. The object of this addition is to produce a reduction and decomposition of the bichromate employed, in order that the so reduced bichromate may be deposited on and combined with the fibre. Various compounds have been employed for this purpose. Bitartrate of potash or argols and oxalic acid are preferred in alizarin dyeing; sulphuric acid, lactic acid, and the lactates are preferred in case of logwood dyeing. A new method has been suggested in Germany, by which the bichromates are first decomposed by means of sulphuric acid, after which lactic acid or a lactate is added in order to still more reduce the so-decomposed bichromate. It will be seen that the object of all these secondary acid salts and acids is to produce as complete a reduction of the chromium salt on the fibre as is possible. None, however, obtain this much-sought-for result in a perfectly satisfactory manner. On examining the mordants so produced a yellowish tinge can always be observed, this being due to an incomplete reduction. E. Knecht (in Hummel and Knecht) has proposed to reduce the remaining chromic compounds by means of a warm solution of bisulphite of soda.

The object of the new mordant (Amend process) was to combine chromium with animal fibre in such a way as to leave the fibre in its most natural state, and in order to obtain this result, boiling had to be avoided. It was found that free chromic acid combined with animal fibre at ordinary temperatures. The nature of this chemical combination has not been definitely determined, for the simple reason that the chemical composition of animal fibre is not thoroughly known. But if a classification was to be made this new compound would be placed under the heading of a chromic salt, the animal fibre taking the place of the base, in fact, it might be compared with some of the more sparingly soluble bichromates.

To show how similar the reactions of this new chromic acid animal fibre compound are to the inorganic bichromates, may be illustrated by its action towards the aromatic amines. It is well known that bichromates acting on aniline or its salts produce so-called aniline blacks. The same reaction takes place when using this new chrome fibre compound. The aniline black thus produced is formed on the animal fibre, thereby producing a fast black on furs, wool, &c. It has been laid down in all the literature on this subject, that no satisfactory aniline black could be produced on animal fibre, on account of the reducing action of the latter. Nölting and Lehne (page 57, Anilinschwarz und seine Anwendung, &c.) in describing a proposed process for aniline black on wool or silk, quote :—" Dieses Verfahren ist ebenso unpraktisch wie alle übrigen bisher zum färben der Wolle vorgeschlagen."

This reducing action has been explained by various authors in many different ways, but whatever may be the cause of the same, it is a fact that it has been overcome in the new chromic fibre compound.

As the chromic fibre compound was of such great value in producing a fast aniline black, the next step was to produce a chrome oxide fibre compound, by simply substituting some other reducing agent in place of the aromatic amines. It was found that any of the well-known reducing agents could be used, and after a great number of trials, sulphurous acid, in shape of the commercial solution of bisulphite of soda, proved to be the most practical. The chromic acid animal fibre compound is subjected to a very dilute solution of bisulphite of soda, and thereby reduced to a chrome oxyhydrate animal fibre compound. As this reduction takes place at ordinary temperature and in very dilute aqueous solutions, it is easily seen that the fibre is not acted upon in any injurious way. The bright yellow chromic acid compound is now converted into a bluish-green chrome oxyhydrate compound. It had always been the object of the dyer to produce a chromated fibre with as little injurious action in the latter as possible. Although they used the chemicals in very dilute solutions, a temperature of the boiling point of water was necessary during the mordanting operation, which lasted from 1 to 1½ hours. This certainly had an injurious action on the fibre, such as felting, weakening, loss in weight, and harsh feeling. The new process overcame these difficulties as the solutions employed were still more dilute and were applied at ordinary temperatures.

It was shown by actual analytical research that any of the old boiling mordanting methods had injurious effects on the fibre. The boiled fibre was found to have lost from ¼ of 1 per cent. up to 2½ per cent. of its actual weight, according to the material employed: consequently the hot mordant rendered a part of the material soluble, thereby changing its chemical composition. The loss expressed in percentage does not seem great, but in converting it to practical figures it shows as follows:—In a mill dyeing 10,000 lb. of wool a day, and supposing the average loss to be 1 per cent., it would represent a loss of 100 lb. of wool per day, which loss is due to the extracting action of the boiling mordant bath.

Any practical comparative tests between the old boiling and the new mordant, will show great physical differences, so far as elasticity, feel, and appearance of the surface are concerned.

In the new process, invented by Mr. O. P. Amend, the loss in weight of the wool is scarcely appreciable, and this would prove conclusively that no destructive change has taken place. The second action of the old mordant boiling bath is an oxidising one, which can easily be demonstrated in an unmistakable way, by applying such mordant to a so-called indigo-bottomed material. Although indigotine is quite a stable compound, it is oxidised by this hot mordant to a large extent, and it is therefore self evident, that the much weaker animal fibre must necessarily suffer to a still greater degree. By applying the new Amend mordant to the same indigo-bottomed wool no perceptible change takes place, the indigotine is not oxidised, and therefore it is safe to assume that there is no oxidising action on the fibre. It is well known in the art of dyeing that the harsh and brittle properties of the fibre are due to oxidation, such that explains the superior qualities as to the feel and textile strength of fibre mordanted according to the Amend process, when compared to that mordanted in the boiling bath. The fibre resulting from hot mordant always produces a material of inferior spinning qualities, while that mordanted according to the Amend method shows no perceptible change from the original fibre (show slubbing).

It was found that chromic acid combined very readily with animal fibre in very dilute solutions (the solution containing $\frac{1}{30}$ of 1 per cent. of chromic acid), and that almost the theoretical amount (that is to say, about 97 per cent. of chromic acid) was taken up by the fibre in presence of a secondary acid (the amount of such acid employed being one-tenth of 1 per cent.). This secondary acid also proved to be of value in the reducing step of the process. Knowing that chromic acid combined with animal fibre at ordinary temperature, the next question to be considered was: Would this secondary acid, or part of it, also combine with the fibre at ordinary temperature? That acids combined with animal fibre at the boiling point of water is known, and is so stated by Hummel and Knecht. It was found by analysis that 66 per cent. to 70 per cent. of the secondary acid employed combined with the fibre. The next step was to get rid of this acid so as not to interfere in the subsequent dyebath. Could it be removed by simple washing, or would it be more practicable to remove it by means of a chemical agent? Both ways proved successful.

After this treatment we have the chrome-oxyhydrate in its pure form on the fibre, free from chromic and all other acids; and we have before us the ideal form of a lake-forming mordant. The application of the alizarine series in ammoniacal solution (this renders them more soluble and thereby admits of a more even and penetrating dyeing) has always been the desire of the dyer, and the new mordant is especially adapted to this method. A short reflection will show why any of the old chrome mordants will not answer as well. It has been admitted by all authorities, that in the old styles of mordanting, the fibre contains chromic acid, chromic salts, or combinations of chrome which are easily acted upon by ammonia or alkali, and therefore the dyeing in such alkaline baths was rather difficult and very uncertain. That the new mordant, after having been washed by an alkali, is free from all the above-mentioned objections, is self evident. The conclusive proof of this assertion can easily be shown in an experimental way. *Show:* It will be seen that any of the old mordants treated with distilled water will colour the water a yellowish tinge due to soluble chromic acid compounds, whereas the new mordant does not give up anything to the distilled water. The same reaction will take place in a more vigorous manner in the hot dyebath, that is to say, the old mordant will give off a part of its chrome in soluble form, which will necessarily produce precipitates or lakes with the dyestuff in the dyebath, thereby producing a so-called smutting of the material. This involves not only a loss of colouring matter employed, but it also produces colours which will not stand a thorough scouring or stand a severe crocking test.

Now let us look at the product of the new Amend mordant. The first thing one's attention is drawn to, is the condition of the dyebath, and here the most striking illustration is given in case of the logwood dyebath, logwood being the most delicate and difficult dyestuff. In using any of the hot mordants, the logwood bath will turn an inky colour, which shows conclusively that an oxidation or a formation of a dye-lake has taken place in the bath. It is known to everyone skilled in the art that such a dyebath will not produce a perfectly clean dye. In the new or cold mordant the bath will show all the way through a typical logwood colour, absolutely clear and transparent and not having a changed shade due to any oxidation. This fact explains the superiority of the colour produced by the Amend mordant.

It is, moreover, easily understood, that as there is no loss by precipitation or oxidation, the amount of colouring matter used with the new mordant must be considerably less than with any of the hot mordants. This fact has been ascertained in a long run in many of the dye-houses, the actual saving having been from 15 to 30 per cent. in logwood and logwood extracts. The hot mordant has been shown to produce a loss in colouring matter where the reactions are clearly visible in the result, as in the case of logwood. But the same takes place in case of alizarines and other mordant combining colours, where the reactions cannot be as easily followed by actual observation, but the loss is shown by the saving of the colouring matter in the new Amend mordanting process.

Another proof of the superiority of the colours produced by the new Amend mordant, is a comparative exposure to air, light, and moisture of samples dyed with equal percentages of dyestuffs by the new and old mordants. One will readily notice the much greater resistance of the colours produced by the Amend mordant, as compared with those by the old mordants. The different alizarine blues, for instance, show a characteristic bearing towards the action of direct sunlight. It is well known that the alizarine blues fade to a greyish shade, whereas indigo fades simply to a different shade in blue. Alizarin Blues dyed by the Amend mordant fade also, but instead of the grey shade produced by the old mordants, they, like indigo, fade to a different shade of blue.

A simple explanation of these reactions can easily be found, bearing in mind that any one of the old mordants contains chromic acid or acid salts of chromium, whereas the Amend mordant is free from these objectionable compounds; we have already demonstrated the destructive action of chromic acid and chromic salts in the dyebath, and in presence of moisture and light they continue their ruinous action on the colour as well as on the fibre. The correctness of these assertions is evident, inasmuch as every practical dyer admits that colours produced on heavily mordanted material will not stand the action of light and air, as well as the same colour would on a less heavily mordanted fibre; this plainly shows that the more heavily mordanted the material is by the old boiling method, it contains proportionately more chromic acid or its salts; whereas, in the Amend method, no matter how heavily mordanted the material might be, there is no possibility of these undesirable compounds being present.

We think therefore, that the saving of steam, the saving in colouring matters and wool weight, and the improvement of stock and in colours, as before shown, will appear favourably to anyone, and will be convincing of the advantages to be obtained by the use of the Amend process.

DISCUSSION.

Dr. H. SCHWEITZER stated that he had seen, especially in Germany, very careful trials made with the new process, and it was an unqualified success, especially in regard to the saving of dyestuffs and to the preservation of the textile fibre.

Mr. ALAN A. CLAFLIN asked Dr. Amend if, in the whole process of mordanting, the subsequent dyeing would not be conducted in a hot bath just the same as with the ordinary mordant. The use of this new process of chrome mordanting brought up a subject which he had investigated as well as he could with his limited opportunities, and he had the testimony of the dyehouses as corroborative evidence as to the best results to be obtained on a chrome mordant. On very many colours dyers preferred to use assistants which had no reducing power. On some of the new chrome blacks—for instance, the so-called Alizarin Blacks—the dyestuff producers recommend oxalic acid. Dyers frequently used bisulphate of soda, and from the results of the best textile chemists in the United States it seemed to be shown that a completely reduced chrome mordant was not often required. Similar results had also been found in the case of lactic acid. By the use of lactic acid with chrome, more complete and quicker reduction was obtained if the bichromate were first decomposed by sulphuric acid. Yet that process had not been adopted generally in the dyehouses for various reasons, such as unevenness in the dyeing, resulting from a too quick deposition of chrome, and the complicated nature of the process. If the lowest reduction of the chrome gave the best result, the adaptability of the new process was mainly a question of manipulation. He would like to see demonstrated whether the lowest oxide of chrome would give the best results on all colours, or whether it depended on the colour. He thought such experiments had been tried before, and that the testimony of the mill people had been the other way, but he was not certain that the results had been fully corroborated. Dr. Amend assumed that lactic acid could go on to the wool directly without change, whereas in the boiling bath with bichromate the lactic acid was decomposed and could not remain on the wool as an acid. In the cold process lactic acid might be found on the wool in an undecomposed state.

Dr. O. P. AMEND, in answer to Mr. Claflin, replied that as a matter of course it would. It had to be heated to a certain point before the colours could combine with the mordant, but it would take a lower temperature and less time; that was to say, one could develop logwood colour in 20 to 30 minutes at a temperature of 92° to 95° C. That was not actual boiling. The boiling mordants, being of an acid nature, always developed a hard fibre, kinked and harshened it, and there was always a loss in weight. The unevenness in the dyeing was always due to some trace of acid on the fibre. If one took a large piece, say 100 yards, they would have spots or streaks. If they cut out these spots or streaks and compare them with other parts more evenly dyed, they would find they were due to the destructive action of some chromic acid or a chromic acid salt, or else some acid left on that part of the material. Another thing which demonstrated the effect of chromic acid or any other acid, in any form, on the fibre, was by exposing the so-produced colour to the action of sunlight and air. It was under such conditions that its oxidising and destructive action kept going on. In case of Logwood Black, it would turn a beautiful green. If every trace of the acid were removed and the washing is properly done, in the new process there would be no cause of unevenness, and the logwood black (a very handsome colour) would then stand any amount of sunlight. It had been demonstrated practically at Dobson's, in Philadelphia. They had dyed a large quantity of black, and found the results absolutely even. He was sorry he had not the samples to show to the members of the section, but that he would send them if they wished. Chrome oxide hydrate combined with the dyestuff as a base would with an acid; and, if there were no trace of sulphuric, chromic, or any other acid on the wool, a destructive action would not take place. Indigo bottomed wool combined with the chromic acid directly, without any appreciable change taking place; whereas there would be a change in lactic or any other boiling acid mordant, that is to say, about 50 per cent. of the indigo would be lost. This pretty stable compound was actually oxidised by the boiling mordant. In the new mordant there was scarcely a perceptible loss of indigo.

THE NEW GOVERNMENT LABORATORY.

THE new Government Laboratory, which has been two years in course of erection, was formally opened on October 1st. It is situated in Clement's Inn Passage, adjoining King's College Hospital, on a site covering 7,900 square feet. It is a plain red-brick building, with bands and corners of Portland stone, and has the advantage of being lighted on three sides. It consists of three floors and a sub-basement, and stands in an area, the surrounding wall of which is faced with white glazed bricks.

The main entrance, fronting Clement's Inn, leads directly to a central corridor on the ground floor. The corridor runs the length of the building, and communicates by staircases at each end with the other floors. All the internal walls, with exception of certain offices used as sitting-rooms, are faced with glazed bricks—white above, with border and tinted dado below; while the floors of the rooms are of pitch-pine "parqueterie," or of granolithic pavement.

The ground floor contains the private offices of the principal and deputy-principal, the reference library, the waiting-room, the research laboratory (a room 34 ft. long by 17 ft. wide), the Crown contracts laboratories—a set of three rooms, communicating with each other by arched openings, and having a total length of 69 ft. by 17 ft.—and the reference sample laboratory, 28 ft. by 20 ft.

The basement is similar in design to the ground floor, and has a central corridor running throughout its length. The boiler house, with engineer's workshop, various store rooms for glass, chemicals, stationery, &c., and a receiving room for goods, occupy the further end of this floor, the remaining portion being taken up by laboratories for mechanical work, water analysis, and bacteriological work, the standardising of the instruments and apparatus required in the laboratory, the examination of hydrometers and saccharometers used in the Revenue Service, and the examination of the chemicals and the preparation of the solutions in use.

In the area—roofed in at this place with Hayward's lights—is a Hall's carbonic anhydride evaporating and condensing engine, the evaporation of the liquid carbonic anhydride being employed to cool a strong "brine" or calcium chloride solution. The cooled brine is used for making ice, for lowering the temperature of the water used in the main laboratory, and for circulating in the tanks in the cold-storage room.

The first floor of the building differs in design from the lower floors. The greater portion is occupied by the main distillation laboratory—a large room, 49 ft. by 43 ft., lighted by lofty windows and a dormer lantern roof. Opening on to a short corridor leading from this room to the main staircase are the two tobacco laboratories, and two rooms for the superintending analysts. Above these, on the second floor, are two laboratories, typewriter's office, and a museum. A flat roof, reached by a spiral staircase, covers this part of the building, and may be used for operations requiring to be done in the open air.

On the other section of the second floor, at the opposite end of the main laboratory, are photographic rooms and two laboratories, while the roof here carries the water-storage tanks.

A hydraulic lift communicates between the basement and the various floors.

The building is lighted throughout by electricity supplied from the public mains.

The pipes throughout the laboratories are easy of access, and are painted in different colours to indicate their purpose. In the basement the main supplies are carried below the ceiling of the corridor, and are visible throughout their length. On the first floor, in the main laboratory, a channel 5 ft. square, running beneath the central gangway, is cut off from the corridor below, and utilised for the conveyance of the gas-, water-, and steam-pipes. The service to the

benches is carried in a space behind the bench-cupboards, which have loosely-fitting backs to give access to these pipes.

With the exception of the rooms used for offices, where Teale's slow-combustion stoves are fitted, the building is heated by steam circulating through iron radiators standing in the corridors and on the floor of the various laboratories. Beneath the radiators are channels covered with slate slabs, and communicating directly with the outer air. In the main laboratory these ducts, carrying the fresh air, open into the central channel beneath the gangway along which the steam pipes are carried, the heated fresh air passing into the room through an iron grating. Along the base of the dormer lantern roof is fixed a copper steam-pipe for preventing a down draught.

The main ventilating system is worked by a fan situated in the basement. Leading from the top corners of the main laboratory down to the basement, and opening by gratings in the rooms through which they pass, are four air-shafts, which connect by oblique passages with a main flue, 7 ft. square, running underneath the basement corridor. The flues in the various evaporating and fume cupboards, with one exception, also lead downwards to the same main channel. The entire system is exhausted by the fan, the vitiated air being driven up the shaft surrounding the boiler flue. The fumes from the cupboard in which sulphuretted hydrogen is generated, pass away by means of a shaft entering directly the boiler flue.

There are three distinct water systems. High-pressure water is conveyed directly to the laboratories by branches from the New River Company's main, and is available for working filter pumps and doing other mechanical work. The water used in this way does not pass to the drain, but is conveyed to a clean waste reservoir below the basement floor. The New River Company's service is also connected with tanks on the roof for furnishing the ordinary bench supply to the various laboratories. This water, however, if simply used for condensing purposes, is received into a pipe, which conveys it to the clean waste cistern. The service to the benches in the main laboratory is from a special insulated tank of 1,000 galls., supplied with water cooled by the brine from the refrigerator. After passing through the condensers this "cooled" water also flows to the clean waste, serving to reduce the temperature of the general supply. The contents of the clean waste reservoir are pumped up to the storage tank on the roof; the boiler feed-water is a separate supply.

Reduced steam from the boiler is laid on to the various laboratories for heating and drying purposes, and for furnishing distilled water. The steam evaporating cupboards are constructed of crown glass in oak framing. The bottom is of slate, with an oak-faced projection or ledge in front, 6 ins. wide. The roof slopes from the front to just above the downward opening of the draught flues in the wall at the back. The doors slide up and down with balance-weights, and are prevented from shutting close down by india-rubber buffers. Underneath a bevelled hole in the slate slab is fixed a conical copper vessel. A removable porcelain collar, rising 3 ins. above the slate, fits in the hole, and carries a disk, 12 ins. in diameter, made of a steel plate thickly coated with "woodite," a material which has been found to be little attacked by steam, acid, or alkali. The disks are perforated with different sized holes. The pan is connected with two pipes; one of these, the steam supply, is opened by a valve when the bath is required; the other, fixed slightly below the steam pipe, carries away any accumulation of water from the condensation of the steam to a glass-faced indicator box, containing an overflow pipe at the same level as the outflow from the copper pan. This box, being connected with the water supply, serves to provide a constant level of water in the pan when, the steam from the boiler not being available, the vessel is heated by gas—a Bunsen burner being fixed below for this purpose. In those cases where the slate bottom carries more than one pan, the cupboard is divided by glass partitions into compartments, each separately connected with the flues.

Cupboards similar to these, but fitted with a gas supply in place of the steam arrangements, are also erected for conducting operations requiring a higher temperature or evolving noxious vapours. The metal connections of the gas are outside the cupboard.

Distilled water is obtained in the various laboratories by condensing the excess steam used for heating the sand-bath and drying oven, the whole forming a connected system. Fixed to the wall, at a height of 5 ft. from the floor, is the drying oven, made of stout copper, and protected externally with asbestos and lagging of oak. The bronze front has plate-glass panels. A regulated current of air may be introduced, but the apertures are so arranged that the air passes through a considerable portion of the heated covering before entering the bath. On opening a valve, steam is admitted to the surrounding jacket of the oven, and, after circulating there, is carried by means of a copper pipe to a worm of block tin contained in an iron drum provided with a supply of cold water. The condensed water runs into a large earthenware jar fitted with glass tap. An overflow pipe of stout glass leads from the bottom of the jar, and at the same time indicates the level of the water within.

Beneath the steam oven stands a small press, the top of which consists of a tin-lined copper box. This box is heated by the steam supply, and also receives the hot water obtained by the partial condensation of the steam in and around the drying apparatus. It is provided with a tap and furnishes hot distilled water. The box has an overflow pipe communicating with a steam trap, and returns all surplus water to the boiler. The top of this small hot-water cistern is tray-shaped, and is used as a sand bath, while in the warm cupboard beneath, rails are arranged for the drying of towels, &c.

The working benches in all the rooms except the main laboratory are arranged along the walls beneath the windows, and consist of a mahogany top overhanging by 3 ins. a row of drawers and cupboards below, the whole being raised from the floor to allow of recessed toe-space. The gas supply is carried along the front of the bench beneath the projecting ledge, where are also the taps on the branch pipes, which pass under the top to the nozzle at the back of the bench. Between each two workers is an oblong porcelain basin with water standard, fitted with a Kelvin tap delivering water to the basin, and with branch pipes for drawing off water for condensers. A Fischer metal filter pump with indicator gauge, and connected with the high-pressure water system, is fixed on the bench, and also a pipe for conveying away the clean waste water from condensers, &c. The waste water from the basin passes into a V-shaped wooden trough, lead lined and coated with pitch. This trough, after receiving the waste from other benches, empties into a funnel, also pitched; a continuing pipe leads to the drains outside the building.

The reagent shelves are of stout plate glass resting upon gun-metal brackets, the ledge of the bracket being faced with a strip of corrugated rubber.

In the main laboratory are eight blocks of benches arranged in two rows on either side of a wide central gangway, to which the benches run transversely. Each block is divided into four working benches, a white-ware basin being at each end of the block for the joint use of the two men working opposite to each other. Down the centre of the block, and raised a foot above the mahogany top, is a pewter-covered shelf, beneath which is the service pipe of cool water with which this room is supplied for distillation work. The electric light wires are carried beneath the shelf to stand-lamps at each end, and buttons are also fixed in the circuit for the attachment of electric motors. Each block is connected independently with the main supply and return pipes, and may be cut off without affecting the other benches in the room. A shallow pewter-lined tray with draining hole rests on the edge of the basin, for holding the ice-bath and flasks, and when not in use slides into recess below the bench top. The cupboards and drawers are similar to those in the other rooms, being of oak wainscot, and the pipes are conveyed in a space between the backs of the benches.

A dark room for polarimetric work opens from this laboratory, which is also provided with a cold-storage chamber, cooled by a circulatory system of brine from the refrigerator.

The standard solutions are kept in small oaken cabinets with glass-panelled doors. The top of the cabinet is of

crown glass, and carries a light framework of brass, with small spring clips, for holding the burettes. A side tube, with stop-cock, at the bottom of burette is connected by piece of rubber tubing passing through a hole in glass top with the solution bottle contained on the shelf opposite the glass door of the cabinet, the burette being filled by suction. After a titration the liquid is run back into the storage bottle, which is protected by a soda-lime tube and stop-cock. A reserve stock of solution is stored at the bottom of the cabinet.

One of the tobacco laboratories is constructed of fire-proof materials, and contains furnaces and drying ovens. The drying ovens are arranged so that the steam may pass through one or more as required. They are connected with a self-filling boiler heated by gas, arranged so as to maintain a supply of steam over night.

There are three large muffle furnaces, standing on a wide stone hearth, beneath a capacious flue, and a specially constructed piece of apparatus for charring vegetable substances. This consists of 160 small Bunsen burners arranged in five double rows and regulated in fours from the front. A nickel-plated grid, for holding the dishes, is fixed above the whole.

In the different laboratories are arrangements adapted for the work of the particular department.

For melting butter and other fats, there is an oven with sliding glass doors, and beneath it an open copper bath in which to carry on the filtration—both being heated by steam as required. For combustion work there is a stone table with glass hood, and connected with draught flue; and a Muencke trompe connected with the high-pressure water supply is attached to the wall above the blow-pipe table.

The laboratory has been built, under the superintendence of the Office of Works Department, from designs furnished by Dr. T. E. Thorpe, F.R.S.

Obituary.

SIR HENRY DOULTON,

Member of the Society of Chemical Industry.

Sir Henry Doulton died on Nov. 17, at his residence in Queen's Gate Gardens, South Kensington. Death was due to an affection of the heart, with which he was seized only a few days ago. Sir Henry, who was the second son of Mr. John Doulton, of Lambeth, was born in 1820, and educated at University College School. At the age of 15 he began his technical training as a potter, and in 1870 commenced the manufacture of art pottery and Doulton ware, and ultimately became the senior partner in the firm of Doulton and Sons, which has establishments at Lambeth, Smethwick, St. Helens, Burslem, and Paisley. He was Knighted in 1887, having already been created a Chevalier of the Legion of Honour. The Albert Medal of the Society of Arts was specially conferred on him at Lambeth by the Prince of Wales. He had also received 105 diplomas of honour and gold medals, and 102 silver medals. The speciality of the work of Messrs. Doulton was terra cotta, but it was not confined thereto. Sir Henry greatly stimulated the art education of Lambeth, and it was his experienced eye which detected the artistic work of Mr. George Tinworth, all whose beautiful bas reliefs have been produced at Lambeth, and who has continued throughout his life in the service of his first patron. Sir Henry provided the best accommodation for his workpeople, who were considered in every way.

In the year 1889, when the Annual Meeting of the Society was held in London, Sir Henry Doulton entertained the members to luncheon at his Art Pottery, in Lambeth, and gave a most interesting demonstration. (See this Journal, 1889, 510—511.)

He married, in 1849, Miss Kennaby, and was left a widower in 1888. During the whole of his business life he identified himself with most of the philanthropic and charitable works started for the benefit of South London.

Journal and Patent* Literature.

I.—PLANT, APPARATUS, AND MACHINERY.

PATENTS.

Coffey's Distilling Apparatus, Impts. in the Packing of the Tubes in the Rectifiers of. R. M. McDougall, Glasgow. Eng. Pat. 24,405, Nov. 2, 1896.

A ferrule is soldered on to the still tube, where it passes through the frame of the rectifier, and is screwed externally to receive one or more screw collars or glands, by means of which the packing material is compressed into recesses in the frame surrounding the tube; this represents the claim. A gland may be provided both on the exterior and interior of the frame, or on the exterior only.—R. A.

Filter Presses [Potter's Slip, &c.], Impts. in. J. Critchlow and T. Shore, both of Stoke-on-Trent, Stafford. Eng. Pat. 26,112, Nov. 19, 1896.

The invention relates to the class of filter press described in Eng. Pat. 17,024 of 1893. Instead of mounting the filter trays on rollers, as in the prior specification, they are provided with shafts or studs carrying rollers, which run on racks on the side frames of the machine. The trays are brought together by rotating the shafts, &c. by means of suitable handles, and the required pressure is applied to the end tray by means of a screw, which is tightened up first by hand and then by worm gearing.—R. A.

Absorbing Gases in Liquids, An Improved Absorption Apparatus for. H. Hirzel, Leipzig-Plagwitz, Germany. Eng. Pat. 18,578, Aug. 10, 1897.

The apparatus comprises a vessel through which the gas is slowly passed, and a number of sets of spraying nozzles arranged one set above the other within the vessel. The nozzles of each set are directed tangentially so as to produce a rotation of the sprayed liquid and the gas, the nozzles of adjacent sets being set in opposite directions to ensure a thorough mixture of the gas with the liquid. To facilitate the removal or insertion, or the opening or closing, of any of the nozzles, they are attached by means of connecting pipes and couplings with a multiple-way cock which controls the liquid supply to each nozzle.—R. A.

* Any of these specifications may be obtained by post by remitting 8d.—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

II.—FUEL, GAS, AND LIGHT.

Coal Ash, Relation between the Composition and Fusibility of. E. Prost. Bull. Assoc. Belge des Chim. **11**, [4], 119—126.

Taking as a basis, on the one hand, the results of determinations of composition and fusibility of 29 samples of Belgian coals, and on the other, the formula established by Bischof for the fusibility of clays, the author finds that the quotient obtained by dividing the amount of oxygen in the silica by that in the alumina of the ash, and the result by the oxygen of the other bases present (Fe_2O_3, CaO, MgO), affords an indication of the relative fusibility of the ash. The result is, however, dependent on the percentage of the last-named bases, and especially of iron, since, as this percentage increases, the fusibility decreases, in consequence of the fusible silicates becoming embedded in the excess of bases and the whole partaking more of the refractory character of the latter.

Combining the above considerations, the practical result obtained is, that when the said quotient amounts to or exceeds 2·25, in presence of not over 7 per cent. of iron, the fusing point of a coal will be about 1,500° C.; *i.e.*, the ash is considered as refractory. On the other hand, when the value of the quotient ranges between 1·6 and 2·25 (or even higher), but the iron exceeds 7 per cent. to any appreciable extent, then the ash is moderately fusible (1,350° to 1,500° C.). And finally, all samples of ash yielding a quotient inferior to 1·6 will be found fusible at about 1,350° C.—C. S.

Coke-Oven Gases, Utilising. Report of the Commissioner of Labour and Inspector of Mines, Tennessee, 1896. Eng. and Mining J. 1897, **64**, [13], 369.

In the utilisation of coke-oven gases for the heating of boilers, the following points should be observed:—The flue for conducting the gases from the ovens to the boilers should be built of the best material, and of ample size, and should be so constructed as to be without a descending grade. The boiler flues and stack should be of such size that the gas may meet with as little friction as possible in its passage. The ovens should be promptly and systematically drawn and charged, and should be kept covered when in use. The flue and boiler setting should be kept air-tight. —A. S.

Water-Gas, Progress in the Production and Use of (Dellwik's Generator.) H. Dicke. J. für Gasbeleucht 1897, **40**, 665—669.

Water-gas is made for 15 minutes, and producer gas (CO + N) for 45 minutes per hour, in generators of the usual type; but in Dellwik generators, water-gas is made for 45 minutes and spent gases (CO_2 + N) for 15 minutes per hour. 18 kilos. of steam are decomposed to form water-gas by 12 kilos. of carbon by the aid of 28,760 calories, derived from the action of the air-blast on the fuel. 12 kilos. of carbon under the air-blast, afford 14,905 available calories by the old, and 61,598 available calories by the Dellwik process. The Dellwik process therefore, it is said, makes available for water-gas production about four times as much heat as the old process does. The Dellwik generators yield no producer gas, and give at least twice the production, it is stated, of the old water-gas generators; they are therefore specially adapted, it is claimed, for the supply of towns with water-gas for lighting by incandescence. Water-gas, odorised by mercaptan or carbylamine, is the cheapest and best gas for such a purpose. It may, however, in some cases be carburetted by passage through a retort in which oil is being carbonised. The heat of the retort is easily controlled, and the supply of either water-gas or oil to the retort can be regulated merely by turning taps. A more uniform carburetted water-gas can be produced by this means than by the generators, with large regenerative carburetting chambers attached, which are in use in America and England. Where an increased make of gas is required in existing gasworks of limited area, water-gas may be introduced and mixed with the coal-gas. If 1 kilo. of coke affords only 1·6 cb. m. of water-gas, the surplus coke from the coal-gas retorts, after firing them, will suffice to make so much water-gas that the total volume of the mixed gases would be 2·6 times that of the coal-gas. A mixed gas, consisting of coal-gas and as much water-gas as that coke which is not used as fuel will afford, if burnt at the rate of 200 litres per hour, gives a light of 100 Hefner units. Coal-gas similarly burnt in incandescent burners at the rate of 110 litres per hour, yields 60 Hefner units. Given a works supplying 3,600,000 cb. m. of coal-gas per annum, and obtaining 12·823 Pfennige per cb. m., the same amount of light will be secured from 4,083,000 cb. m. of mixed gas, and the equivalent price will be 11 Pf. per cb. m. It is computed on this basis and with coal at M. 137 per 10,000 kilos., that the net profit per cb. m. will be 3·818 Pf. for coal-gas and 5·082 Pf. for the mixed gas. Also, that for a given calorific value, the holder cost of the mixed gas will be about 40 per cent. less than that of coal-gas.

The latest Dellwik generator (this Journal, 1897, 431 and 525) yields, however, 2·4 to 2·5 cb. m. of water-gas per kilo. of coke. In one trial, in which a $1\frac{3}{4}$ minutes' blast alternated with a 12 minutes' make, and 100 cb. m. were made per hour, 2·4 cb. m. of water-gas were obtained per kilo. of smithy coke from the Consolidation pits (Ruhr district). The spent blast gases contained, on an average, 18·2 per cent. of carbonic acid, and 100 vols. of the water-gas consisted of:—Carbonic acid, 3·6; carbonic oxide, 40·4; hydrogen, 51; nitrogen, about 4·5; and marsh-gas, about 0·5. There was about 92 per cent. of carbon in the coke; hence about 2·6 cb. m. of gas were obtained per kilo. of carbon. Therefore 2·5 cb. m. may be expected with certainty; and, at a calorific value of 2,600 per cb. m., and with carbon yielding 8,080 calories per kilo., about 81 per cent. of the calorific power of the carbon will be found in the water-gas. In another trial, with $1\frac{1}{4}$ minutes' blast and 7 minutes' make, and a production of 100 cb. m. per hour, 2·55 cb. m. of gas were obtained per kilo. of Essen gas-coke. The larger make per kilo. appears to be due to the greater porosity and more extensive surface of the gas-coke. In a 27-hour trial, 120 cb. m. per hour and 2·58 cb. m. per kilo. of Charlottenburg gas-coke were secured. The injuries caused to incandescent mantles by deposition of iron from traces of iron carboxide in water-gas have been exaggerated. Water-gas from which iron has not been separated has given satisfactory results at Radkersburg, where it has served as the town supply for $1\frac{1}{2}$ years. —J. A. B.

Calcium Carbide, Impurities in Commercial. H. Le Chatelier. Bull. Soc. Chim. 1897, **17**, 793—794.

Besides calcium and carbon there are present in commercial calcium carbide, silicon and iron. By decomposing by water and then by acid, and floating the residue in methylene iodide, the whole of the iron is found at the bottom as silicide. If the calcium be in excess of the carbon, the remaining silicon appears as calcium silicide; but if the carbon be in excess, carbon silicide is formed. Thus the determining affinities are those of iron for silicon and of carbon for calcium. There seem to be two silicides of calcium—one attacked by hydrochloric acid, but not by nitric; the other easily attacked by nitric or acetic acid, and giving with hydrochloric acid an insoluble white residue in place of the yellow one (the "*Silicone*" of Wöhler) which the other leaves. The attack of calcium silicide by acid leaves a mixture of these residues, analyses of which vary between the formulæ $Si_2O_4H_4$ and $Si_2O_3H_4$.—J. T. D.

Acetylene, Poisonous Action of. U. Mosso and F. Ottolenghi. Ann. di Chim. e di Farmacol, **25**, 163; Chem. Zeit. Rep. **21**, [59], 158.

See under XVIII. B., *page* 930.

Total Ammonia in Gas Liquor, Estimation of. E. Donath and K. Pollak. Zeits. angew. Chem. 1897, 555.

See under XXIII., *page* 936.

PATENTS.

Incandescence Bodies for Gas and other Burners, Impts. in. A. Kiesewalter, Limburg-on-the-Lahn, Germany. Eng. Pat. 20,221, Sept. 2, 1897.

The novelty claimed is "the preparation of incandescence lighting mass" by fusing in a closed vessel a mixture of pure oxides of barium and magnesium together with a small quantity of antimony oxide or bismuth oxide. The glassy mass is pulverised, dissolved in acid, and the solution used for impregnating organic materials. If it be desired to avoid a yellow or reddish-yellow tint of light, in place of silica or zirconia, lime or thoria may be used, either each separately, or both intermixed, whereby a bluish or yellowish light is obtained.—H. B.

Incandescent Bodies. An Improved Solution or Fluid for Impregnating. L. Freudenthal, Berlin. Eng. Pat. 20,225, Sept. 2, 1897.

Thorium oxalate is converted "by suitable treatment" into nitrate, which is dissolved in water, and "a small quantity of the chlorides of other metals is added." No specific example is given, but "the choice of the metal chloride to be added is made according to the actual requirements as regards (for instance) strength of light, colour of light, permanency of the light, or durability of the incandescent body."—H. B.

Incandescent Oil Burner, Impts. in or relating to Burning Liquid Hydrocarbons in conjunction with an Incandescent Hood or Mantle to produce an. J. L. Waldapfel, Cologne. Eng. Pat. 20,713, Sept. 18, 1896.

The arrangement of the lamp is such that "that part of the apparatus in which the gases are mixed and filtered, is built into the reservoir for the liquid." The initial gas pressure is obtained by applying an auxiliary flame directly to the bottom of the reservoir containing the liquid to be vaporised. Within the reservoir, and fastened to its upper cover, is a cylindrical upright vessel, into which the gases liberated from the heated liquid hydrocarbons, flow, and are made to pursue a tortuous up-and-down course through a series of pipes and chambers within the vessel. On their way the gases have to pass through a filter or mixer, consisting of a number of small layers of felt with intermediate wire gauze, and finally they issue into a small gas-holding chamber at the top of the lamp, through a tube with a downwardly directed mouth. The gasholder is surmounted with a burner and mantle as usual. From the bottom of the internal cylindrical vessel passes a small pipe to the outside of the lamp, fitted with a valve-cock, and terminating in a burner under the bottom of the reservoir. As soon as gas is generated, the auxiliary lamp is removed, the valve-cock is opened, and the gas issuing from the burner is ignited, so as to make the lamp act continuously.—H. B.

Hydrocarbon Incandescent Lamps, Impts. in or connected with. R. Haddan, Buckingham Street, W.C. From J. W. Allison, New York. Eng. Pat. 3146, Feb. 9, 1897.

To avoid the use of capillary tubes, the vaporiser consists of a straight tube of relatively large diameter, having at its upper end a dismountable nozzle and orifice, which are connected with a device wherein the gas mixes with air, which is sucked in by the rush of gas. The rate of flow of the gas through the orifice is regulated by a needle which extends throughout the length of the vaporising tube, and is actuated by a lever at its base. The admission of petroleum to the vaporising tube is controlled by means of a lever provided with an eccentric, operating upon a diaphragm which carries a valve. "The two essential elements" are the vaporiser and air-mixing chamber, and they may be combined for the production of incandescence by means of mantles, in various ways. There are twelve claims for various combinations of parts.—H. B.

Incandescence Bodies for Heating Purposes, Impts. in and relating to. A. Quentin, Brussels. Eng. Pat. 23,006, Oct. 16, 1896.

A "heating mass" is to be prepared which will become incandescent on contact with a mixture of air and certain combustible gases. The heating surfaces of suitable apparatus (boilers, &c.) are to be coated with this preparation. After the preliminary heating of a part of the coating, the gaseous mixture is to be turned on, causing incandescence over the whole surface: ordinary fuel is to be dispensed with. To obtain such a "heating mass," a mixture of kaolin and powdered pumice is treated with a solution of a suitable metallic salt (such as platinum chloride), and on calcination a porous mass containing metallic platinum is obtained. This process of manufacture is claimed, and there is a general claim for "a heat-giving mass for a heating surface" of the nature above described.—H. B.

Incandescent Lighting or of Heating [by Acetylene], Impts. in Apparatus for applying Acetylene Gas to the Purpose of. E. Edwards, Chancery Lane. From R. Terr, Paris. Eng. Pat. 26,041, Nov. 18, 1896.

Acetylene gas is to be burned in a form of Bunsen burner, the flame raising a mantle of the well-known kind to incandescence. The air is admitted to mix with the gas in such proportion that the flame is smokeless and forms a small blue-green cone surrounded by a reddish outer one. The burner claimed differs from the ordinary Bunsen burner (1) by having the upper end of the burner-tube fitted with a conical or any other burner-mouth the sectional area of which is less than that of the burner-tube; (2) by having at the lower end of the burner-tube a tubular acetylene inlet jet which is movable within the burner-tube, so that the latter can be lengthened or shortened; and (3) by being fitted internally with a cylindrical piece which "is pierced with a hole which allows the mixture of gas and air to be drawn in more quickly." The application of mantles for producing incandescence by the combustion of acetylene is claimed.—H. B.

Mantles or Incandescing Bodies [Self-Lighting] for Incandescent Gas Lamps, Impts. in the Manufacture of. J. F. Duke, West Dulwich. Eng. Pat. 26,618, Nov. 24, 1896.

Reference is made to Eng. Pat. 969 of 1895, which described an automatic gas igniter consisting of a block of meerschaum containing in its pores platinum black. According to the present invention a very fine line of highly concentrated platinum chloride solution is painted downwards from the top of the mantle, before the organic fibre is burnt out. After burning out, the line of metallic platinum left is streaked over with a solution of calcium carbonate (prepared from calcium hydrate and carbonic acid water) containing in 20,000 parts of solution 1 part of platinum bichloride. The latter is reduced to the metallic state on heating the mantle in a gas flame. To the mantle and in contact with the upper end of the line of platinum, is fitted a gas-igniting meerschaum block, such as above mentioned. On turning on the gas, the igniting block and then the platinum line become red hot, communicating the heat to the minute quantity of platinum mixed with the lime; the lime thereupon becomes incandescent and ignites the gas.—H. B.

Incandescence Mantles, Impts. in. A. Kiesewalter, Limburg-on-the-Lahn, Germany. Eng. Pat. 15,963, July 5, 1897.

The patentee claims a process of preparing mantles by fusing "the oxides suitable for incandescence purposes" along with silicic acid and caustic potash or caustic soda. The glassy product is "dissolved in water" and organic fabrics are impregnated with the solution. "The incandescence material is then precipitated in the said fabric by means of a mixture of suitable acids and reagents, and the residual salts which are soluble in water, are washed out of the fabric." For instance, in using an "alkaline calcium water-glass solution," the calcium compound is to be "precipitated" within the fibre by means of sulphuric acid, or a mixture of oxalic and hydrochloric acids.—H. B.

Fuel, Impts. in or relating to the Manufacture of. P. R. de F. d'Humy, Liverpool. Eng. Pat. 20,156, Sept. 11, 1896.

Cheap or waste fibrous combustible materials, such as shavings, sawdust, peat, pine and fir cones, resinous bark,

refuse from cotton and linen mills, &c., are mixed with a mineral oil, ozokerite, naphthalene, an alkali, as caustic soda, and an oxidising compound, as sodium nitrate, chlorates, bichromates, &c., and the whole is heated to about 600°—700° F.—R. S.

Promoting the Combustion of Smoke or Inflammable Gases in Steam-Boiler and other Furnaces, New or Improved Means for. H. Whitaker, Newent, Gloucestershire. Eng. Pat. 21,329, Sept. 26, 1896.

Resting on the fire bars at the rear part, and at right angles thereto, are a number of parallel vertical metal plates, which become intensely hot, and between which a large supply of air from the ash-pit passes, to supply the desired quantity of heated air wherewith to consume the smoke or inflammable gases.—R. S.

Treating Smoke for the Extraction of the Carbon and other Residual Products resulting from Imperfect Combustion, Impts in the Mode, Means, and Apparatus for. W. M. Ward, Handsworth. Eng. Pat. 262, Jan. 5, 1897.

This invention consists in interposing a washer or scrubber in the flue, the said scrubber being formed of a chamber wherein a horizontal shaft, carrying beaters or arms, is caused to revolve in such a manner as to force the smoke into and through a shallow layer of water. This chamber has a perforated cover, through which the smoke has to pass before reaching the chimney, and which is supplied with water from a perforated pipe.—R. S.

Gas-Retort Settings, and Regenerator Furnaces for the same; Certain Impts. in the Construction of. G. E. Stevenson, Claremont, Crumpsall, Manchester. Eng. Pat. 22,887, Oct. 15, 1896.

1st. The gas-producer arch and the side walls thereof are built forward of and through the front wall of the retort setting, and a firing hole is formed in this projecting part. The end wall of the producer is built separate from the front wall of the retort setting, so as to fill in the end of the arch between the side walls and to receive the fire-grate fittings.

2nd. The regenerator flues, both for air and for waste gases, are formed of vertical fire-clay tiles, each one the full depth of each flue, having curved sides, so that the flues become narrower at the top and bottom, and thereby afford a wider bearing surface for the fire-clay top and bottom covering tiles. The latter are either provided with a projecting lug, fitting between the edges of the vertical tiles, or are made flat, in which case thin slabs are used to fill in the same space.—R. S.

Hydrocarbons, Apparatus for Vaporising; applicable to Engines worked by Gas, Petroleum, Alcohol, and the like. E. Petréano, Paris. Eng. Pat. 23,270, Oct. 20, 1896.

The object of this invention is to prepare a homogeneous explosive mixture of hydrocarbons (or alcohol) and air, outside the engine cylinder, and to prevent the access to the latter of any of the liquid substances used.

This is effected by introducing the air and liquid to be vaporised into an annular vertical chamber surrounding and deriving heat from either the cylinder or the exhaust pipe therefrom, and containing a number of superimposed cones. These are fixed alternately to the outer and inner walls of the chamber, and thus constitute a series of baffles facilitating the production of a uniform mixture. The resulting gas passes into a bell or chamber, whence it is drawn by the stroke of the engine piston, whilst the unvaporised oil is drained off through a pipe in the bottom of the vaporising chamber.

By applying the same apparatus to the ordinary gas engine, it is claimed that in comparison with the usual method of mixing the gas and air in the engine cylinder, an instantaneous and more perfect combustion of gas, and therefore increased efficiency, is ensured. Moreover, the water jacket of the cylinder can be dispensed with, the entire heat due to the explosion being confined to the compression chamber; and the introduction of steam into the cylinder to reduce the temperature of the explosion can be effected without risk of decomposition and the consequent corrosion of the cylinder.—C. S.

Oil, Vaporising; Impts. in or connected with Apparatus for. C. Crastin, London. Eng. Pat. 24,793, Nov. 5, 1896.

To prevent surplus oil entering or remaining in the vaporising chamber, the oil is allowed to fall from a sight-feed device on to a perforated disc situated below the air inlet, the mixture of air and vapour being drawn off through a lateral tube to the vaporising chamber, whilst the excess of oil runs down into a collecting vessel. The vaporising chamber consists of the annular space between two vertical concentric tubes, and is closed at both ends, except for the inlet pipe below and the outlet above, leading to the motor, &c. Heat is applied to the chamber by the flame of a lamp situated below the open end of the inner tube, and radiation is prevented by a case lined with insulating material. —C. S.

Gas [Oxygen, Carbonic Acid, &c.] Absorbing Apparatus for ascertaining the Quantity of a particular Gas in a Gaseous Mixture [Smoke Gas, &c.]. M. Arndt, Aachen, Germany. Eng Pat. 25,762, Nov. 16, 1896.

See under XXIII., *page* 935.

Regenerative Furnaces, Certain Impts. in or connected with. J. W. Hall, Bilston. Eng. Pat. 26,937, Nov. 27, 1896.

This invention consists in providing the ordinary Siemens regenerative furnace having four regenerators, with two additional regenerators, which are so connected by means of valves of the usual kind, that the whole of the products of combustion, after leaving the usual pairs of regenerators on the exhaust side of the furnace, are passed through one of the additional regenerators, while the air passes through the other one before entering the usual air regenerators. These regenerators are reversed in the usual manner. —R. S.

Carbonizing, Charring, or Coking Combustible Substances, An Improved Process for. A. Basch and H. Graepel, Budapest. Eng. Pat. 5698, March 3, 1897.

Combustible substances which heretofore have been found to be either entirely incapable of coking, or which could only be coked with difficulty (as for example Transylvanian Crown-coal), are mixed with products obtained from the distillation of tar. They can then be coked in a comparatively short time. In the above case, the addition of one-half per cent. of naphthalene enables the coking to take place at about 275° C.; but the temperature of distillation varies according to the nature of the fuel.—R. S.

Bunsen Gas Burners, Impts. in. H. Marshall, Edinburgh. Eng. Pat. 238, Jan. 5, 1897.

Instead of the gas entering the mixing tube through a central nozzle, it is introduced through one or more apertures at the side, or through an annular slit with an upward inclination. The air tube is left open at the base, and may be closed when desired by a sliding cover working on a pivot. —C. S.

Blue Flame, Impts. in Apparatus for Generating a Vaporous or Gaseous Mixture from Hydrocarbon-containing Liquid and Water for the Production of. F. Altmann, Berlin. Eng. Pat. 15,261, June 25, 1897.

In this lamp the water and the hydrocarbon liquid are contained in separate reservoirs, and are conveyed thence—either by capillary attraction or hydrostatic pressure—into vessels where they are vaporised by the heat from a flame below. The resulting gases ascend into a mixing chamber communicating direct with the main burner. The relative heat applied to the liquids may be regulated by altering the position of the heating flame. It is claimed for the invention that, the mixing chamber and burner being so close together, condensation of the vaporised substances is precluded.—C. S.

Illuminating Gas, Carburetting: Impts. relating to, and to Apparatus therefor. E. F. J. C. Bauweraerts, Brussels. Eng. Pat. 15,369, June 26, 1897.

The inventor finds that the most suitable substances for mixing with acetylene and mixtures of the same with ordinary illuminating gas in order to produce a smokeless flame, are such volatile bodies containing carbon, hydrogen, and oxygen, as ethyl oxide, acetic acid, acetone, and the alcohols, preferably methyl alcohol. This latter also frees the gas from fatty and other impurities, the addition of a little sulphuric acid also ensuring the removal of hydrogen sulphide.

The apparatus for mixing and "enriching" the gases, consists of a funnel-shaped mixing chamber into which the gases are delivered, and which discharges them into a vessel below, where they pass over the surface of a layer of methyl alcohol and are thereby "enriched," in the sense that "the hydrogen of the methyl alcohol will increase the heat of combustion, and be aided by the oxygen, thus effecting the complete combustion of the carbon, and thereby increasing the luminosity of the flame." The scum of impurities floats on the alcohol, and is drained off through a suitable overflow pipe into a closed receptacle, whilst the gas is led through a main to the burners. The admission of acetylene to the mixing chamber (and thus its proportion there) is suitably regulated.—C. S.

Petroleum Blue-Flame Burners for Incandescent Lighting and Heating Purposes, Impts. in. M. Bernstein, Berlin. Eng. Pat. 17,927, July 30, 1897.

In order to shorten the time requisite for gasifying the oil when the lamp is first lighted, and thereby to minimise the deposition of soot on the mantle, the inventor mounts above the wick a flame divider and a circular hollow chamber, wherein a portion of the air, ascending the space enclosed by the wick, is superheated and is delivered to the inner side of the flame. A supply of heated air to the outer side of the flame is secured by a perforated metal cylinder (of sheet metal) surrounding the flame, and also serving as a support for the incandescent mantle. A set of baffles is mounted in the central air space enclosed by the wick duct, in order to impart a whirling motion to the incoming air, and thus facilitate its mixture with the upper gases of combustion.—C. S.

Gas suitable for Heating, Motive Power, or Lighting Purposes, by the aid of Volatile Hydrocarbons; Impts. in and relating to Apparatus for Producing. W. P. Thompson, London. From A. Julien, Brussels. Eng. Pat. 18,005, July 31, 1897.

The essential part of the apparatus is the mixing carburettor, a cylinder filled about one-third full with the carburetting liquid employed. The mixing is effected by the rotation of a number of perforated discs or paddles mounted on a shaft and partly covered with an absorbent material and sponges. Into this vessel the air to be enriched is forced from a compressor, either direct or *viâ* a gasholder containing some carburetting vapour, and is intimately and evenly mixed with vapour by the movement of the paddles.

For heating purposes, the discharge pipe of the carburettor may lead direct to the furnace and there be provided with a burner nozzle. It is also claimed that the apparatus will produce a superior explosive mixture for use in automotors, as well as an illuminating gas burning with a smokeless flame.—C. S.

Acetylene Gas, Impts. in Apparatus for Generating. W. J. Mackenzie, Glasgow. Eng. Pat. 23,752, Oct. 26, 1896.

A water-jacketed bell linked to its containing tank, covers a carbide container to which a pipe with cock operated by the movements of the bell of the gasholder gives access to the water in the tank of the latter. A spring brake on the operating lever prevents the cock being entirely closed until the gasholder is nearly filled with the acetylene generated. A device for automatically shutting off an exhausted generating apparatus and putting in action a fresh apparatus is described.—J. A. B.

Acetylene, Impts. in Apparatus for Automatically Producing and Storing. E. Chesnay, Dijon; L. Pillon, Dijon; and C. Bertolus, St. Etienne, France. Eng. Pat. 25,236, Nov. 10, 1896. (Under Internat. Convention.)

Carbide is fed into a vessel containing water, through a shoot to which it is supplied from a hopper by means of an Archimedean screw, the rotation of which is controlled by the balance weight of a gasholder bell, into which the gas passes as generated. The screw is rotated by any mechanical motor under the control of the balance weight.—J. A. B.

Acetylene Gas, Improved Apparatus for the Production of. A. Fournier, Paris. Eng. Pat. 25,488, Nov. 12, 1896.

A hopper for carbide is attached to the crown of the bell of a gasholder. The lower constricted end of the hopper is closed by a conical valve, which opens when an attached rod strikes the base of the holder, owing to the sinking of the bell, and closes when the bell again rises. When the valve is open, carbide passes from the hopper into the water in the tank of the holder and generates acetylene.—J. A. B.

Acetylene Gas, Impts. in and connected with Apparatus for the Production and Utilisation of. J. Vaughan-Sherrin, London. Eng. Pat. 26,897, Nov. 26, 1896.

A fixed or rotating water tank is connected with a number of carbide containers which are put in communication with it in turn by means of taps operated automatically or at a signal given by the gasholder when collapsed to a certain degree.—J. A. B.

Acetylene Gas, Impts. in or connected with Apparatuses for Producing. W. P. Thompson, Liverpool. From L. L. H. Gérard, Paris. Eng. Pat. 29,188, Dec. 19, 1896.

The orifice at the bottom of a carbide receptacle placed above a water reservoir is opened and shut by valves on a rod, moved by a diaphragm exposed on one side to the pressure of the acetylene generated, and on the other to the atmospheric pressure. When the valve is open, crushed carbide falls into the water. Or the diaphragm may, by means of a pawl and ratchet, rotate a recessed drum, which discharges the carbide into the water.—J. A. B.

Acetylene, Impts. in Apparatus for the Manufacture of. P. De Résener and H. Luchaire, both of Paris. Eng. Pat. 29,320, Dec. 21, 1896. (Under Internat. Convention.)

Improvements on the apparatus claimed under Eng. Pat. 24,440, Nov. 2, 1896 (this Journal, 1897, 790), are described. They consist of a regulator to proportion the flow of water to the carbide receivers to the consumption of gas; and a pipe with cock to cause the water to flow to a second receiver when the first is exhausted.—J. A. B.

Acetylene, An Improved Apparatus for Manufacturing. P. de Réseuer, Paris. Eng. Pat. 30,037, Dec. 30, 1896.

A carbide receiver is placed above a water tank, the cover of which contains a conveyor screw, which passes the carbide into the tank when a crank is turned. Taps and connections for charging either a gasometer or high-pressure receivers are provided.—J. A. B.

Acetylene from Calcium Carbide and Water, Impts. in and connected with Apparatus for the Production of. T. Thorp, Whitefield, and T. G. Marsh, Manchester. Eng. Pat. 19,823, Aug. 28, 1897.

A bell gas holder, by means of a rod passing within the inlet pipe, operates the valve between a carbide chamber and a water tank. Granulated carbide passes from the chamber to the tank when the holder descends. A chamber with interlocking lid and outlet valve to the carbide chamber, is provided for recharging the latter. The interlocking device is described in detail.—J. A. B.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Petroleum Springs in Italy. T. Tamarelli. R. Instituto lombardo di Scienze e Lettere di Milano, May 20, 1897. Chem. Zeit. 21, [60], 603.

According to Prof. Tamarelli, petroleum has been found in a boring recently made near Salsomaggiore. It issues from clefts, which traverse the saliferous marls in planes at right angles to the dip, and is mixed with brine (which is found in all borings in the district). The petroleum, from a depth of 618 metres, is accompanied by quantities of gaseous hydrocarbons, which cause intermittent sputterings in the stream. Further discoveries of oil are to be expected from the geological nature of the ground.—J. T. D.

Petroleum, Purification of. Badische Anilin und Soda-industrie (Ger. Pat. 93,702). Zeits. für angew. Chem. 1897, [19], 629.

It is found that the sulphonated products formed and left dissolved in the oil when treated with fuming sulphuric acid, can be removed by agitation with a small quantity of ordinary (75 to 100 per cent.) H_2SO_4 previous to the alkali treatment, thus effecting a degree of purification unattainable by the ordinary method. The quantities given as most suitable in the examples cited in the specification are, in the case of—

Galician Crude Benzine (sp. gr. 0·730).—Ordinary H_2SO_4 to remove water, 0·25 per cent.; fuming acid treatment, 0·5 per cent. of H_2SO_4 (60 per cent. free SO_3); after-washing, 0·25 per cent. of ordinary (92 per cent.) H_2SO_4.

Cracked Galician Petroleum (sp. gr. 0·825).—Freed from water, then mixed with 1 per cent. of 20 per cent. fuming H_2SO_4, followed by $\frac{1}{2}$ per cent. of ordinary (94 per cent.) acid.

American Machine Oil (sp. gr. 0·905).—Fuming acid treatment with 3 per cent. of H_2SO_4 (5 per cent. free SO_3); after-treatment with 2 per cent. of (92—96 per cent.) ordinary acid.

Russian Lubricating Oil (sp. gr. 0·930).—Treated with 4 per cent. of 5 per cent. fuming H_2SO_4, followed by 1 per cent. of ordinary concentrated acid.

Galician Paraffin Press Oil (cracked distillate, sp. gr. 0·902).—Fuming acid treatment with 7 per cent. of 10 per cent. fuming H_2SO_4: after-treatment with 2 per cent. of 85 per cent acid.

The treatment is claimed as efficacious in the case of all distillates, cracked or uncracked, though more particularly adapted for heavy oils and cracked distillates.—C. S.

Petroleum, Contribution to the Knowledge of the Chemical Behaviour of. Action of Sulphuric Acid. R. Zaloziecki. Zeits. für angew. Chem. 1897, [18], 587—592.

A number of samples of Galician and Russian crude petroleums were treated by agitation with an equal volume of concentrated sulphuric acid at room temperature, the same treatment being also applied to samples of the same oils after division into four fractions of distillate between 150° and 300° C., and a residue boiling above 300° C. The proportion of the lighter fractions absorbed by the acid, was comparatively small, ranging between 0·20 and 1·05 per cent., whereas from 11·80 to 27·20 per cent. of the residue above 300° C. was absorbed. On the other hand, the difference between the amount of heavy constituents present in the crude oil, and in the same oil after the above acid treatment (after deduction of the proportion absorbed by the acid), indicated an increase in these heavier components of from 8·04 to 12·97 per cent. This increase is most easily explained as the result of polymerisation of the unsaturated hydrocarbons, and this confirms the assumption of Engler and Jezioranski (this Journal, 1896, 191) respecting the composition of the heavier fractions of petroleum.

In view of these results the author does not consider that such a treatment would have any beneficial practical result on the medium fractions of petroleum distillate, since any consequent increase in the yield would be chiefly confined to benzine. On the other hand, in the case of lubricating oils, a considerable proportion of the intermediate oils is converted, by polymerisation (in the treatment of the crude oil), into heavier and more viscous products, resulting in an increased yield of cylinder- and similar oils, the quality of the distillate being also improved by the elimination of the lighter impurities which ordinarily increase the difficulty of distillation.

From experiments made with a view to the utilisation of this acid treatment in practice, the author finds the most suitable quantity of acid to be 5 per cent., larger amounts effecting but a slight extra separation of acid tar, whilst reducing the consistency of the same. The latter result also ensues in proportion as the percentage of monohydrate in the sulphuric acid used, decreases, acid of 66° B. producing a thicker and harder tar than weaker grades of acid. The difficulty of separating this tar from the oil prevents the application of this treatment to residuum.

The purified crude oil requires to be neutralised before distillation in order to avoid the decomposition of the residual acid therein. Owing to the readiness with which troublesome emulsions are formed on the addition of alkali, this operation is far from easy, and involves a protracted period of rest for the deposition of sediment. On the other hand, no final washing with water is required.

The purified crude oil is almost entirely free from pitchy, asphaltic, and resinous matters, and is much lighter in colour, the fluidity being also greater; the distillates require but little purification, and the residue left after the petroleum fractions have passed over, will form a very good consistent lubricant without any further treatment than distillation by superheated steam.

The author turned his attention to the utilisation of the acid tar for the manufacture of lac in the same manner as the acid tar from the ordinary refining process, but found that the resulting product is different, more nearly resembling true asphaltum lac. This circumstance strengthens his opinion that the product of the action of sulphuric acid on crude petroleum is, when freed from acid, identical with natural asphaltum or bitumen.

This utilisation of the 10 to 15 per cent. of crude oil, otherwise wasted, should nearly or quite cover the cost of the acid treatment. It is, however, pointed out that the method is not a universal one, since not all petroleums can be advantageously worked up for asphalt and bitumen.
—C. S.

Petroleum: Modifications produced by Heat and Pressure. R. Zaloziecki. Zeits. für angew. Chem. 1897, [19], 619—623.

In these researches the behaviour of the oil towards sulphuric acid before and after distillation (or heating under pressure) was taken as an index of the alterations produced. The test consisted in shaking up equal volumes (50 c.c.) of oil and concentrated acid in a graduated tube and reading off the amount of oil absorbed at the end of 48 hours. In examining the effect of distillation, all the distillates up to 300° were, on cooling, united, returned to the retort, and filtered, this being considered preferable to testing the distillates and residue separately. The results obtained were as follows:—

Crude Oil from	Percentage Volume of Crude Oil absorbed by H_2SO_4 in the	
	Undistilled.	Distilled Condition.
	Per Cent.	Per Cent.
Sloboda Rungurska	26·8	23·09
Krosno	24·2	23·20
Schodnica	11·8	12·40
Balachani	25·2	21·60
Bibi Ejbet	27·4	22·00
Sabuntschi	22·6	18·60

the absorption (contrary to anticipation) being less after distillation than before, except in the case of the Schodnica oil. Nevertheless, as repeated distillations of the fraction

250°—300° C. of the Sloboda oil (as well as of the crude oil itself) showed, decomposition does occur, though its effects on the ratio of absorption by H_2SO_4 are masked by other phenomena, which the author attributes to polymerisation. In favour of this view the author cites the constant increase in the amount of residue obtained when a previously distilled crude oil is fractionated. This polymerisation occurs only during the first distillation—a circumstance testifying against the prevalence of a distillation temperature of 300° C. at the time the oil was in course of formation; and from one experiment it seems probable that temperatures above 200° C. are also out of the question.

The results obtained by heating crude oil free from water, in sealed tubes for 3 to 4 hours at 200° C. and 240°—250° C., viz.:—

Crude Oil from	Percentage Volume absorbed by H_2SO_4 in 48 Hours.		
	Ordinary Crude Oil.	Heated in Sealed Tube to 200° C.	Heated in Sealed Tube to 240°—250° C.
	Per Cent.	Per Cent.	Per Cent.
Sloboda Rungurska ...	26·80	27·06	20·63
Krosno	24·20	..	63·68
Balachani	26·20	32·90	30·86
Sabuntschi	22·60	..	68·06
Romani................	17·85	..	38·19

show a great increase in constituents soluble in the acid, except in the case of Sloboda oil, which was rich in paraffin (9 per cent.). The acid was added to the second and fourth oils in this table as soon as cooled after the heating was completed, but the other oils were left without acid until the second day.

From the above results the author concludes that only the petroleums poor in paraffin and consisting principally of naphthenes, are so changed by heat as to be partly modified by intermolecular alteration into unsaturated hydrocarbons, the temperature employed precluding the decomposition of the paraffin series. The presumption is that ring-naphthenes, *i.e.*, polymethylenes isomeric with olefines, are here converted into the latter bodies; but this explanation does not exclude the possibility of dynamic isomerism between naphthenes and olefines. At any rate, the conversion of the isomer of propylene—differing from propylene in its behaviour towards bromine and sulphuric acid—into true propylene by the influence of heat justifies the assumption that analogous modifications are possible among higher members of the naphthene or cyclo-olefine series.—C. S.

PATENTS.

Benzene, Toluene, and other Products from Mineral Oils and Liquid Tars; Impts. in Obtaining. J. Meikle, Maryhill, Glasgow. Eng. Pat. 23,619, Oct. 24, 1896.

This invention consists in operating on the oil in a retort containing coke or other suitable carbonaceous material and maintained at about red heat, the oil being injected by the aid of superheated steam. The gaseous mixture is led through pipes to suitable condensers, the benzene and toluene being thereby separated from the gas, which is then used for heating or illuminating purposes. The material employed may, for example, be an oil such as is obtainable from coal or coal-tar, and after all the benzene has been separated from it by the ordinary process. Oils very suitable for the purpose are light creosote oils or light tar oils.—D. B.

Benzol and similar Hydrocarbons, Improved Process for obtaining. [*Decomposition of Tar Oils by Heat.*] G. Schultz, Munich, Germany. Eng. Pat. 26,771, Nov. 25, 1896.

This invention relates to the production of benzene and similar hydrocarbons in considerably larger quantities than has been hitherto possible in the ordinary process of gas manufacture. According to the methods formerly employed, only comparatively small quantities of benzene, toluene, xylene, naphthalene, and anthracene are obtained, about 50 per cent. of the tar constituting pitch, and 40 per cent. oily residues. The latter are the oils remaining from the treatment of coal tar, after the removal of the hydrocarbons contained therein. By the present invention, these oils are conveyed into highly heated or glowing retorts or hot coke ovens, which are preferably provided with a suction appliance. The vapours, which, in consequence of the decomposition resulting thereby, contain aromatic hydrocarbons, such as benzene, toluene, and the like, are condensed in the usual manner.—D. B.

Orthotoluenesulphonic Chloride, Method of Producing and Purifying. W. Majert, Berlin, Germany. Eng. Pat. 6581, March 12, 1897.

Hitherto orthotoluenesulphonic chloride has been separated from the para compound by crystallising the former out of a mixture made in the cold. The liquid portion thus obtained, contains, as a rule, 70 per cent. of ortho- and 30 per cent. of para-chloride. These two chlorides cannot be separated by fractional distillation in a vacuum. It has, however, been found that almost pure orthotoluenesulphonic chloride can be obtained in the following manner:—About 30 to 40 parts by weight of the above mixture are distilled off in a vacuum. The distillate consists of almost chemically pure ortho compound. The residue is cooled, and from the same there are obtained by crystallisation from 10 to 12 parts of para compound. This is separated from the liquid and a further portion of ortho compound is distilled off, and so on. The residue is cooled to the ordinary temperature.—D. B.

IV.—COLOURING MATTERS AND DYES.

Indoïns, Note on the. L. D'Andiran. Bull. Soc. Ind. Mulhouse, 1897, 245—248.

The Indoïns are basic colouring matters prepared by combining diazo-safranines with β-naphthol. Their production was patented by Beyer and Kegel in 1885, but the patent was allowed to lapse, and in 1891 the Badische Anilin und Soda Fabrik again patented the process, including the treatment of the resulting compounds with acid in order to render them soluble. According to a later patent of the same firm, soluble dyestuffs are also obtained by repeated washing with water of the insoluble azo compounds. Various other patents, of which the author gives a *résumé*, and which differ from the above only in minor details, have been subsequently taken out.

In the first patent of the Badische Anilin und Soda Fabrik the transformation of insoluble bases into their soluble salts is spoken of, while in their second patent the insolubility is attributed to the presence of mineral matter, which is removed by repeated washing. According to a patent of Dahl and Co., insolubility is due to a combination of safranine-azo-naphthol with sodium. Analysis, however, reveals only slight traces of sodium; and, in the author's opinion, insolubility is caused here, as in many other cases, by the presence of a slight excess of alkali.

An excess of mineral acid also precipitates the colouring matter from its solution, the precipitate consisting of a mixture of mono- and diacid salts.

The Indoïns are sold in the form of black pastes or bronze-like powders, and frequently contain a slight admixture of Methylene Blue to increase the brilliancy of the colour. The shades which they yield on tannin-mordanted cotton are very similar to indigo blues, and are much faster to soap and to light than other basic colours. They can also be dyed on cotton without a mordant, but with less satisfactory results.—R. B. B.

Bismarck Brown. E. Taüber and F. Walder. Ber. **30**, 2111—2117.

Although one of the earliest of the artificial organic dyestuffs, the constitution of Bismarck Brown (Vesuvine, Phenylene Brown) cannot be considered as established, and probably none of the Bismarck Browns prepared on a manufacturing scale, or in the laboratory, are chemically identical products. According to Caro and Griess (in 1867),

Bismarck Brown consists of the hydrochlorides of three bases, of which triamidoazobenzene preponderates. More recently, doubt has been thrown upon this view, owing to the fact that the amount of nitrite required in the preparation of the brown on a large scale points to the molecule consisting of the residues of three *m*-phenylene diamines, connected by two azo groups. This leads to the assumption that phenylene-disazo-*m*-phenylene diamine is the chief constituent. There are, however, three formulæ possible for a substance having this composition. In order to convert the whole of the *m*-phenylene diamine into dyestuff, a weighed quantity of *m*-phenylene diamine hydrochloride was treated with sodium nitrite. The formation of the dyestuff is accompanied by a copious evolution of nitrogen, which is not due to an excess of nitrite, since a considerable amount of *m*-phenylene diamine is also present. The amount of nitrogen evolved corresponds to 29·1 per cent. of that contained in the *m*-phenylene diamine, since half the evolved nitrogen comes from the nitrite. The proportion of nitrite to *m*-phenylene diamine is 5 : 6. By taking the mono- instead of the dihydrochloride, no evolution of gas takes place, but when the reaction is finished, unchanged nitrite is found side by side with *m*-phenylene diamine, so that triamidoazobenzene is not the only product. In order to obtain a yield of dyestuff without any evolution of gas, the authors find that three molecular proportions of *m*-phenylene diamine, together with four molecular proportions of hydrochloric acid, require two molecular proportions of nitrite, but the product obtained is not homogeneous. In order to obtain triamidoazobenzene of undoubted constitution, acetyl-*m*-phenylene diamine was diazotised and combined with *m*-phenylene diamine, the acetylated dyestuff being finally hydrolysed. Thus prepared, triamidoazobenzene crystallises from hot water or benzene, in needles melting at 144° C. This product was not found in commercial Bismarck Browns, but only in that product obtained from three molecular proportions of *m*-phenylene diamine, four molecular proportions of hydrochloric acid, and two molecular proportions of nitrite. No triamidoazobenzene could be detected in the brown from *m*-phenylene diamine dihydrochloride or in a good commercial sample. The brown for this purpose, after basifying, was extracted in a Soxhlet's apparatus with benzene, 15 grms. of base giving 10 grms. of crystalline extract. The pure substance melts at 118° C., is almost insoluble in water (difference from triamidoazo benzene), and gives numbers on analysis corresponding to a disazo dyestuff containing three *m*-phenylene diamine radicles.—T. A. L.

Bismarck Brown. R. Möhlau and L. Meyer. Ber. **30**, 2203—2206.

The authors have commenced a research on the products obtained by the action of sodium nitrite on *m*-phenylene diamine known commercially as Bismarck Brown, Vesuvine, Phenylene Brown, and Manchester Brown (see also Täuber and Walder, Ber. **30**, 2111; see previous abstract). In order to test the statement of Caro and Griess that triamidoazobenzene is the chief product in the mixture obtained, the authors, in an analogous manner to Täuber and Walder, have prepared this substance in a pure state by the action of diazotised phenylene oxamic acid on *m*-phenylene diamine. The product so obtained, after crystallisation, forms groups of concentric prisms melting at 189° C. On hydrolysis with dilute sulphuric acid (100:7·5) they obtained a product which possessed all the properties already ascribed to triamidoazobenzene. When boiled with fused sodium acetate and acetic anhydride it yields the triacetyl compound melting at 264° C. Attempts to produce triamidoazobenzene exclusively, by the action of sodium nitrite on *m*-phenylene diamine were unsuccessful, the best results being obtained by reacting with two molecular proportions of *m*-phenylene diamine hydrochloride on one molecular proportion of sodium nitrite in presence of three molecular proportions of sodium acetate. The investigation of five commercial Bismarck Browns stated to be obtained from *m*-phenylene diamine, led to the result that all contained a product having the composition of a triamidoazobenzene, although the amount present was small in proportion to the bulk of the product.—T. A. L.

Nitronaphthalene, The Commercial Preparation of. W. H. Krug and J. E. Blomen. J. Amer. Chem. Soc. 1897, **19**, 532.

See under XXII., *page* 933.

Hæmateïn, The Estimation of, in Logwood Extracts. Agiot. With Report by E. Jaquet and O. Michel. Bull. Soc. Ind. Mulhouse, 1897, 250.

See under XXIII., *page* 938.

Indigo, Assay of. A. Brylinski. Bull. Soc. Ind. Mulhouse, 1897, 331.

See under XXIII., *page* 938.

PATENTS.

Dyes, Impts. in the Production of. L. P. Marchlewski, Manchester, E. S. Wilson, Strood, and E. Stewart, London. Eng. Pat. 21,498, Sept. 28, 1896.

According to Eng. Pat. 24,418 of 1895, a substance has been separated from crude cotton-seed oil which can be obtained from glacial acetic acid and alcohol in yellow crystals, and is easily soluble in alcohol, ether, boiling acetic acid, and alkalis, but insoluble in water. This proproduct has been termed *Gossypol*, and can be used, according to the specification, in two ways, for the production of dyestuffs, according to the following methods. *Oxygossypol Brown* is obtained by dissolving 20 kilos. of Gossypol in a solution of 15 kilos. of caustic soda in 300 litres of water, and blowing a current of air through the solution for several hours, until no further increase in the tinctorial value takes place. The dyestuff produced gives brown shades on mordanted wool or cotton, and when a sample so dyed attains a maximum in strength, the current of air is stopped and the solution is acidified, when the dyestoff is precipitated. Another dyestuff may be obtained from Gossypol by heating 10 kilos. of it with 50—100 kilos. of crystallised sodium sulphide, with or without the addition of sulphur. The temperature is maintained for some time at 120° C. and is then raised to 250° C. for several hours. The dyestuff produced gives brown shades on unmordanted cotton, and may be employed in the crude state, or purified by dissolving in sodium sulphide and precipitating with an acid. The shades may be intensified by passing the dyed goods through solutions of metallic salts, such as copper sulphate, potassium bichromate, &c. The products obtained according to the specification may also be used for printing. —T. A. L.

Blue Dyestuff suitable for Dyeing Wool, Production of a. S. Pitt, Sutton. From L. Casella and Co., Frankfort-on-Main, Germany. Eng. Pat. 24,134, Oct. 29, 1896.

Although the diazo derivatives of the periamidonaphthol sulphonic acids are very sensitive to alkalis, they will combine in weak acid solutions to form azo dyestuffs. For instance, according to Eng. Pat. 12,556 of 1896 (this Journal, 1897, 531), a technically useful dyestuff has been obtained by combining diazotised 1.1′.3.3′-amidonaphthol disulphonic acid with alkylnaphthylamine. The patentees now find that when the same diazo compound is combined with 1.5-amidonaphthol, a blue dyestuff is obtained, very fast to light, and giving level shades :—32 kilos. of 1.1′.3.3′-amidonaphthol disulphonic acid are diazotised with 35 kilos. of hydrochloric acid and 7 kilos. of sodium nitrite, and the diazo compound is filtered off, washed with brine, and suspended in 500 litres of water. To this is then added a solution containing 16 kilos. of 1.4′-amidonaphthol, dissolved in 15 kilos. of hydrochloric acid and 300 litres of water. Finally, a solution of 30 kilos. of sodium acetate is run in, when, after a short time, a clear blue solution is obtained. After neutralisation with 20 kilos. of sodium carbonate, the dyestuff is salted out. In place of the 3.3′, other disulphonic acids, such as the 4.3′, 3.2′, or 2′.3′ may be used, or the 4-sulphonic acid and analogous dyestuffs are also obtained if the 1.4′-amidonaphthol be replaced by the 1.4′.2′ or the 1.4′.3′-amidonaphthol sulphonic acid.—T. A. L.

Sulphinic Acids of the Aromatic Series, The Manufacture or Production of. H. E. Newton. From "The Farbenfabriken vorm. F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 26,139, Nov. 19, 1896.

By acting on the diazo compound of an aromatic amine with copper powder in presence of sulphurous acid, nitrogen is evolved, and the corresponding sulphinic acid is produced. For example, 1 kilo. of aniline is dissolved in 15 litres of water and 3 kilos. of concentrated sulphuric acid, and diazotised in the usual manner. The diazo solution is then added with rapid agitation to an excess of an aqueous solution of sulphurous anhydride to which 6 kilos. of copper powder have been added. A rapid evolution of nitrogen takes place, and when the diazo compound has disappeared the reaction is finished. The sulphinic acid produced can be extracted with ether, and has the properties of benzene sulphinic acid, melting at 84° C. 1 kilo. of 1.4-naphthylamine sulphonic acid is mixed with 3 kilos. of concentrated sulphuric acid and 15 litres of water, and diazotised in the usual manner by means of sodium nitrite. A current of sulphurous acid is then passed in whilst carefully cooled, and subsequently 6 kilos. of copper powder are added. When the evolution of nitrogen has ceased, the formation of the sulphinic acid is complete. After making alkaline with sodium carbonate, the sodium salt is separated by means of salt. The new acid is easily soluble in water, and crystallises therefrom in leaf-like plates. The barium salt is a flaky, white precipitate, sparingly soluble in water, and is obtained by adding barium chloride to a solution of the sodium salt. On treating the new acid with potassium permanganate, it is converted into 1.4-naphthalene disulphonic acid.—T. A. L.

Azo Colouring Matters [Violet, Blue, Brown], Production of New. W. H. Claus, A. Rée, and L. Marchlewski, Manchester. Eng. Pat. 26,210, Nov. 20, 1896.

According to Eng. Pat. 18,020 of 1896 (this Journal, 1897, 676), dyestuffs are obtained from the azo compound produced by combining diazotised γ-amidonaphthol sulphonic acid with itself in an alkaline solution. The present specification describes dyestuffs which are formed from the azo compound produced in an acid solution. These products have the following general formula :—

$$X.N_2.D.N_2.G.2.N_2.D.N_2.X,$$

where D is the radicle of a tetrazotisable *p*-diamine, X is an amine, phenol, amidophenol, naphthol, amidonaphthol, or a sulphonic or carboxylic acid thereof; N represents nitrogen atoms, and G.2 an amido-azo compound, produced by combining diazotised γ-amidonaphthol sulphonic acid with itself in an acid solution. The combinations take place according to different methods, of which the two following are the most general:—(1.) 2 mols. of a tetrazotised *p*-diamine are combined in an alkaline solution with 1 mol. of the amido-azo compound G.2, and the intermediate compound is allowed to act on 2 mols. of an end component. (2.) 1 mol. of a *p*-diamine is combined in an acid or alkaline solution with 1 mol. of an end component. 2 mols. of the intermediate conpound obtained are then allowed to react with 1 mol. of the amido-azo compound G.2. The following example illustrates one of the methods by which dyestuffs may be obtained. The tetrazo solution from 36·8 kilos. of benzidine is poured into a cold solution of sodium carbonate so that the whole remains slightly alkaline, and to this is added 52·2 kilos. of γ-amidonaphthol sodium sulphonate. When the formation of the intermediate compound is complete, 200 litres of water containing 39 kilos. of sodium carbonate and 56·9 kilos. of the o-amido compound (from diazotised γ-amidonaphthol sulphonic acid and amidonaphthol disulphonic acid H in an acid solution) are added, and the whole is stirred for some time and finally heated to 50—80° C. The dyestuff produced gives reddish black-blue shades on unmordanted cotton. It may be diazotised either on or off the fibre, and on combination with *m*-tolylene diamine, resorcinol, or β-naphthol gives dark-blue and violet black-brown shades. —T. A. L.

Colouring Matters (Greys, Blacks) Suitable for Dyeing Unmordanted Cotton, Impts. in the Manufacture of. A. Ashworth, Bury. Eng. Pat. 18,762, Aug. 13, 1897.

The dyestuffs referred to are obtained by the action of sulphur and sodium sulphide on (1) aromatic nitro, amido, or nitro-amido hydroxycarboxylic acids; (2) a mixture of any of these acids with substances such as phenols or naphthols, nitro- or nitrosophenols or naphthols, amidophenols or naphthols, sulphonic acids of the foregoing, sulphonated or nitrosulphonated compounds of the benzene or naphthalene series, diamido benzenes and naphthalenes or their sulphonic acids, cane sugar and glucose; (3) azo dyestuffs of the Alizarin Yellow type (action of diazotised nitraniline on salicylic acid), or such containing as one component an amido or nitro-amido hydroxycarboxylic acid. The following is an example of the method employed:—A mixture of 20 kilos. of nitrosalicylic acid and a concentrated solution of 60 kilos. of sodium sulphide is heated for 2 hours at 125° C., when 16 kilos. of sulphur are added and the temperature is increased to about 200° C., at which it is maintained until the melt becomes solid. The product is easily soluble in water, and the solution dyes cotton grey to black shades, which may be intensified by treatment with oxidising agents.—T. A. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

Oxycellulose. L. Vignon. Comptes Rend. 1897, **125**, [12], 448.

See under VI., *page* 908.

PATENTS.

Jute Fibre, Method for Improving the Colour of Raw. C. O'Brien and J. Shearer, Dundee. Eng. Pat. 20,737, Sept. 19, 1896.

The jute is immersed for one minute in dilute acid—phosphoric, sulphuric, hydrochloric, or oxalic—and then dried in a hydro-extractor or by other means.—R. B. B.

Waterproofing Silk, Cotton, Wool, and other Threads, and Fabrics made therefrom, Impts. in and relating to. W. C. Kipling and E. Arnold, Sudbury, Suffolk. Eng. Pat. 22,197, Oct. 6, 1896.

The waterproofing solution is prepared by dissolving 3 oz. of aluminium sulphate, 4 oz. of lead acetate, and ½ oz. of tannin in 1 gallon of distilled water, and consists therefore essentially of acetate of alumina. The advantages claimed for its use are decreased cost, and freedom from odour; and it is stated that the waterproofing is in no way noticeable.—R. B. B.

Nitrocellulose, A New or Improved Solvent or Solvents for, whereby Improved Solutions are obtained. R. W. Strehlenert, Stockholm. Eng. Pat. 22,540, Oct. 10, 1896.

Artificial silk, when prepared in the ordinary way from cellulose, has a marked tendency to absorb water, and when in a wet condition, its strength is reduced by about 90 per cent. The patentee claims the process for the production of artificial silk which shall be proof against the action of water, consisting in either adding to or mixing with the solvent (ethyl alcohol, methyl alcohol, ether, &c.) for the nitrocellulose, before drawing out the threads, formic aldehyde, acetic aldehyde, paraldehyde, benzaldehyde, or other substances of the same group, to the extent of as much as 15 per cent. of the weight of nitrocellulose, or in treating the silk, after being spun, with one of the abovementioned substances.—A. S.

Felt, Impts. relating to, and Process for its Production. G. Kreuter, The Hague, Holland. Eng. Pat. 17,541, July 26, 1897.

This invention consists in the manufacture of a felt composed of silk and wool fibres. Silk rags are washed, carbonised, disintegrated, and carded by the methods employed for woollen rags, and subsequently treated with alum and manganese ore. A treatment with oil and soap next follows for the purpose of cleansing and opening the

fibres. The material thus obtained, is mixed with natural or recovered wool in the mixing machine, about 30—40 per cent. of silk fibre being employed. The mixed material is then made into felt in the usual manner.

The advantages claimed for this felt are, that it is softer and more delicate than woollen felt, possesses a finer felt or "grip," and receives a very high gloss, whilst the hat, &c., does not need such high pressure in finishing as it otherwise would, and can be produced out of less material. Further, it is stated that the shape and appearance are unaffected by rain.—R. B. B.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Oxycellulose. L. Vignon. Comptes Rend. 1897, **125**, [12], 448—450.

The author describes the preparation and some of the properties of oxycellulose. Pure cellulose was treated for half an hour successively in the following four baths (aqueous solutions):—(*a*) 1 per cent. Na_2CO_3 at 100° (30 minutes at 100° C.); (*b*) 1 per cent. NaOH, for 30 minutes at 100°; (*c*) 0·1 per cent. solution of HCl (of 22° B.) for 30 minutes in the cold; and (*d*) 1 per cent. Na_2CO_3 for 30 minutes in the cold. After this treatment the cotton was washed with boiling distilled water, alcohol, and then dried. It lost about 10 per cent. of its weight during the process. The effect of several oxidising agents was tried, but a mixture of hydrochloric acid and potassium chlorate gave the best results.

Preparation.—30 grms. of the purified cotton were introduced into a boiling solution of 150 grms. of potassium chlorate in 3,000 c.c. of water, and then 125 c.c. of hydrochloric acid (of 22° B.) were gradually added. The liquid was kept near the boiling point for an hour, decanted, washed by decantation with distilled water until free from acid, then with alcohol, and dried in the air, and finally on filter-paper and porous tile. The white powder thus obtained possesses the following properties:—

Properties.—Under the microscope, oxycellulose has the appearance of very short filaments. It turns yellow at 100° C., is insoluble in neutral reagents, and is coloured blue by iodine and sulphurous acid; the coloration being more rapid and distinct than in the case of cellulose itself.

From the results obtained on fusion with potash at 180° C. it appears that oxycellulose is a mixture of 75 per cent. of oxycellulose, and 25 per cent. of cellulose. The heat of combustion varies between 4,133 and 4,124, that of cellulose being 4,224—4,190. The heat disengaged on immersing 10 grms. of substance in normal potash at 13° C. was:—

	Per 100 Grms.
Cellulose	0·74 cal.
Oxycellulose	1·30 „

The following quantities of dyestuff, as measured by the colorimeter, were taken up by 1 grm. of the substance on dyeing for one hour at the boiling point:—

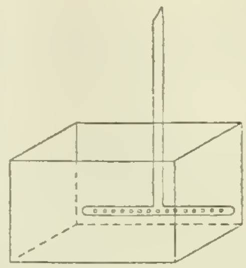
——	Safranine.	Methylene Blue.
	Grm.	Grm.
Cellulose	0·000	0·002
Oxycellulose	0·007	0·006

Oxycellulose and also the yellow liquid obtained on extraction with potash act as powerful reducing agents on Fehling's solution. The substance remaining behind, after extracting with hot potash, possesses no reducing properties. Since oxycellulose gives Schiff's reaction with magenta and sulphurous acid, it appears to be of an aldehydic nature.—J. S.

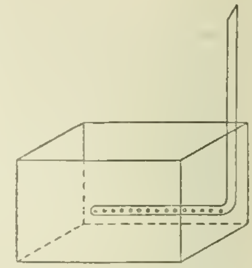
Heating Apparatus for Dye Vessels. W. Stermer. Färber Zeit. 1897, **8**, [11], 165—166.

Of three forms of heating apparatus shown in the figures, used for the purpose of heating dye-baths with directly-introduced steam, it is known that form I. is unsatisfactory, owing to the baths becoming more rapidly heated at the end where the steam enters the horizontal pipe—a circumstance which leads to irregular dyeing. Form II. has recently been recommended (Oesterr. Wollen- und Leinen-Ind., **16**, No. 17). The author, however, prefers arrangement

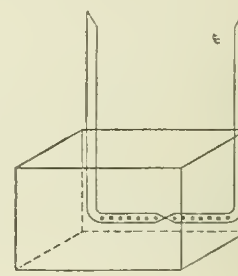
I.



II.



III.



III., which he has used for some time with satisfactory results. In this the main steam-pipe is divided into two branches as shown, and care is taken that the perforations in the horizontal pipes are of exactly the same size, and that, in working, they are kept free from threads, lint, dyewoods, &c.

The author, further, points out the necessity in introducing the dyestuff and mordant solutions, of distributing them regularly at the back of the steam-pipe so that they may be diffused evenly into the bath.—E. B.

Plumbite of Soda [Na_2PbO_2], with and without Glucose, The Comparative Mordanting Power of. A. Scheurer. Bull. Soc. Ind. Mulhouse, 1897, 258—260.

In 1894, Bonnet stated that the power of caustic soda to dissolve lead oxide is increased by the addition of glucose, and the mordanting power of the plumbite of soda thereby improved. The present author finds, however, that although the first of these statements is correct, yet, when a solution containing glucose is compared with one not containing this addition, by mordanting samples of cotton with each and passing into potassium bichromate solution, the colour is of equal depth in both cases. The percentage of lead in the solution containing glucose was 9·1; in the other, 5·4; and the author therefore concludes that the additional amount of lead brought into solution in the presence of glucose cannot be fixed on the fibre. He adds, however, a statement of Bonnet, that the advantage of the plumbite of soda containing glucose, consists in its less alkalinity compared with the ordinary plumbite.—R. B. B.

Organic Solvents in Printing [with Induline Dyestuffs]. C. Gassmann. Bull. Soc. Mulhouse, 1897, 237—244.

The author has compared the use of a large number of derivatives of glycerin as solvents for the Indulines. Many of his results will be found described in a previous paper (Monit. Scient. 1896, **10**, 348; this Journal, 1896, 584). No good results are obtained with derivatives containing the aldehyde group COH. The most useful solvents are the glycerides of levulinic acid and of tartaric acid, and their acetyl derivatives, these compounds having a greater solvent power for Indulines than is possessed by acetin. The glyceryl derivative of tannic acid is not suitable, owing to precipitation of tannate of Induline. The above solvents are not found useful in the case of Rosindulines.

—R. B. B.

Dyeing with Cotton Substantive Dyestuffs at Low Temperatures. E. Grossmann. Färber Zeit. 1897, **8**, 141, 166—167.

It is usually recommended that dyeing with cotton substantive dyestuffs should be performed at or near 100° C.

This is found in the majority of instances to be the most effective method of applying the dyestuffs in question, but there are a number which dye better at 40°—50° C. These are, for example: Erikas B N and 2 G N; Brilliant Congos G and R; Congo Rubin; Orange T A; Curcumin S; Chrysophenin G; Mikado Yellows 3 G and 6 G; Chicago Blues 6 B, 4 B, 2 R, and 4 R; Columbia Blues G and 2; and Zambesi Black D. Of these, at the lower temperature, Chicago Blues 6 B and 4 B and Columbia Blue G give slightly redder shades, Brilliant Congo R and Orange T A give yellower shades, and Brilliant Congo G is brighter than when dyed at 100°. Other dyestuffs dye at 40°—50°, and at 100° C., colours similar in intensity but different in shade. Examples are Brilliant Orange G and Heliotrope B, the former of which, at 40°—50°, dyes much redder, the latter considerably bluer, than at 100°. Also, at the lower temperature, Columbia Red 8 B yields bluer, Chrysamine G greener, Zambesi Brown G and Deltapurpurin G give clearer, and Congo Sky Blue and Mikado Browns B and G give redder and purer shades than are usually obtained.

It is a striking fact that the dyestuffs which dye cotton better at the lower temperatures, are all compounds which dye with difficulty. It is found, indeed, as a rule, that the less readily a cotton-substantive dyestuff is absorbed by cotton, the better, relatively, does it dye at 40°—50°.

Chrysophenin G and Erikas B N and 2 G N may be satisfactorily applied at the ordinary temperature, and, in general, for pale shades, dyeing in the cold is to be recommended in the case of dyestuffs dyeing with difficulty.

It is also advantageous in the dyeing of mixed woollen and cotton fabrics with dyestuffs which at 100° yield darker colours on wool than they do on cotton, to dye at 40°—50°, in order to obtain even shades. Chrysophenin G, Erikas B N and 2 G N, Brilliant Congos G and R, and Zambesi Black D are instances of this class.

The colours on cotton obtained at 40°—50° C. are hardly so fast to washing as those dyed at 100°; but it is to be remarked that the cotton-substantive dyestuffs generally are not very fast to washing, and the difference is almost inappreciable.—E. B.

Mordanting of Wool with Chromium. M. Last. Färber Zeit. **8**, 181—184 and 201—203.

The principal chromium mordants used for wool are potassium bichromate with either sulphuric acid, tartar, lactic acid, or oxalic acid, and chromium fluoride with oxalic acid. With certain dyestuffs, *e.g.*, logwood, where the useful mordant is chromic acid, sulphuric acid is the best assistant, the organic assistants being useful only on account of, and in proportion to their acidity. Tartar and lactic acid, being stronger reducing agents than oxalic acid, are to be preferred when a basic chromium mordant or a mixture of basic and acid mordant is required. This mixed mordant gives a good result with most dyestuffs, and is, on the whole, the best mordant.

When wool is mordanted with the object of fixing chromium in the basic form, the appearance of the spent liquor and the mordanted fibre is as follows:—

Mordant.	Spent Liquor.	Fibre.
$K_2Cr_2O_7$ and tartar	Deep greenish-yellow	Greenish tint
$K_2Cr_2O_7$ and oxalic acid ..	Deep yellow	Yellow
$K_2Cr_2O_7$ and lactic acid ..	Pale yellow	Dirty green
Cr_2F_6 and oxalic acid	Green	Pale green

It appears from this that more chromium in the basic form is taken up by the wool when mordanting with bichromate and tartar, or chromium fluoride and oxalic acid, than with bichromate and oxalic or lactic acids.

A comparison was made of various dyestuffs dyed on the above four mordants, with results shown in the following table:—

Dyestuff.	Percentage.	Depth and Shade of Colour obtained. 3 per Cent. $K_2Cr_2O_7$, 2½ per Cent. Tartar.	3 per Cent. $K_2Cr_2O_7$, 2½ per Cent. Oxalic Acid.	3 per Cent. $K_2Cr_2O_7$, 3 per Cent. Lactic Acid.	3 per Cent. Cr_2F_6, 1 per Cent. Oxalic Acid.
Alizarin Red S (B.A.S.F.)	3	Deep bluish tone	Similar to tartar	Paler	Similar to tartar
Anthracene Acid Brown R (C.)	3	Deepest	Next in depth to tartar.	"	Bright, but paler
Alizarin Orange N paste (M. L. & B.).	15	"	Paler	"	"
Alizarin Yellow G G paste (M. L. & B.).	15	Next in depth to lactic acid.	"	Deepest, but dull	Paler
Coerulein S (B.A.S.F.)	3	Deepest	Duller	A little paler, and brighter than with tartar.	Very yellowish
Chrome Fast Yellow G G (Berlin).	3	As with lactic acid	Paler	As with tartar	Bright, but paler
Anthracene Brown S W (B.A.S.F.).	3	As with oxalic acid	As with tartar	Palest; very red	Deepest
Alizarin Blue S W paste (B.A.S.F.).	15	Very similar to lactic acid.	Palest; bluer	Deepest and reddest	A little bluer than lactic acid.
Alizarin Blue R R paste (M. L. & B.).	15	As with lactic acid	Paler	Redder than with tartar.	Paler
Alizarin Blue R paste (M. L. & B.).	15	"	A little deeper	"	Very similar to tartar and lactic acid.
Alizarin Blue D N paste (M. L. & B.).	15	"	Similar to lactic acid	"	Similar to tartar
Alizarin Blue A paste (M. L. & B.).	15	Similar to lactic acid	Palest	Deepest; reddish	"
Alizarin Dark Blue S paste (M. L. & B.).	15	"	Paler and redder	Similar to tartar; a little redder.	Deeper than oxalic acid
Alizarin Cyanin G G (Bayer).	3	Deepest and reddest	Paler: greenest	Between tartar and oxalic acid; reddish.	Paler; greenish
Alizarin Cyanin R R (Bayer).	3	Similar to lactic acid	As with lactic acid	Deepest	Similar to lactic acid; greenish.
Brilliant Alizarin Cyanin G paste (Bayer).	15	Similar to oxalic acid	Deepest	Paler; very greenish	Paler: very greenish
Alizarin Black W R paste (B.A.S.F.).	15	Deepest	Paler	Very similar to tartar; a little bluer.	Paler

Alizarin Blue is deepest on bichromate and tartar or lactic acid mordant. As a rule, tartar gives the bluest shades; with lactic acid, and generally with oxalic acid, redder tones are obtained. Chromium fluoride and oxalic acid is a combination influencing the shade towards green.

The differences of shade also depend on the proportion of

organic assistant to mordant. It cannot be definitely said which is the best chromium mordant, since for each dyestuff there exists a particular mordant which yields the best result. In practice, however, it is impossible to vary the mordant in this manner, and bichromate and tartar may be considered the most generally useful.

Whittaker and Denison (J. Soc. Dyers and Colourists, 1895, 98) have estimated the amount of chromium taken up by the wool when the different assistants are employed, and they conclude that the greatest amount is fixed by using the bichromate and oxalic acid mordant. Since, however, this mordant does not give the deepest shades, it is evident that the depth of colour does not depend on the total amount of chromium fixed on the wool, but on the amount fixed in acid or basic form according to the dyestuff employed. It should also be noted that when the three assistants are used in acid-equivalent amounts, less chromium is fixed with oxalic acid than with lactic acid or tartar.

In selecting the best mordant, it is also necessary to consider the fastness of the resulting colours, especially as regards washing and light. A series of tests has therefore been carried out in this direction. With regard to fastness to light, very slight differences are shown, but, in respect of fastness to washing, the shades dyed on bichromate and oxalic acid mordant are inferior to the rest. In order to ensure the best amount of each assistant being used, patterns were mordanted with 3 per cent. of mordant and 1, 2, 3, and 4 per cent. of assistant. The best proportion of assistant and the fastness to washing are illustrated in the following table:—

Dyestuff.	Percentage.	Best Amount of Assistant, and Fastness to Washing of the Shades obtained.			
		3 per Cent. $K_2Cr_2O_7$, 1—4 per Cent. Tartar.	3 per Cent. $K_2Cr_2O_7$, 1—4 per Cent. Oxalic Acid.	3 per Cent. $K_2Cr_2O_7$, 1—4 per Cent. Commercial Lactic Acid.	3 per Cent. Chromium Fluoride, 1—4 per Cent. Oxalic Acid.
Alizarin Red S................	3	2—3 per cent.; differences slight.	1 per cent. best, especially in fastness.	1 per cent. 4 per cent. is much paler.	1 per cent. best, especially in fastness.
Anthracene Acid Brown......	3	1 per cent.	1 per cent. deepest	1 per cent. deepest, but dull.	1 per cent.
Alizarin Orange X paste......	15	2 per cent.	1 per cent.	1 per cent.	1 per cent. best in shade and fastness.
Alizarin Yellow G G..........	15	2—3 per cent. deepest, 1 per cent. brightest: fastness good.	1 per cent. deepest and fastest.	1 per cent. deepest	1 per cent. deepest and fastest.
Cœrulein S....................	3	1 per cent. best: differences slight.	"	"	4 per cent. deepest, but 1 per cent. fastest.
Chrome Fast Yellow G G.....	3	Differences slight: 2 per cent. deepest.	1 per cent. deepest; difference slight.	Differences slight; 2—3 per cent. deepest.	1 per cent.
Anthracene Brown S W......	3	2 per cent. best in shade and fastness.	1 per cent.	3 per cent.	1 per cent. best in shade and fastness.
Alizarin Blue S W............	15	2 per cent.	"	"	1 per cent.
Alizarin Blue R R............	15	"	"	"	"
Alizarin Blue R.............	15	"	"	2 per cent.	1 per cent. 3 per cent. is redder, deeper, and much less fast.
Alizarin Blue D X............	15	2 per cent.: differences slight.	"	3 per cent.	2 per cent. deepest and reddish: 1 per cent. faster.
Alizarin Blue A..............	15	2 per cent.	"	"	1 per cent.
Alizarin Dark Blue S..........	15	"	"	3 per cent.; differences slight.	"
Alizarin Cyanine G G.........	3	2—3 per cent.	1 per cent. best and fastest.	3 per cent.; fastness very good.	1 per cent. greenish; 4 per cent. redder and less fast.
Alizarin Cyanine R R.........	3	1 per cent.	1 per cent.	3 per cent.	2 per cent. deepest; 1 per cent. fastest.
Brilliant Alizarin Cyanine G..	15	2 per cent.	"	2 per cent.	1 per cent. greenish and fast; 4 per cent. reddish and not fast.
Alizarin Black W R..........	15	1 per cent. deepest; fastness good.	1 per cent. best and fastest.	2 per cent. deepest; fastness good.	1 per cent. deepest and fastest; 4 per cent. very reddish.

It appears from the above table that when tartar or lactic acid is used, the best proportions are 3 per cent. of potassium bichromate with 2—3 per cent. of assistant; in exceptional cases less assistant should be used. With oxalic acid the best proportion is 3 per cent. of bichromate or chromium fluoride and 1 per cent. of oxalic acid. These amounts give the deepest shades, and as a rule dyes which are the fastest to washing. It is noteworthy that with oxalic acid the fastness is improved as the oxalic acid is decreased, and when a small percentage is employed, the fastness compares much more favourably with that of the shades obtained with tartar or lactic acid.

An excess of reducing agent is harmful, since the increased acidity of the bath retards fixation of chromium; but a certain acidity is necessary, as shown by Whittaker and Denison, *loc. cit.*, who obtained much worse results with neutral potassium oxalate than with the acid salt or the free acid. The author cannot confirm the statement of Körner that by employing the correct proportion of reducing agent, the chromium is converted into the violet modification, for he finds that the mordant bath remains yellow even after two hours' boiling.

In the case of chromium fluoride mordant, an entire change of shade frequently takes place when excess of oxalic acid is used. Since oxalic acid cannot act as a reducing agent towards chromium fluoride, its effect must be wholly due to its acid properties, whereby hydrofluoric acid is liberated and the chromium brought into solution.

A chromium mordant has lately been introduced which contains organic assistants (especially lactic acid) and also sulphuric acid. It possesses the advantage of completely exhausting the mordant bath, but the disadvantage of "going on" very quickly and therefore less evenly.—R. B. B.

Indigo, Printing of, on Cotton Fabrics. C. Kurz. Revue Gen. des Mat. Colorantes, 1897, 79.

The author recommends the following as a simple method of printing with indigo:—

40 grms. of refined Java indigo are dissolved in 200 c.c. of aniline by 15 minutes' boiling. The clear solution is poured off, the residue being preserved for a second operation. On cooling, the solution deposits indigo in a finely divided condition, which enables it to be readily mixed with the thickening employed, the latter consisting of caustic soda and roasted starch. The colour is printed on goods padded with a solution of glucose at 5° B., and after printing, the pieces are dried, steamed for 2 minutes, and washed. A deep blue is produced with 40 grms. of indigo per litre of printing-colour, and the advantage of the method depends upon the extremely fine state of subdivision of the reprecipitated indigo.—R. B. B.

Colouring Matters Dyed on Nineteen Metallic Mordants. A. Scheurer and A. Boylinski. Bull. Soc. Ind. Mulhouse, 1897, 161—231.

The authors have exhaustively investigated the mordanting properties of the compounds of 19 metals, *viz.*, Gl, Sn, Ce, Pb, Bi, Mn, Hg, Cd, Zn, Cu, Cr, Ur, Ni, Co, Fe, Al, Y, Zr, Th. The mordants employed were usually the acetate or aceto-nitrate, which were printed on calico in stripes, the printing-mixture consisting of mordant solution thickened with Alsace gum, of such a concentration that 1 litre corresponded to 0·11 of the atomic weight of the metal in grammes. After printing, the mordants were dried, fixed with gaseous ammonia, passed through a chalk bath, and the pieces well washed. A sample of the calico thus printed with the 19 mordants was then dyed with 1 grm. of colour per litre of water, with the addition of chalk or calcium acetate when considered necessary. The dyed patterns were divided into four portions, of which one was immediately dried, a second soaped at 60° C., the third soaped at 60° C. and then exposed to light (the results of exposure will be published later), and the fourth soaped at the boil with 10 grms. of white soap per litre.

Tables are given showing the colour obtained with each of the 66 colouring matters employed and each mordant, also the results of soaping at 60° C. and 100° C. respectively. The following is a list of those colours, which are interesting both on account of the shade obtained and by reason of their fastness to boiling soap :—

Mordant.	Colouring Matter.	Shade.
Ur	Methylene Blue	Very bright deep violet
Zr, Th, Cr	Gallocyanin	Black-violet
Bi, Y, Zr, Th.	Cœrulein	Greenish-olive
Ur	Safranine	Bright deep crimson
Bi	Rhodamine	Very bright violet-pink
Gl	Chrome Violet	Pink
Zr, Th, Bi, Cu	Weld	Yellow
Gl	„	Yellow (less fast)
Zr, Th	Gambier Cutch	Cutch brown
Bi	Sumach	Rich olive-yellow
Cu	„	Mode shade
Zr, Th	„	Yellow mode shade
Y, Cu, Ce, Zr, Th.	Tannin	Mode shades
Ur	Quercitron Bark	Puce
Zr, Th, Bi	Persian Berries	Orange-yellow
Ur	„ „	Bright red-orange
Ni	Erica B (Berlin)	Violet-crimson
Ni	Acid Ponceau (Durand)	Light orange-red
Mn	Bismarck Brown	Orange bistre
Ni	Benzo Orange (Bayer)	Red
Ni	Congo G R (Berlin)	Dull red
Ni	St. Denis Red (Poirrier)	Light red
Ni	Diamond Black (Bayer)	Violet-black
Ni	Ponceau 6 R B (Berlin)	Violet-pink
Gl	Alizarin R, V, & J.	Reddish-black
Ur	„ „	Black
Zr	„ „	Dark crimson
Gl	Alizarin Black	Bluish-grey
Zr	Alizarin Yellow A	Pink (after soaping)
Cr	„ „	Bright yellow
Cu	Carbazol Yellow (Badische).	Very deep yellow-olive
Zr, Th, Gl	Carbazol Yellow	Orange-yellow
Ur, Ni, Zn, Cd, Th.	Methyl Violet B (Badische).	Violet
Al	Patent Blue (M. L. & B.)	Bright violet-blue
Ni	Azo Green (Bayer)	Green
Sn	Alizarin Blue X (Badische).	Red-violet (in acid bath)
Sn	Alizarin Green S (Badische).	Violet-grey
Ur	Alizarin Maroon (Badische).	Violet-black
Y, Zr, Th	Alizarin Maroon	Puce, or deep violet
Gl, Al, Zr, Th.	Alizarin Orange	Crimson
Y, Zr, Th	Anthracene Brown	„
Ur	Quercitron Bark	Puce
Zr, Th	„ „	Olive-yellow
Ur	Old Fustic	Bright deep wood colour
Y, Zr, Th, Cr.	Cochineal	Red to red-violet
Ur	Pyronine G (Leonhardt)	Dark crimson
Ur	Benzoflavine (Oehler)	Very bright deep orange
Pb	Gallein	Blue
Zr	„	Deep prune
Ur, Bi, Zr, Th	Pegu Cutch	Wood brown
Sn	„	Cutch brown
Ur	Magdala Red	Very deep purple

Metallic Oxides Acting as Resists for Colouring Matters.

Resist.	For
Sn	Soudan G Malachite Green Azo Green
Pb	St. Denis Red
Cu	St. Denis Red Diamine Violet
Ur	St. Denis Red
Co	Alizarin Blue X

In the two final chapters the authors give the constitution of the artificial dyestuffs used in their experiments, and a list of the chief deposits which serve as sources of the rarer metals employed. The prices of certain pure salts of these metals in December 1895 are also given as follows :—

	Francs per Kilo.
Glucinum (sulphate)	100·0
Cerium (acetate)	25·0
Bismuth (acetate)	17·5
Uranium (acetate, crystallised)	27·5
Yttrium (nitrate)	500·0
Zirconium (nitrate)	75·0
Thorium (nitrate)	375·0

—R. B. B.

PATENTS.

Dyeing Cotton and other Vegetable Yarns, Threads, and Fabrics; Impts. in. Ad. Liebmann and Al. Liebmann, Manchester. Eng. Pat. 21,253, Sept. 25, 1896.

The authors have discovered that if dyed vegetable fibres are mercerised, or if the colouring matter be added to the mercerising solution itself, the colours become fixed in the fibre. Dyeing and mercerising may be performed simultaneously when the dyestuff to be used is soluble in the mercerising bath. When this is not the case, the goods are impregnated with a solution of the dyestuff, and are then mercerised in the usual way.—I. S.

Cotton Threads and other Textile Materials, Impts. in the Method of and in Means for Dyeing and Finishing or Dressing. C. H. Jacob, Lyons, France. Eng. Pat. 23,199, Oct. 22, 1896.

The two essential features claimed for this invention are the method of preparing a solution of "*Hai-Thao*" or vegetable gelatin (or "Gelose," as it is also termed), and the means for applying it. 30 litres of glycerin at 28° C. are mixed with 30 litres of water, and 3 kilos. of "*Hai-Thao*" are dissolved in this at boiling temperature. When solution is complete, and whilst still hot, 200 grms. of zinc sulphate, previously dissolved in water, are added. The yarns are passed in single threads through this solution, thence through eyelets to remove surplus liquor and to smooth down the nap of the yarn, and finally through a heated chamber for drying.—I. S.

Kiers and like Vessels for Bleaching, Dyeing, and similarly Treating Textile Materials, Yarns, and Fabrics; Impts. in connection with. E. Makin, jun., Manchester. Eng. Pat. 23,635, Oct. 24, 1896.

The principal objects of this invention are to enable the bleaching or other liquor to be rapidly and economically heated and freely circulated throughout the materials under treatment. Any convenient kier or vessel is connected at or near the bottom by a pipe, to a series of hollow shells or vessels in the form of cones placed in a suitable furnace flue and arranged in a row, the larger end of the second cone overlapping the small end of the first, but preferably without touching it, and so on; thus leaving spaces between the cones as well as a central passage through them for the gases from the furnace. The hollow cones are fed or supplied with bleaching, dyeing, or other liquor in any convenient manner, and are also connected by a discharge pipe to the upper portion of the kier.—I. S.

Scouring and Dyeing Hanks of Yarn or Fibrous Materials, Impts. in Apparatus for. H. Bentley, Bradford. Eng. Pat. 23,718, Oct. 26, 1896.

A RECTANGULAR movable framework, which can be raised or lowered into a suitable vat, is fitted along the top at one side with a number of sockets geared together and adapted to receive the square ends of sticks similar to those usually employed to support hanks of yarn in vats. The other ends of the sticks are fitted with metal journals (screwed into and capped over the end), which rest in bearings formed in the framework at the other side: and provision is made to confine these journals longitudinally in their bearings, and so keep the other ends in their sockets. A similar arrangement is provided below to receive a bottom set of sticks. The sticks are revolved by means of a shaft fitted at one end of the vat, and geared by means of toothed wheels with the pinions of the sockets.—I. S.

Mordanting Yarns, Threads, Fabrics, and other Materials composed of Fibres having a Vegetable Origin: Impts. in the Method and Means for. Ad. Liebmann and W. Kerr, Manchester. Eng. Pat. 23,741, Oct. 26, 1896.

THE metallic oxides to serve as mordants are dissolved, with or without the aid of glycerin, in a strong solution of caustic soda, in which the goods are then mercerised and mordanted in one operation.—I. S.

VII.—ACIDS, ALKALIS, AND SALTS.

Glover Tower, Construction of. H. H. Niedenführ. Chem. Zeit. **21**, [68], 664—666.

IN the construction of a sulphuric acid plant, it is not only necessary to consider the suitability of the individual portions of the apparatus for their own particular work, but each part must be studied in its mutual relationship to all the other parts. This is especially true of the Glover tower: and the author thinks that by suitable construction it can be made much more valuable than at present, especially since, in addition to its own particular function, it also acts as a much more intense acid-producing apparatus than the lead chamber itself.

In the sulphuric acid manufacture we have to deal with reactions between gases and vapours, and also of these with liquids. The energy and completeness of these reactions depend upon a suitable composition of the gaseous mixture, the time of reaction, and the nearness of the molecules to one another, that is, to the pressure under which the gases are. The liquids to be considered, are weak and strong vitriol and nitrous vitriol, and they must naturally present as large a surface as possible to the gases, and be maintained a considerable time in contact with these. To obtain a suitable composition of the gases, a correct working of the pyrites burners is essential, since this lies in the closest connection with the draught as the source of the oxygen. The draught at the end of the system does not, however, indicate the working of the individual parts of the plant, and these should be so arranged that an intense and uniform reaction takes place in the near end of the chamber. For this it is essential that the gas entering the lead chamber should not be diluted, by diffusion, with the less active and partially spent gases resulting in the process.

This is readily managed in a plate-tower system, which, by its symmetrical construction, ensures a uniform movement of the whole gas.

Here is an advantage of the plate tower over the cone, the dead space in which leads to a lagging behind of the slower-moving molecules. In the lead chamber this dilution cannot be avoided; it is partially lessened by combining several chambers in one system, and thus effecting a space separation of the gases according to the concentration changes. When the space is thus divided between three chambers, a common proportion between the sizes of these is 3 : 2 : 1. From observation, the author states that the maximum space should not be alloted to the first chamber; this agrees with the above considerations; but he would not, however, recommend extreme smallness for this chamber. For cross-section, on which the gas velocity mainly depends, and with this the duration of reaction, the author has found for systems of 3,000 cb. m. a chamber cross-section of 45—50 sq. m. to be most advantageous, and larger or smaller systems should be constructed proportional to this standard. He also deems it advisable to diminish the height and width of the individual chambers in proportion to the diminution of absolute gas volume.

Connected with energetic and uniform production in the first chamber, is the maintenance of a definite time of reaction and pressure, and care must be taken to diminish neither this nor the correct working of the burners in the introduction of the Glover tower. The draught must be diminished as little as possible. For this the burners should lie as deep as possible, and the roof of the chamber following the Glover should be higher than that of the latter. Attention must be paid to the tower packing, to obtain with the least loss of draught the most intimate contact between the gases and liquid. The loss of draught is less, the greater the cross-section of the tower, but since in wide towers the contact of gas with liquid is incomplete, the author recommends the use of two towers lying parallel. For a 3,000 cb. m. system the base area of these should be 2·75 × 2·75 m. (measured from lead to lead), and it is important, that two equally long and wide pipes should connect these with the burners, and a second pair with the chamber. The inlets to the chamber are so arranged that each is equally distant from the chamber outlet situated on the opposite wall. The increase in cross-section allows of diminished height: this simplifies the placing of the chamber at a higher level than the tower. The lesser height and greater section favour a retardation in the gas velocity and increase the contact of the gas with the nitrous vitriol, that is, the reactions in the tower. A still greater effect is produced by increasing the time during which the nitrous vitriol remains in the tower, which also allows of a diminution in height. That this is correct has been shown in the use of Lunge-Rohrmann plates as tower packing. The Administration of Mines of Buchsweiler, with the unsuitable burner gas obtained from gas-purifying residues, performed 80 per cent. of the total denitrification in a plate tower only 1·75 m. high; the daily production being 4,000 kilos. of H_2SO_4. At works in Copenhagen and Japan the effects of the Glover towers have been increased by the introduction of plate layers.

Neither the material nor the size of the holes in the ordinary Lunge-Rohrmann plates are suitable for Glover towers; but, recognising the value of the plate idea, the author has altered these by substituting for the material the ordinary acid-proof firebrick, and while retaining the network arrangement of the holes, altering the canals around these so as to maintain in circulation the largest possible quantity of acid. A Glover tower of 4 sq. m. section would require 128 stones of the modified pattern for each layer, and these would hold about $18\frac{1}{2}$ litres of acid, or $\frac{1}{7}$ litre of liquid surrounding each gas passage.

Regarding the possibility of a deposit forming in these canals, the author points out that, according to his own experience and that of others, the main bulk of flue dust and condensable matter in the burner gas always settles out in the lower layers of the Glover filling, that is, where the gases first come into contact with the acid. Slimy deposits from the packing are prevented by the movement of the vitriol, and the removal of crusts would be effected by allowing the periodical flow of a strong stream of acid for a few minutes, as is done with the Glover at present. The author therefore recommends that the lowest three or four layers should consist of a network of the ordinary stones, and for the more ready removal of obstructions, he separates these layers from the upper and succeeding ones by constructing these latter upon a special framework so arranged as to leave a space between the two groups of packing material. Through an opening in the side of the tower, closed during the manufacture, with stone, it is an easy matter to remove the blocked lower layers and replace with new packing. By this means the process interruption is diminished, and loss of packing material reduced. The total height of a Glover tower containing 18 layers and the above space, is only 6·15 m., whilst a tower with the same number of the known network rows would be 7·75 m. high.

For larger systems this difference in height becomes still greater, and for a plant of 5,000 cb. m. might easily reach 3 m.—J. T. C.

Persulphuric Acid; Electrolytic Formation of. F. Richarz. Ber. **30**, [14], 1826—1827.

The formation of persulphuric acid, as the result of the association of two HSO_4 ions (referred to by Nernst, Ber. **30**, [12], 1826), was observed and thus explained in 1884 by the author (Wiedemann's Ann. **24**, 183), who determined all the conditions of its formation. In the same paper and a later one (Wied. Ann. **31**, 912), he showed that the hydrogen peroxide formed at the same time resulted from a secondary action of the persulphuric acid. Later researches have added nothing new to these results.

Helmholtz (Wied. **11**, 737), in 1880, observed the sudden increase of current as the E.M.F. of 1·08 volts is overstepped, Glaser's observation of which was described by Nernst (*loc. cit.*). The author and Lonnes (Zeits. f. Physikal Chem. **20**, 145) have confirmed this, and have shown that at the same point, the formation at the cathode of hydrogen peroxide (from the reduction of dissolved oxygen) begins.—J. T. D.

Plumbates, Alkaline Earth [$XPbO_3$]*: Manufacture and Properties of.* G. Kassner. Rev. Chim. Ind. 1897, **8**, 227.

The plumbates of the alkaline earths are produced by heating an intimate mixture of litharge with a small excess of the carbonate of the desired base for an hour or so in a current of air and, preferably, steam, in any kind of furnace which will allow of a temperature of 600 to 1,000° C. being maintained. They may be employed as oxidising agents by treatment with (1) water under pressure, (2) alkali carbonates, or (3) mineral acids, in order to yield lead peroxide; or they may be heated to dull redness in a current of carbonic acid gas, to produce free oxygen. They are practically insoluble in water, and their colour deepens, with the increasing atomic weight of the basic metal, from the yellow of the calcium compound to the black of the barium salt. Calcium plumbate in particular varies in its activity according to the temperature at which it is made; and if it has not been heated above 800° C., it is readily powdered, decomposes alkali bicarbonate solutions in the cold, caustic alkalis at the boiling point, forms a hydrate with water on standing, and gives off oxygen in an atmosphere of CO_2 at a low red heat. If the substance has been formed at or above 950° C., it possesses none of these properties; but they may be restored to it by heating under water at a pressure of several atmospheres. In order to ensure the mass being of uniform composition, it should be stirred at intervals in the furnace, or it may be advisedly mixed with 5 or 10 per cent. of combustible or volatile matter, such as sawdust or ammonium carbonate, so as to render it more porous. The presence of moisture is rather an advantage than otherwise.

Depending on the facts that they are insoluble and do not evolve gaseous products, these plumbates are indicated as suitable for many industrial purposes. They may be employed to replace the chlorate and part of the binding material in lucifer matches and coloured fires, and the articles are thus rendered odourless. In electric accumulators they yield a very porous coating, and the insolubility of the sulphate resulting from the action of the acid augments the cohesion of the covering. They form good filtering material for crude spirit, their basic nature determining the removal of the impurities, including amyl alcohol, especially if the liquid be slightly acidified with sulphuric acid before treatment. In the manufacture of lead or calcium glass, their use confers the advantage of increased facility in the combination of lime with the silica, since it is not necessary first to expel carbonic acid, as would be the case had chalk been employed, as it usually is. The plumbates can also, it is said, be employed in the preparation of potassium ferricyanide and the utilisation of this salt in a continuous process of bleaching without chlorine.—F. H. L.

PATENTS.

Chlorine Gas, Impts in Pumping Apparatus for, and the like. P. Jensen, London. From H. Cappelen and D. Cappelen, Gjemsö Kloster, Skien, Norway. Eng. Pat. 20,423, Sept. 15, 1896.

The pump cylinder is of cast iron, lead lined internally. The plunger is of wood, as well as the washer to the stuffing box, which is lined with wood. The valves and their seats are also of wood, the casings to the valves being of lead.—E. S.

Sulphuric Acid, Improved Method of and Apparatus for Evaporating. M. Schwab, Manchester. Eng. Pat. 22,512, Oct. 10, 1896.

The acid to be concentrated, is run into the uppermost of a series of shallow vessels arranged on an incline, each vessel being supported on an iron tray bedded in seatings in the side walls of the run, in a chamber heated directly from a fireplace in front, which chamber is thus separated into two flues (between which there is no passage), the flue below for the heating gases, and that above for the acid vapours, which are taken off near the top. The dishes are of flattened V-shape, with plane bases inclined upwards from a vertical rear wall to a curved lip at the discharging end. Above each dish, and supported by the side walls of the run, so as to form a roof for the evaporation chamber, a removable fire-clay plate or slab is fitted; access is also provided for by gas-tight doors in the sides of the chamber. An iron pipe, communicating with the external atmosphere, traverses the heating flue, and is connected in front to a projecting valved pipe, recurved inwards to enter the lower part of the evaporating chamber or flue, to which it conveys a current of heated air, assisting the evaporation. The advantage of using shallow or flat evaporating dishes containing but a small volume of acid at a time, is that no bumping, and so no considerable vibration, is caused during the boiling process, and hence no deterioration, such as cracking of the apparatus, with consequent leakage, and escape of acid fumes into the chimney, can take place.
—E. S.

Bleaching Compounds, Impts. in the Manufacture of. J. W. Chenhall, Totnes, Devon. Eng. Pat. 22,792, Oct. 14, 1896.

Sodium or potassium carbonate, bicarbonate or hydroxide, in strong cold solution, is saturated with chlorine, and finely divided carbonate or bicarbonate of the alkali used, is slowly added with stirring until the liquid thickens to the consistency of dough. The mass is then left to harden.
—E. S.

Sulphuric and other Acids, Impts. in Apparatus for Concentrating. E. Dyson, Manchester. Eng. Pat. 27,769, Dec. 5, 1896.

This invention relates to improvements in Eng. Pat. 17,699, 1893 (this Journal, 1894, 1059). The apparatus consists of a double row of stepped retorts, overflowing by lips one into the other, and each connected by a tube from its top to an inclined condensing pipe on one side, which receives the vapours. The retorts are enclosed below in a flue, the bottom or floor of which, according to the present improvements, is constructed of arched blocks, flat and horizontal on the top surface, on which the retorts rest, and are arranged in steps. The blocks are perforated to admit of the passage of heated gases from the chamber below. Each retort is provided with a loose division-plate extending from near the top to near the bottom, a groove being formed in the sides to admit of asbestos packing, the plate being held in position by ledges. In addition to, or instead of the division-plate, a distributing tube or fountain may be placed so as to receive the liquor flowing from one vessel and deliver it at or near the bottom of the next. The tubes are constructed with branches or outlets so placed as to prevent obstruction by sediment. A perforated plate is attached near the upper edge of the distributing tube, about on a level with the spout. Violent ebullition or splashing of the acid is thus prevented. Three or more of the lower retorts are cut off from the heating flue by a

division-plate, in order that the acid in them may cool gradually before it passes to the cooling receiver.—E. S.

Alkaline Cyanides, Method of Drying. J. D. Gilmour, Lennoxtown, N.B. Eng. Pat. 29,922, Dec. 29, 1896.

Air, heated preferably by the condensed water from steam, as the temperature must not exceed that of boiling water, is passed over the cyanide to be dried, then through a condenser, and, when deprived of moisture, is again passed over the cyanide, and so on repeatedly. The continuous use of the same portion of air is to prevent the introduction of an injurious quantity of carbonic acid, and consequent decomposition of the cyanide.—E. S.

Metallic Chlorides, such as those produced from Sulphide Ores; Impts. in the Treatment of. E. T. Turner, Adelaide, South Australia. Eng. Pat. 17,834, July 29, 1897.

This invention relates to improvements on Eng. Pat. 15,749, 1896 (this Journal, 1896, 741), according to which sulphide ores of zinc and lead containing small proportions of gold, silver, and other metals, were subjected to the action of gaseous and aqueous hydrochloric acid to obtain chlorides, which, after fusion, were treated by lixiviation and precipitation processes, the hydrochloric acid being regenerated. According to the process now described, the chlorides obtained are electrolysed while molten, to separate gold, silver, and lead. The residue of zinc, iron, and manganese chlorides is heated in a retort in which a partial vacuum is obtained by means of a steam jet. The zinc chloride volatilised, is decomposed by the steam into zinc oxide and hydrochloric acid, which latter is condensed for re-use. The iron and manganese chlorides remaining undecomposed are treated with steam in revolving calciners to obtain the oxides and hydrochloric acid. Or, alternatively, the iron and manganese chlorides may be decomposed by sulphur dioxide and steam in vertical cylinders.—E. S.

Nitric Acid, Impts. in Apparatus for Converting Nitric Peroxide into. O. Guttmann, London. Eng. Pat. 18,189, Aug. 4, 1897.

The object of the apparatus is to bring the whole of the acid formed, into contact with the ascending unconverted and uncondensed gases. The nitric peroxide gases pass from an ordinary denitration apparatus into an earthenware receiver filled with coke, broken glass, or balls, as described in Eng. Pat. 14,774, 1896 (this Journal, 1896, 700), whence they are drawn by a compressed-air ejector into two or more vertical coke-charged towers, down which water trickles. The liquid nitric acid thus formed runs into a distributing tank, and the gaseous portion passes from near the top of the towers to a Guttmann-Rohrmann water-cooled battery. The distributing tank has a number of sockets, screened internally, through tubes connected, to which the acid flows to the arched tops of the condensing tubes. A portion of the acid also flows to the earthenware receiver first mentioned. The gases remaining unabsorbed in the condensing tubes, are led into another condensing tower, and return as liquid acid. From the condensing apparatus the acid flows into a collecting vessel, which also receives the acid formed in the first-named receiver.—E. S.

VIII.—GLASS, POTTERY, ENAMELS.

Flue-Dust from Glassworks, The Composition and Origin of. O. Grosse. Chem. Ind. 1897, **20**, 430—434.

The author has analysed the flue-dusts from three glass-works using the Siemens regenerative system. In each case two samples were taken: the one close to the furnace, and therefore deposited at a high temperature; the other from a point near the chimney, deposited at a lower temperature.

I. *Fürstenberg Works.* — The two dusts were readily distinguishable by their colour. The sample collected near the furnace was pure white in colour, uniform, and distinctly crystalline in structure (prisms). It was completely homogeneous and coherent, and broke with a gritty sound in an agate mortar. Chemical analysis showed it to be a mixture of silicates with free silica.

The second sample, deposited at the lower temperature, was of a dark reddish-brown colour. It consisted, like the preceding, of coherent, preceptible hard crystals. It was not completely homogeneous, hard particles of oxide of iron being found in it. The first contained 96·7, the second 9·6 per cent. of silica.

II. *Josephine Works.*—The two samples differed very markedly from those of the Fürstenberg works. With the exception of a small residue of quartz-sand from the walls of the flue, both consisted of an extremely fine powder showing no tendency to agglomerate, and exhibiting under the microscope no trace of crystalline structure. Both samples were light grey in colour. They exhibited a reddish tinge, and the sample taken furthest from the furnace was less dark in colour than that collected nearer to it. This is due to the different proportion of sand containing iron, which was present in the two cases. Both were soluble in dilute warm hydrochloric acid of sp. gr. 1·01; only the admixed sand remained behind.

III. *Wiesauer Works.*—These dusts, in addition to sand, contained non-homogeneous particles of indistinct crystalline structure, some yellowish and others light green in colour. Like the Josephine dusts they were soluble in dilute hydrochloric acid, but they differed in colour, the dust from near the furnace being ochre yellow, the other deep black, owing to admixed finely divided carbon.

The author indicates three possibilities for the formation of these dusts: the particles are either carried mechanically forward from the furnace mixture by the gaseous current; or they are due to volatilisation occurring at the high temperature of the furnace, the volatilised substances depositing in the cooler portions of the system; or both these processes may occur simultaneously. In either case the material deposited in the flues is subjected to the further action of the gases evolved from the fused mass.

The author inclines to the view that the dust is due to sublimation. In support of this he refers to the well-known volatilisation of glass galls, to the crystalline nature of certain of the dusts, and to the analyses given in the original paper, which only show SiO_2, CaO, PbO, Al_2O_3, Fe_2O_3, and alkali, with arsenic and sulphuric acids in estimable quantity; Cr_2O_3 and Mn_2O_3 were either entirely absent or only present in traces in the dust, although at the Josephine and Wiesauer works, relatively large quantities of each were added to the melt. The author points out that the earths present in the dust, form chlorides volatile at relatively moderate temperatures—he gives an experiment to prove the volatility of $CaCl_2$—whilst chromium chloride decomposes to oxide on heating in air, and manganese chloride is only volatile at very high temperatures.

The formation of chlorides in the melt has its origin in the alkali chlorides present as impurities in the sodium or potassium carbonates or sulphate used in the manufacture. These alkali chlorides react either to produce hydrochloric acid with the silica and water-vapour, or else by direct substitution—

$$CaO(SiO_2) + 2NaCl = Na_2O(SiO_2) + CaCl_2.$$

Other silicates will react similarly to the calcium silicate.
—J. T. C.

Glass, Elasticity of: Its Dependence on Temperature. A. Winkelmann. Wied. Ann. **61**, 105; Chem. Zeit. Rep. **21**, [59], 153.

Elasticity is usually regarded as an additive property, that of a mixture being the sum of a number of terms, each made up of two factors: a constant depending on the nature of the constituent considered, and the proportionate mass of that constituent. The author's experiments do not confirm this, and in the case of glasses it is only approximately true for the members of a group of similar composition. The dependence of elasticity on temperature has been examined up to the softening point. The simultaneous addition of sodium and potassium oxides to a glass, increases, but that of boric anhydride, decreases the variability with temperature. In all cases, however, when the expansion is taken into account, the variation of elasticity with temperature is slight. The coefficient of elasticity is uniformly greater after heating than before.—J. T. D.

PATENT.

Plumbates, Alkaline Earth; Manufacture and Properties of. G. Kassner. Rev. Chim. Ind. 1897, **8**, 227.

See under VII., *page* 913.

Filter Presses [Potter's Slip, &c.], Impts. in. J. Critchlow and T. Shore, both of Stoke-on-Trent, Stafford. Eng. Pat. 26,112, Nov. 19, 1896.

See under I., *page* 899.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Hydraulic Cements, Action of Sea-Water upon. W. Michaëlis. Proc. Inst. Civil Eng. 1896–97, **129**, Part III., 1—11.

Portland and other hydraulic cements, containing excess of lime, segregate such excess as hydrate during the hardening process, so that, in the case of Portland cement, the hardened mass becomes completely permeated with crystals of calcium hydrate. When the mortar hardens in sea-water, the excess of lime is converted, in the first instance, into sulphate, and ultimately into silicate: but hand in hand with the formation of calcium sulphate goes the production of calcium aluminate-sulphate requiring certainly 30, and probably 60, equivalents of water for its crystallisation, and resulting in such enormous increase of volume that cohesion is destroyed. The true Roman cements, containing 1 part by weight of silicate to 1·1 or 1·2 parts of lime, form more stable compounds, without unsaturated residues, and therefore from a chemical point of view form the best hydraulic mortars. They resist the action of sea-water well, because the much smaller proportion of the double salt with alumina which is formed, finds sufficient space for expansion in the pores of the mortar. Such cements, however, are burnt at so low a temperature that they are deficient in density, so that mortars made with them shrink on exposure to the air, through the evaporation of the water which they have absorbed.

Portland cement, having been burnt at a higher temperature than Roman cement, is denser, but is defective from the chemical point of view, owing to the excess of lime already referred to. Hydraulic limes, such as those of Teil, bear a close resemblance to Roman cements, but are even looser still in texture. They contain, however, but little alumina, and the chief increase in volume they sustain is due to the crystallisation of calcium sulphate—not to the double salt above referred to.

The author's experiments show that these Portland cements richest in lime offer the least resistance to sea-water, from which it follows that for marine work the addition of lime or of highly calcareous cements to Portland cement is unsuitable. Moreover, modern Portland cements of high strength, but rich in lime, are less suitable for this class of work than the older cements containing less lime.

Mortars containing free caustic lime are very unstable. Great internal strains are produced, so that expansion and cracking may go on for years. This, however, may be remedied by the addition of sufficient hydraulic silica or alumina to form more stable compounds with the lime which remains or becomes free during the hardening process.

The addition of trass or of an efficient puzzuolana to Portland or other hydraulic cements containing excess of lime, can increase the strength of the mortars twice or three times, and render them stable in sea-water.

From a Portland cement with an average amount of lime, about 33 per cent. of calcium hydrate would be segregated. It is, therefore, advisable to add at least 125 parts by weight of trass to 100 parts of cement. The more lime present, the more trass is required.

In further experiments with various mortars, the author used artificial sea-water and solutions of calcium, magnesium, and sodium sulphates. One of the mixtures which withstood the action of calcium sulphate best was Teil lime, mixed with 5 parts of standard sand; all mixtures, however, containing hydraulic silica, either as hydrate or in the form of trass or burnt kaolin, were only slightly acted upon by either calcium or magnesium sulphate, and remained intact after the lapse of two years. Neat Portland cement mixtures, without the addition of hydraulic substances, soon disintegrated, especially when rich in alumina.

Hydraulic cements which contain more lime than is required to form stable hydrosilicates and hydro-aluminates, should not be used for marine work unless improved by the addition of substances such as those named. The author suggests that it would be preferable for the manufacturer of the cement, rather than for the consumer, to add the necessary quantity of puzzuolana or trass, as the proportion to be added would vary with the composition of the cement, which would be best known to the manufacturer.—H. H. B. S.

Blast-Furnace Slag that has been treated with Nitric Acid as an Addition to Hydraulic Cement. A. D. Elbers. Eng. and Mining J. 1897, **64**, [13], 364, and [16], 454. (See also this Journal, 1897, 680 and 800.)

In the tests for tensile strength, the briquettes had broken near the middle of the stem; two test-pieces for the crushing experiments were prepared from each fractured briquette, in the following manner. The stems of the broken pieces were ground until one piece measured $1\frac{5}{8}$ in. in length—measuring through the centre of the stem to the top of the head—and its counterpart, $1\frac{3}{4}$ in. in length. The superficial area of the pieces thus prepared, averaged about 2 sq. in., each piece differing from its counterpart about one-sixteenth part in volume. The following results were obtained:—

A.—Tests with High-Class Portland Cement.

Composition of Test Piece.	Tensile Strength per Square Inch after 16 to 18 Days.	Resistance to Crushing per Total Surface Area (about 2 Sq. In.) after 128 to 130 Days.
	Lb.	Lb.
Neat Cement	563	12,000—15,150
"	526	13,200—15,000
Cement, 80 per cent.; prepared slag, 20 per cent.	426	11,180—12,780
Cement, 60 per cent., prepared slag, 40 per cent.	305	11,870—12,910

B.—Tests with an Inferior Portland Cement.

Composition of Test Piece.	Tensile Strength per Square Inch after 19 Days.	Resistance to Crushing per Total Surface Area (about 2 Sq. in.) after 131 Days.
	Lb.	Lb.
Neat Cement	310	6,100—6,400
Cement, 60 per cent.; prepared slag, 40 per cent.	335	6,300—6,650
Cement, 40 per cent.; prepared slag, 60 per cent.	310	6,800—7,350

Note.—The resistance to crushing is stated separately for each piece of the broken briquette, the first number denoting the resistance of the shorter piece, and the second number, the resistance of its counterpart.

The test pieces prepared from the briquette made by casting the "slurried" mixture into the mould, and composed of 90 per cent. of high-class Portland cement and 10 per cent. of the prepared slag, with 30 per cent. of water added, gave the following results:—Tensile strain per square inch after 17 days (as previously reported), 350 lb.; resistance to crushing of the shorter piece, 9,450 lb.; resistance to crushing of the larger piece, 10,770 lb.

It is stated that cement "pats" containing slag that has been treated with nitric acid, after having been sufficiently long under water to become properly hardened, and then treated with weak solutions of strong acids, evolve very little, if any, more sulphuretted hydrogen than neat cement under the same conditions.

With regard to mixtures of cement, prepared slag, and sand, it was found that unfavourable results were apt to be obtained at first, but that after some time, especially if kept under water, the strength of the cement mixtures containing prepared slag and sand, increased more rapidly than that of the mixtures containing sand alone. The following table gives the results of tests made with an inferior Portland cement (the same as used for the tests given in Table B). The briquettes were tested for tensible strength on May 26th, when 19 days old; the resulting halves were kept in a dry place until September 15th, kept under water for 20 days, and crushed on October 5th.

Composition of Mixture.	Tensile Strength in Pounds per Sq. In. after 19 Days.	Resistance to Crushing in Pounds for about 2 Sq. In.
1 part of neat cement and 3 parts of sand.	160	2,775—2,820
1 cement, 1½ slag, and 3 parts of sand..	90	2,340—2,675

On comparing the relative results for tensile strength of the neat cement (see Table B) and the mixture of cement and sand, the author considers that if the average of 5 or 10 tests of the former had been taken, a higher result would have been obtained. On the other hand, the ratio between the resistance to crushing and the tensile strength of the mixture of cement, prepared slag, and sand, seems to indicate that the average of five or ten tests would have shown a much higher tensile strength than 90 lb. to the square inch.—A. S.

PATENTS.

Kilns for Burning Bricks, Tiles, Lime, and the like; Impts. in. F. D. T. Lehmann and P. N. Kohlsaat, both of Chicago. Eng. Pat. 14,673, July 2, 1896.

This invention consists of a brick kiln of the continuous type, but constructed with movable instead of permanent walls. The bricks are set up in the open air and the walls built round them, and similarly, after the bricks are burnt, the walls are taken down and the bricks exposed to the air. There are 10 chambers, and each day the one which has been fired the longest is taken down and set up with fresh bricks at the other end. The walls, floor, and covering are of fire proof tiles and the chimney of sheet iron. The flue is laid underground, beneath the kiln, and has connections, provided with dampers, leading into each of the chambers, but it can be taken up and relaid, as occasion requires, to suit the alterations in the position of the chambers. For the same reason the chimney is mounted upon wheels and runs upon a track.—H. H. B. S.

Fire Bricks and Fire-Proof Mortar, Impts. in the Manufacture of. A. C. Ponton, Bedford. Eng. Pat. 21,781, Nov. 2, 1896.

One ton of finely-ground burnt flints, with or without the addition of fine sand, is mixed with about 20 gallons of silicate of soda or potash and 3 or 4 cwt. of damp finely-cut fibrous material, such as hay or straw. The mixture is moulded into bricks, which are dried and burnt at a sufficient temperature to volatilise the soda or potash and to consolidate the silica.—H. H. B. S.

Finely Pulverised Substances, for Use in the Production of Portland Cement, Pigments, and the like; Impts. in the Manufacture of. R. Hunter, Glasgow. Eng. Pat. 24,711, Nov. 5, 1896.

The ingredients are crushed in a stone breaker and then, by an arrangement of elevators, successively conveyed to and passed through a pan mill, mill stones, and a revolving screen fitted with blades, the powder being finally received in a chamber, from which it can be drawn off for use as required.—H. H. B. S.

Bricks, Tiles, and the like; Impts. in the Manufacture of. A. W. Fludder, Great Shelford, Cambridgeshire, and G. H. Innes, Hitchin, Herts. Eng. Pat. 25,225, Nov. 10, 1896.

Dried and ground chalk, marl, and sand, or ground flint, are mixed with the requisite proportion of water and kneaded into a semi-plastic mass, which is then moulded into shape and burnt in the usual way. A little blue gault is sometimes added, which enables the burning to be carried out at a lower temperature.—H. H. B. S.

Impregnating Fibrous Substances, such as Wood, Peat, or the like; An Improved Process for. W. P. Thompson, London. From F. Hasselmann, Munich, Germany. Eng. Pat. 25,240, Nov. 10, 1896.

The object of this invention is the rendering of wood and other fibrous substances proof against destruction by fire, damp, insects, &c. The wood is successively impregnated with two solutions, which combine to form a solid substance in its pores, after replacing the easily decomposable saps. The following combinations are proposed:—Copperas and chloride of calcium; chloride of zinc and caustic baryta; copperas and caustic baryta; chloride of zinc and chloride of calcium; caustic baryta and chloride of calcium. The impregnation may be carried out either by soaking in the cold or by boiling, or by pumping out the air and sap from the wood, so as to facilitate the admission of the fluid into the very interior.—H. H. B. S.

Tight Joints, A New or Improved Composition for Making, and similar Purposes. H. Redhead, Newcastle-on-Tyne. Eng. Pat. 26,673, Nov. 24, 1896.

The composition is intended to be used in place of red or white lead for making tight joints in steam, water, gas, and other fittings. It is compounded of Portland, Roman, or other cement; Venetian red or brick dust; boiled linseed or other oil; litharge; and chalk or whiting.—H. H. B. S.

Grinding and Polishing Material, Artificial, and Grinding and Polishing Tools, Impts. in the Manufacture of. W. von Floryanowicz, Warsaw. Eng. Pat. 6793, March 15, 1897.

The patentee produces a powder, equal in hardness to emery, and applicable either as a grinding or polishing powder or for further manufacture into polishing wheels, grindstones, &c. Fire-clay is mixed into a paste with water, the mass dried, and burnt at a temperature of about 1,200° to 1,300° C. If the fire-clay does not fuse at this temperature, about 10 per cent. of felspar or 2 per cent. of burnt lime is added. The burnt clay is ground and sifted. To make grindstones, &c., 40 per cent. of the powder is mixed with 45 per cent. of ground fire-clay and 15 per cent. of manganese, the mixture moistened with water, pressed into shape, dried, and burnt at about 800° C.—H. H. B. S.

X.—METALLURGY.

Gold Field, Canada's Newest. J. T. Donald. Eng. and Mining J. 1897, **64**, [13], 369.

Auriferous quartz veins, from 8 in. to 2 ft. and 3 ft. in width, have been discovered on the north shore of Lake Superior in the vicinity of the Michipicoten River. The samples which have been tested have yielded high results. —A. S.

Hand-Jig, An Experimental. P. Buttgenbach. Eng. and Mining J. 1897, **64**, [11], 305.

The author uses the following apparatus for making preliminary tests, previous to treating the ore on a large scale, and also when only a small sample of ore is available for examination. A glass cylinder B, open at both ends, is fixed inside an outer glass vessel A, by means of an airtight india-rubber stopper C. Through C passes a copper tube D, which is connected outside the vessel to an air-ball F.

Within the vessel, the tube D is connected to a hollow copper ring G, which encircles the inner glass cylinder. This ring is perforated on its upper side with small holes, increasing in size as they lie further from the junction with the tube D. The lower part of the inner glass cylinder is provided with a projecting flange L, to which a copper wire screen is fastened by means of a clamp ring K. The ring

P, carrying the screen, has an india-rubber ring fixed on the face nearest the glass, in order to insure a tight joint. The apparatus is filled about three parts full of water and the ore to be treated is placed in the inner vessel. By squeezing the air-ball with the hand, the air between the two vessels is evenly compressed and forces up the water, as well as the ore, in the inner vessel. On continuing this action, the separation of the minerals according to their specific gravity is effected, just as in the ordinary piston-jigs. When the separation is complete, the air ball is disconnected, and the inner vessel, together with the india-rubber stopper, is withdrawn in a vertical position and allowed to drain. A strong wadding is then rammed into the vessel until it is in good contact with the ore, the vessel is inverted, and after removing the clamp and screen, the ore is forced out in layers by pressing on the wadding, and each layer cut off. For jigging "through the bed," a screen is used having meshes of 1 mm. and a bed of shot, 2—4 mm. in diameter. The ore of high specific gravity glides through the spaces between the single shot, without it being necessary to force the stroke to such an extent, that the latter is moved from its position. A considerable quantity of light tailings passes through the screen at first, but after a few minutes, nothing but heavy matter will pass, and when this is observed to be the case, the mixed product deposited previously, is taken out and passed through the apparatus again.

For separating very fine material, below, say, $\frac{1}{2}$ mm., the author employs the German "Handsichertrog," which acts like a small percussion table.

When working with a small jig of the kind described above, the author has frequently used a saturated solution of boro-tungstate of cadmium (sp. gr. 3·3) instead of water, and has in this manner been able to separate minerals having a specific gravity of more than 3·3, but which would not separate with water, owing to the specific gravities of the different constituents being too near together.—A. S.

Cyanide Process, Limitation of the. P. Argall. Eng. and Mining J. 1897, **64**, [10], 278.

It is stated that the cyanide process is inapplicable in the case of oxidised ores, in which the gold occurs partly in a coarse state, unless the cyaniding is preceded or followed by amalgamation. Copper ores are also unsuitable for cyanide treatment, even with dilute solutions, as the copper which dissolves, accumulates, and is finally deposited on the zinc in the precipitating boxes. Skey found that a 0·03 per cent. cyanide solution, after standing for one hour in contact with crushed and thoroughly washed samples of chalcopyrite and copper glance, contained a perceptible quantity of copper cyanide and traces of sulphur and oxidised sulphur compounds. Sulphide of antimony is also soluble in cyanide solutions. With regard to the action of sulphides, Skey found that a 1 per cent. solution of pure cyanide dissolved a given weight of gold in 19 minutes, but on the addition of 1/100,000th part of sulphur (as a sulphide) it required two hours to dissolve the same weight of gold. The author, however, considers that the presence of minute quantities of alkaline sulphides is not seriously injurious in the practical application of the cyanide process. If the sulphur of a soluble mineral combines with the cyanogen, it does not interfere with the solution of the gold; for instance, galena, though soluble is usually quite amenable to cyanide treatment. Zinc blende and pyrites are practically insoluble in cyanide solutions.

It is stated that a large consumption of cyanide—which may be due to the presence of free acid in the ore, of acid or basic salts, or to the ore being easily soluble in the solution—is invariably accompanied by a low extraction of gold.
—A. S.

Cyanide Mill Solutions. P. Argall. Eng. and Mining J. 1897, **64**, [9], 246.

The following table shows the changes which take place in ordinary mill solutions where zinc is used as a precipitant. The solution was originally made from pure potassium cyanide, and its strength was maintained by the addition of pure cyanide from time to time as occasion demanded :—

—	After 6 months' continuous use, during which time the Solution passed through 11,000 Tons of Ore.		After 13 months' continuous use, during which time the Solution passed through 26,000 Tons of Ore.	
	Before Precipitation.	After Precipitation.	Before Precipitation.	After Precipitation.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
KCN	0·501	0·484	0·505	0·505
HCN	0·061	0·079	0·017	0·021
Total simple cyanides.	1·302	1·335	1·470	1·445
$K_4Fe(CN)_6$	0·098	0·117	0·018	0·026
KCNS..........	0·212	0·267	0·058	0·057
Zn..............	0·316	0·360	0·368	0·388
CaO............	0·085	0·082	0·172	0·172
Gold (in ounces)	1·159	0·037	1·810	0·030

There was no material change in this solution after 18 months' continuous use. Zinc does not accumulate in the solution, as the greater part of that which is dissolved in the precipitating boxes is precipitated on the ore charges in the leaching tanks; zinc also separates out in the form of insoluble ferrocyanide, and as sulphide from the action of alkali sulphides, which are usually present in small quantities.—A. S.

Alloys, New. F. J. Davis. Eng. and Mining J. 1897, **64**, [9], 249.

Manganese bronze, free from iron, may be prepared by using a rich alloy of copper and manganese, in the place of the ferromanganese usually employed. An alloy thus prepared, and suitable for gun-wheels, propellers, and mining machinery, had the following composition :—Copper, 53 per cent.; zinc, 42 per cent.; manganese, 3·75 per cent.; aluminium, 1·25 per cent. The absence of iron permits the use of the large proportion of zinc, without risk of rendering the metal brittle. An alloy of the following composition : copper, 67·25 per cent.; manganese, 18·5 per cent.; zinc, 13 per cent.; and aluminium, 1·25 per cent., proved an excellent substitute for German silver, being quite as strong and making better castings, while it is less liable to corrosion. Its electrical resistance is four times as great as that of German silver. The small percentage of aluminium

in the alloys described above, was added to overcome the difficulty of obtaining sound castings.—A. S.

Iron and Copper, Browning of. Illustr. Zeits. für Blechindustr. **26**, [38], 1314.

The (copper) articles, heated to redness and cleaned, are coated with a pulpy mixture of equal parts of verdigris and colcothar (Fe_2O_3) in vinegar, then heated to redness again, and quickly rinsed in copper acetate solution. Another (dark) shade is obtained by the application of a mixture of powdered ferric oxide and spirit, the article being heated over an open fire, rinsed, and polished with a soft brush. For lighter shades the following mixture is used :—Copper acetate, 2 parts; vermilion, 2; salammoniac, 5; alum, 5 parts, in vinegar.

Iron gun-barrels are browned by a mixture of antimony chloride, 2 parts; ferric chloride crystals, 2; gallic acid, 1; water, 4 parts. After drying for about 12 hours in a warm place, the barrels are rubbed over with a woollen cloth and polished with olive oil and wax. Other mixtures suitable for the purpose are: equal parts of antimony trichloride, olive oil, and silver nitrate in 500 parts of water; also, copper sulphate, 54 parts; spirit, 26; nitric acid, 14; iron filings, 3; and water, 200 parts. Cleanliness and freedom from grease are essential to successful results.—C. S.

Passive Iron. J. C. de Benneville. Iron and Steel Institute, Autumn Meeting, 1897.

See under XXIV., *page* 941.

Iron, Thermo-Chemical Study of the Refining of. H. Ponthière. Iron and Steel Institute, Autumn Meeting, 1897.

In this paper an analysis of white iron is taken, and its ultimate composition worked out by assuming the various proportions in which the non-metallic impurities combine with the metals present. The heat produced in the many combustions and changes which take place during their removal in the process of refining is then calculated and expressed in calories for each reaction. The following table is then obtained showing for each constituent the heat of combustion per 1,000 grammes, together with the relative proportions of the same carried off by gases and left in the bath. No account is taken of the heat lost by solid matters ejected or volatilised from the converter owing to the difficulty in approximating to their value. The heat radiated from the converter is not allowed for, but may be taken as a constant proportion to be deducted from the figures given, so that their relative values will remain the same.

Heat in Calories left in the Converter by the Combustion of 1 Kilo. of each of the Constituents of the Iron.

Constituent.	Heat of Combustion.		Heat carried off by the Gases per Kilo.	Heat eft in the Bath per Kilo.	Calorific Efficiency per Cent.
	Per Gramme atom of Free Metal or Metalloid.	Per Kilo. of Constituent or of Free Metalloid.			
Silicon	219·2	7,326	1,096	6,230	84
Silicide of iron	288·2	3,430	450	2,980	86
„ of manganese	105·1	1,666	315	1,351	81
Phosphide of manganese	144·5	1,911	539	1,372	71
Manganese of the phosphide Metal only burnt	22·7	412	285	127	30
Sulphide of manganese	109·4	1,257	178	1,079	85
Carbide of manganese Carbon burnt to anhydride	174·2	2.205	1,009	1,196	54
„ in carbonic anhydride	97·6	8,080	4,592	3,498	45
Carbide of iron Carbon burnt to anhydride	101·5	1,691	327	1,364	80
Carbide of iron Carbon burnt to monoxide	77·7	1,295	217	1,078	83
„ in monoxide	29·4	2,450	2,262	188	8
Carbide of manganese Carbon burnt to monoxide	37·8	478	638	− 160	− 33
Phosphide of iron	86·6	1,305	384	921	70
Free iron	69·0	1,232	346	886	71

—A. W.

Blast-Furnace Slag, Titanium Cyanide Nitride in. Leon Franck. Chem. Zeit. 1897, **21**, [52], 520.

See under XXIV., *page* 942.

Nickel-Copper Alloys, Assay of. A. Riche. J. Pharm. Chim. 1897. **6**, [7], 300.

See under XXIII., *page* 937.

Silicon Carbide. L. Franck. Stahl und Eisen, **17**, 485.

See under XXIV., *page* 941.

PATENTS.

Furnaces, Impts. in, for Welding, Smelting, Metallurgical, and other Purposes. C. Schwalme, Zabrze, Prussia. Eng. Pat. 18,168, Aug. 17, 1896.

Petroleum is conducted in thin jets from a reservoir to a trough fixed above the fireplace, in which trough it is evaporated. The vapours are conducted into a combustion chamber, where they are burnt with hot air, mixed with part of the fire-gases, supplied through a regenerator underneath the chamber. The regenerator is heated both by the gases from the fire-grate and those from the combustion chamber. By the above means a high temperature is obtained suitable for welding, puddling, and smelting operations.—A. W.

Separation of Slime, Gas, or other Fluid from Bodies [Ores, &c.] containing or mingled with the Same, Impts. in or relating to the. W. A. Koeneman, Washington Street, Chicago, U.S.A. Eng. Pat. 20,299, Sept. 14, 1896.

The material to be treated is fed into a centrifugal strainer, which consists of two concentric perforated shells and an intermediate layer of felt, &c. The liquid, &c. separated by the strainer falls into a hopper, and is discharged through a suitable outlet. The solid or other matter left within the strainer is removed by a helical series of fixed scrapers, which transfer it to a conveyor, by which it is discharged from the apparatus. The scrapers are mounted on a shaft carried by a movable headstock, by means of which they can be inserted in or withdrawn from the strainer.—R. A.

Zinc and Copper Ores, Impts. in the Treatment of. A. J. Boult, London. From W. J. Koehler, Broken Hill, N.S.W. Eng. Pat. 20,615, Sept. 17, 1896.

The zinc and copper being present in the form of oxide, the ore (or other material) is treated with ammonium sulphate

or "sulphamate" and heated to 300 – 500° C., whereby the two metals become sulphated, and ammonia is evolved. After leaching out the sulphates, the copper is precipitated by metallic zinc, and the zinc sulphate solution is passed through towers, where it meets the ammonia gas produced in the heating of a subsequent charge, and becomes converted into hydroxide, which may be dried, calcined, and sent into the market as zinc oxide. The ammonium sulphate obtained, is employed to treat another batch of ore. When iron is present it must be removed from solution before precipitating the zinc as hydroxide.—W. G. M.

Nickel Ores, Impts. in the Treatment of, for Extraction of the Nickel and the Production of Iron Oxide Pigment. T. Storer, Glasgow. Eng. Pat. 22,721, Oct. 13, 1896.

This process is said to be specially adapted to the treatment of ores containing silicates, as, for example, those from New Caledonia. The finely-divided ore, which should pass a sieve of 60 meshes per linear inch, is heated for about six hours under pressure to about 370° F. in admixture with ferric chloride solution, whereby nickel chloride solution is obtained, together with a fine red oxide of iron suitable for use as a pigment. The nickel may be recovered from the solution by any of the usual methods. The colour of the oxide of iron may be varied by altering either the temperature at which it is treated, or the strength of the solution. Usually a solution containing $26\frac{1}{2}$ per cent. of solid ferric chloride is suitable for an ore with 5 to 8 per cent. of nickel, and 1,700 lb. of the dry salt would be used per ton of ore.—W. G. M.

Metallic Alloys [Fe, Mn, Ni, &c.], Impts. in the Manufacture or Production of. R. A. Hadfield, Sheffield. Eng. Pat. 24,705, Nov. 4, 1896.

The claims are for alloys of iron containing up to 1·5 per cent. of carbon, 15 to 25 per cent. of manganese and up to 25 per cent. of nickel, with or without chromium or tungsten, or both, up to 12 per cent.; and for similar alloys containing up to 1·5 per cent. of carbon, 7 to 25 per cent. of manganese and below 4 per cent. of nickel, with or without chromium or tungsten, or both, up to 12 per cent. Examples of the actual alloys are given, the composition varying according to the physical qualities required. The alloying metals are preferably melted separately, run into a suitable ladle, and the molten iron or steel poured into them.—A. W.

Steel, Manufacture of; Impts. in. W. P. Thompson, Liverpool. From La Société Civile d'Études du Syndicat de l'Acier Gérard, Paris. Eng. Pat. 25,461, Nov. 12, 1896.

In order to convert pig-iron directly into steel, the addition of an alkali metal (sodium) mixed with calcium chloride and lime is claimed. This mixture is to be charged into the crucible along with fragments of cast-iron, or it is to be introduced with the charge into the cupola, or wrapped in paper and plunged into the bath of metal in the foundry ladle.—W. G. M.

Metals [Iron, Steel] or other Substances with a View to the Manufacture of Steel and for other Purposes, Impts. in the Pulverisation of. W. P. Thompson, Liverpool. From La Société Civile d'Etudes du Syndicat de l'Acier Gérard, Paris. Eng. Pat. 25,462, Nov. 12, 1896.

The fluid pig-iron is run into a fireclay funnel-shaped vessel, provided at its lower extremity with an opening through which the metal runs into an outside casing supporting the vessel. It is here met with a forced current of air through a nozzle or other means, by which it is disintegrated into small globules which fall through another opening into a chamber in which it meets with an opposing current of hot air. By this means the globules are deprived of silicon, decarburised, &c., and the metal deposited into water in grains or as a dust ready for treatment with spiegel for conversion into steel. The disintegration of the stream of molten iron may also be brought about by pouring it on to a revolving clay disc, or by passing it between the terminals of an electric current of high density and low electro-motive force.—A. W.

Crucibles and Furnaces, Lining of; Impts. in the. P. E. Placet, Paris. Eng. Pat. 28,728 (under Internat. Convention), Dec. 15, 1896.

Chromium compounds, such as the bichromate of magnesia, lime, alumina, or potash, are fused on the inner surface of the crucible, and the whole is then calcined; it thus becomes coated with an adherent refractory coating of chromium sesquioxide. The material may be applied initially by melting the bichromate of potash, if that be chosen, in the crucible, and then causing it to flow over the surface, or by heating the crucible and either rolling it in the salt or allowing it to remain sufficiently long in contact with a solution of the compound. It may also be applied in the form of a paste, mixed with tar, molasses, or oil; or in conjunction with ordinary refractory materials, carbon-lined crucibles or metallic surfaces may be coated. Mixed with such substances as magnesia, lime, &c., the material may used as a cement in furnace building.

—W. G. M.

Crucible Steel, Impts. in the Manufacture of. S. Danner, Kladno, Bohemia. Eng. Pat. 29,846, Dec. 28, 1896.

Instead of adding the raw materials to the crucible they are first subjected to a preliminary treatment in a converter or a Martin furnace, whereby the metal is freed from manganese, sulphur, and phosphorus, and the carbon reduced to the necessary quantity. The fluid mass is then poured into the crucible and the remainder of the refining process is carried on in the usual manner.—A. W.

Sheet Metal, Method of Treating and Annealing. W. E. Harris, Niles, Ohio, U.S.A. Eng. Pat. 13,672, June 3, 1897.

This is an improvement on the method specified in Eng. Pat. 9552 of 1896 (this Journal, 1896, 659). The sheets are hot-rolled, annealed, cold-rolled, and then stacked in an annealing box with a mixture of copper filings, soapstone, pulverised iron ore, and plumbago (mixed together, for example, in the proportions of 5, 10, 45, and 40 per cent. respectively) separating the different sheets. When thus finished the sheets have the appearance of Russia- or hammered plates without further mechanical treatment.—W. G. M.

Slag [Tin], Impts. in the Treatment of, Resulting from the Smelting of Tin Ore. C. C. E. Bohne, Kreis-Harburg. Eng. Pat. 14,325, June 12, 1897.

The slags are decomposed in a granulated state by means of hot dilute sulphuric acid of 36° to 46° B. The residue then contains only 0·5 to 0·8 per cent. of tin and is used with two or three times its weight of quartz sand for making the hearths of tin-smelting furnaces, by which means this percentage of tin is also recovered. The tin in the sulphuric acid is recovered by electrolysis and the remaining iron sulphate solution evaporated for copperas.

—A. W.

Metallic Chlorides, such as those produced from Sulphide Ores; Impts. in the Treatment of. E. T. Turner, Adelaide, South Australia. Eng. Pat. 17,834, July 29, 1897.

See under VII., *page* 914.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(A.)—ELECTRO-CHEMISTRY.

Cathode Rays, Modification of Salts by the Action of. R. Abegg. Zeits. f. Elektrochem. 1897, 4, [4], 118—120.

The surface-changes produced by the action of cathode rays upon the haloid salts of the alkalis are the more remarkable because these salts are amongst the most stable known. The discovery was made by Goldstein, who regarded the change as being of a physical nature. These changes were afterwards investigated by Elster and Geitel, and by Wiedemann and Schmidt, and it was suggested that

the change was a chemical one, which took place with formation of sub-haloid salts. The author has made experiments with the object of deciding between these two views.

Potassium chloride, sodium chloride, and potassium bromide are converted respectively into violet, brown, and blue modifications by the action of cathode rays *in vacuo*. There is no evolution of gas nor do the coloured salts when dissolved in water give any alkaline reaction (*i.e.*, if the salts originally taken are neutral and carefully purified). Their colour is not discharged by the action of chlorine, nor do they themselves decolorise permanganate solution. But by the heating action of very powerful cathode rays, they can always be transformed back into the white form; and this again, after cooling, can be changed into the coloured modification by a short exposure to the rays. These facts are not in accordance with the view that the coloured substances are sub-haloid salts; they tend to show that the change is a physical one. The change does not appear to affect the solubility of the salt.

Cathode rays exercise no reducing action upon cupric chloride, although the brown anhydrous cupric chloride is easily transformed into the white cupric chloride. Moist cupric chloride becomes blackened when exposed to sunshine. Cathode rays do not produce this effect; but, like sunlight, they colour calomel faint yellow. They turn silver chloride a deep violet-black. This colour is scarcely changed by the heating action of powerful cathode rays. The behaviour of silver bromide is different. It is coloured by cathode light (as by sunlight) deep grey. This colour is changed by heating to the original yellow (or perhaps nearer orange) and can be darkened again by light or cathode rays. As these changes take place in the dry salt *in vacuo*, they cannot well be due to any reduction or loss of chlorine or bromine.—D. E. J.

Ionic Reactions and their Importance in Electro-Chemistry. F. W. Küster. Zeits. f. Elektrochem. **4**, 1897, 106—113.

A panegyric of Svante Arrhenius's theory of electrolytic dissociation, in the form of a lecture illustrated by experiments. This theory has transformed analytical chemistry from an art into a real science. At first sight electrochemical theory appears to have only a slight connection with analytical chemistry. But in analysis we have to deal almost exclusively with aqueous solutions of salts (in an extended sense, including bases and acids) and it is just in these solutions, according to Arrhenius, that the salts are more or less split up into charged constituents—the ions. Different compounds (consisting of electrically neutral molecules) exhibit very different tendencies to split up into charged ions on solution in water. This tendency to split up into ions cannot be utilised as a source of electrical energy, for the positive charges cannot be separated in space from the negative charges simultaneously produced:—

$$NaCl = Na^{\cdot} + Cl'$$
$$H_2SO_4 = 2H^{\cdot} + SO_4'', \&c.$$

(A point denotes a positive, a dash a negative charge; thus, a sodium-atom is represented by Na, a sodium-ion by $Na^{\cdot}$.)

An electrically neutral substance may withdraw from existing ions their charges, as when copper is precipitated from copper sulphate by metallic iron—

$$Cu^{\cdot\cdot} + Fe = Cu + Fe^{\cdot\cdot}.$$

In this case a separation in space is possible, and the reaction can be used as a source of electrical energy. If in Fig. 1 both metals simply dip into a dilute solution of sodium sulphate, no appreciable (certainly no permanent) current passes along the wire. But if crystals of copper sulphate are introduced so that the copper is surrounded with copper sulphate solution, a current is set up. The sulphuric acid anions SO_4'' play no direct part in maintaining the current; they are only necessary as determining the presence of a sufficiently large number of cations to perform this function.

When an ion is multivalent, a *part* of its charge may be removed by an electrically neutral substance; as when ferric ions are reduced by metallic iron to ferrous ions—

$$2Fe^{\cdot\cdot\cdot} + Fe = 3Fe^{\cdot\cdot}$$

If in Fig. 2 the iron and platinum simply dip into a solution of common salt, no current is produced. But, if ferric chloride be introduced so as to surround the platinum

Fig. 1.

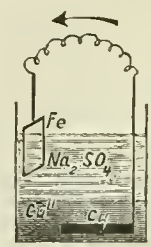


Fig. 2.

with ferric ions, a current is set up. The ferric ions transfer one-third of their charge to the iron plate, thus enabling it to form ferrous ions, and this goes on until all the ferric ions are themselves transformed into ferrous ions. Further examples of the formation and reaction of ions are given. One of these is a reversible process. Ferric chloride can set free iodine from potassium iodide; on the other hand, iodine is absorbed by ferrous salts with formation of ferric salts. Then the reaction—

$$Fe^{\cdot\cdot} + I \rightleftarrows Fe^{\cdot\cdot\cdot} + I'$$

can proceed in either sense until a certain state of equilibrium is attained.

The process of ionisation is itself reversible, as typified by the reaction—

$$HCl \rightleftarrows H^{\cdot} + Cl'$$

In solutions such as are of practical importance, no electrolyte is completely split up into ions. The grade of dissociation depends upon the nature of the substance, the nature of the solvent, and the concentration of the solution. When a bottle containing a saturated solution of hydrochloric acid gas in toluene is opened, it fumes in the air just as the saturated aqueous solution of the gas does. But in the toluene solution the hydrochloric acid is not appreciably split up into ions. A galvanoscope indicates no current when two electrodes connected with a battery of E.M.F. 72 volts, are dipped into the solution. The ions are wanting, and so there is nothing to transfer the electricity. Now, the criterion of an acid is the presence of hydrogen-ions. As these ions are wanting in the toluene solution, it ought not, for example, to be able to evolve carbon dioxide from calcium carbonate; and as a matter of fact it does not. If water be added, carbon dioxide is given off, and the solution conducts well.

We must refer to the original for explanations, based on the electrolytic dissociation theory, of other interesting reactions and experiments, such as the following. Water saturated with carbonic acid gas makes an acid solution of about $\frac{1}{10}$ normal strength; but it only turns methyl orange slightly red. An acetic acid solution of $\frac{1}{100}$ normal strength produces a much more distinct red colour. A mere trace of sodium bicarbonate restores the methyl orange in the carbonic acid solution to its original colour. The acetic acid solution behaves in a similar way towards an acetate; but here a larger quantity of the salt is requisite. Sulphuretted hydrogen precipitates black iron sulphide from a dilute aqueous solution of iron acetate. If the iron solution be acidified with acetic acid beforehand, it remains clear on passing in sulphuretted hydrogen; but if sodium acetate be also added, the iron sulphide is precipitated in spite of the presence of the same amount of free acetic acid. Strong acids behave quite differently. A single drop of hydrochloric acid turns a solution of methyl orange bright red; and, however, much sodium chloride be added, the colour is not altered. Sulphuric acid is considerably weaker than hydrochloric. A similar experiment with sodium sulphate shows that the addition of a very large quantity of the salt does noticeably diminish the action of the acid.

Some of the most interesting ionic reactions are those in which ions disappear as such and reappear in combination with other ions or molecules. There are certain reactions which are exhibited by all ordinary copper salts, and we conclude that these reactions are characteristic of the copper ion; for there is no other constituent which is common to all simple copper salts. But we know of solutions containing copper which do not exhibit these characteristic reactions, and we conclude that the copper is present in them, not as an independent ion, but as a constituent of a complex ion. Now we have every ground for assuming that solutions which are abnormal in their chemical behaviour will also prove to be abnormal in their electrical behaviour. An experiment is described proving that the assumption is quite correct in the case of Fehling's solution: for when this solution is electrolysed, the copper in it, instead of being transferred as a cation in the direction of the current, is transferred as a constituent of a complex anion in a direction opposite to that of the current.—D. E. J.

Aqueous Solutions, Electrolytic Decomposition of. W. Nernst. Ber. **30**, 1547—1563.

The electrolysis of water, at its discovery looked on as a direct decomposition, was later regarded as a secondary action, depending for its accomplishment on the presence of electrolysable substances in solution. More recently still, thanks to the work of Ostwald, Arrhenius, and van't Hoff, the idea of its being a primary decomposition has been reinstated. The modern theory of osmotic pressure and of electrolytic dissociation makes possible more definite views than formerly; the leading idea of this theory is to ascribe to the free ions all the properties of electrically neutral molecules, together with others arising from their electrical charges.

The electrolytic solution or precipitation of a metal is the resultant of opposing tendencies: on the one hand, the electrolytic "solution-pressure," or tendency of the metal to dissolve; on the other, the osmotic pressure of the dissolved ions and the electrostatic force with which the liquid, positively charged by the "initial solution" of metallic ions, opposes the introduction of more. When these opposing tendencies are equal, there is equilibrium; and if we now charge the metal positively or negatively, we have further solution or precipitation till equilibrium is restored.

The formula advanced by the author shows that the potential difference between metal and liquid is directly proportional to the "solution pressure," and inversely proportional to the osmotic pressure of the dissolved ion. The sign of this potential difference will be positive or negative as the electrode yields on solution cations or anions, and in the case of a voltaic cell with two metals and one liquid, the E.M.F. will be the difference between the potential differences at the metal-liquid contacts. Moderate changes in the concentration of either dissolved ion will affect the E.M.F. but slightly; but a *great* reduction in the concentration of one ion may alter the sign of E, or change the direction of the current. This has been experimentally shown by Ostwald in the case of the Daniell's cell, by adding potassium cyanide to the liquid in contact with the copper. The electrolytic cell differs from the battery cell in that solution or precipitation is going on at both electrodes—not solution at one and precipitation at the other; and hence the minimum E.M.F. which must be applied to overcome the potential differences of polarisation is the sum of the potential differences at the electrodes.

In the case of mixed electrolytes, Le Blanc has shown clearly, that as we raise the E.M.F. in the circuit, electrolysis of one of these takes place as soon as the minimum E.M.F. for that one is overstepped; and he has pointed out the applicability of graded E.M.F. for analytical separations. As a particular case of the principle, he has shown that in the case of many solutions, the water needs a lower minimum E.M.F. than the dissolved substance, so that a direct electrolysis of water takes place if too high E.M.F. be not used: in proof of this, he shows that the electrolysis of the most varied salt solutions in which the explosive mixture, $H_2 + O$, is evolved, needs the same E.M.F., 1·68 volt. If this minimum E.M.F. be greatly exceeded the salt also is decomposed, and then the accumulation of alkali at the cathode, and of acid at the anode, rapidly changes the concentration, and the progress of the decomposition is complicated by diffusion and convection currents. For practical purposes, electrolysis of neutral solutions is to be avoided.

While the requisite E.M.F. for the electrolysis of hydrochloric acid (1·3 volt) is also that yielded by a chlorine and hydrogen gas battery, the E.M.F. of a hydrogen and oxygen gas battery (1·08 volt) is far below that needed (1·68 volt) for the electrolysis of water or of one of the solutions just referred to. Le Blanc has attempted to explain this on the assumption that the liberated oxygen while in solution is in a state of supersaturation; but experiments of Glaser and the author, in which sulphuric acid and potassium hydroxide solutions were electrolysed by gradually increased E.M.F.'s have shown that an E.M.F. of 1·08 will decompose water if a sufficient surface of anode is given, and that different ionic decompositions correspond with different E.M.F.'s, the lower E.M.F. (in the case of water) working upon ionic oxygen, which exists but in low concentration, the higher with ionic hydroxyl, which is present in much higher concentration.

The following table shows the necessary "decomposition-electromotive force" for each ion in normal concentration, hydrogen being taken as zero:—

Cations.		Anions.	
Ag	− 0·78	I	0·52
Cu	− 0·34	Br	0·94
H	0·00	O	1·08
Pb	+ 0·17	Cl	1·31
Cd	+ 0·38	OH	1·68
Zn	+ 0·74	SO_4	1·90
		HSO_4	2·60

From this table many practical conclusions can be drawn:—Zinc bromide, for example, needs for decomposition 0·74 + 0·94 = 1·68 volt. The separation of copper from silver, or of iodine from bromine, is seen to be easily practicable, electrolytically, as the figures for each pair differ so widely; and so forth.

It is to be noted that to accomplish electrolysis at any considerable rate, with E.M.F.'s just above minimum, we must have the ions in sufficient concentration; for this reason, while it is true that the direct electrolysis of the water of a solution may be possible, it is usually a secondary action, the higher concentration of the salt ions causing electrolysis of the salt rather than of the water to take place chiefly, in spite of the lower E.M.F. at which the latter can occur. The same thing explains the fact that chlorine displaces oxygen, for example, so much less readily and rapidly than bromine, though the minimum figures for oxygen and for bromine lie so near together.

The same fundamental principles throw light on the decomposition of water by metals with evolution of hydrogen. From the formula for the E.M.F. of a cell, we see that the conditions favourable to the displacement of hydrogen are, high osmotic pressure of the hydrogen ions, low osmotic pressure and high solution-pressure on the part of the metal. Potassium, for example, decomposes water on account of its enormous solution-pressure. Zinc decomposes water in acid solutions because the concentration of the hydrogen ions is considerable; and in alkaline solutions, though the hydrogen concentration is slight, because the zinc concentration, through the formation of zincates, is slighter still; but it will not decompose water in a neutral solution of zinc sulphate, where the zinc concentration is high and the hydrogen concentration low. In the electrolysis of a solution of a salt the same principles still hold: metal or hydrogen will be precipitated according to the solution-pressure of the metal and the relative magnitudes of ionic concentration of metal and of hydrogen. The practical bearings of this are obvious.

Attention is drawn to the formation at the electrodes, of compounds, by the coalescence of two similar ions deprived of their electric charges—not only molecules of elements,

but such bodies as $H_2S_2O_8$ and $K_2C_2O_6$. Both this, and the variation of osmotic pressure at the electrodes by altering the E.M.F. applied, promise, especially in the domain of organic chemistry, to be fruitful in the preparation of new compounds electrolytically.—J. T. D.

Iodoform, Electrolytic Preparation of. K. Elbs and A. Herz. Zeits. f. Elektrochem. **4**, [4], 1897, 113.

See under XX., *page* 932.

Plumbates, Alkaline Earth [$XPbO_3$]; *Manufacture and Properties of.* G. Kassner. Rev. Chem. Ind. 1897, **8**, 227.

See under VII., *page* 913.

(*B.*)—ELECTRO-METALLURGY.

Chromium, Electro-Metallurgical Process for obtaining. Aschermann. D.R.P. No. 93,744, June 30, 1896. Zeits. für Elektrochem. **4**, [8], 214.

A gas-tight electric smelting furnace of cast steel, with a very dense movable carbon electrode, is used. A tightly fitting crucible of graphite is placed within the outer case of steel, and is charged with 10 parts of chromium sesquioxide and 23 parts of antimony sulphide, or with 10 parts of chromium sesquioxide, 10 parts of sulphur, and 23 parts of pure antimony. The former mixture is to be preferred, since a better yield is obtained when it is used, and the crucible suffers less. The apparatus is closed, and a current of from 20—25 ampères is passed through it. This suffices to melt the mixture, and causes an exothermic chemical reaction to occur. When the reaction is completed, a deposit, chiefly amorphous, of antimony trioxide, antimony sulphide, and chromium sequioxide will be found upon the upper part of the crucible and furnace, whilst in the crucible itself there will be found a molten alloy of chromium and antimony, distinctly crystalline in structure. By repeated melting in this furnace it is possible to remove every trace of antimony from the chromium, but this method of purification is inconvenient and costly. A simpler and less expensive plan is to break the alloy when cold into small pieces, and to drive out the antimony from these by volatilisation at a white heat over an ordinary coke or coal fire. Molten chromium takes up, or dissolves, a considerable amount of carbon. On cooling, this carbon separates out as graphite. Chromium is volatilised at temperatures over 2,000° C.

—J. B. C. K.

XII.—FATS, OILS, AND SOAP.

Degras, Artificial, Manufacture of. C. Baron. Rev. Chim. Ind. 1897, **8**, 225.

A vessel holding about 6,000 kilos., built of tinned steel in the shape of a cylinder with an inverted cone at the base, provided with an air agitator, a dry steam coil, and several draw-off cocks at different positions, is charged with 1,000 kilos. of neutral wool fat (extracted with petroleum spirit) and 5,000 kilos. of cod-liver or whale oil. The liquid is heated by means of the steam coil, agitated for three hours, then allowed to rest and cool for the same period, and the water is withdrawn. The oil is again heated to 40°, 150 kilos. of hydrogen peroxide and 450 kilos. of water added, and the whole agitated for five hours at a pressure of two atmospheres. The resulting product forms an excellent moëllon, having a yellow colour, and being easily emulsified and absorbed by the skins. It is very important that the wool grease shall be free from (sulphuric) acid, lest this should dissolve traces of iron, and so give rise to the darkening of the leather owing to the formation of iron inks. (See also p. 937.)—F. H. L.

Degras, Genuine and Artificial, Distinction between. F. Jean. Rev. Chim. Ind. 1897, **8**, 227.

See under XXIII., *page* 937.

Beeswax, The Examination of. S. Weinwurm. Chem. Zeit. 1897, **21**, [52], 519.

See under XXIII., *page* 939.

XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

(*A.*)—PIGMENTS, PAINTS.

Red Lead, The Dissociation of. H. Le Chatelier. Bull. Soc. Chim. 1897, **17**, 791—792.

Red lead at the upper closed end of a vertical tube, the lower end of which was connected with a mercury manometer, was heated to temperatures which were determined by the author's thermopile, and its dissociation pressures found to be, at 445° C., 5; 500°, 60; 555°, 183; and 636°, 763 mm. Thus in air (oxygen at 150 mm. pressure), red lead cannot be formed above 550° C., but is produced most rapidly about 500° C.

Red lead was found to undergo allotropic change at 580°, the oxide melting at 830°. The author considers that when melted litharge is exposed to air, red lead is formed, and is dissolved as formed in the litharge, the dissociation pressure being, in effect, lowered through dilution of that red lead in the excess of litharge. On subsequent cooling, the red lead tends to separate out in the solid form and then suddenly finding itself under a pressure of 30 atmospheres, it is decomposed in the act of solidification and thus breaks up the mass.—J. T. D.

"Mosaic Gold" (Crystalline Stannic Sulphide), Preparation of. J. Lagutt. Zeits. angew. Chem. 1897, 557.

According to most of the text-books, the reason for including ammonium chloride in many of the recipes used for preparing bisulphide of tin, is to prevent the temperature of the mass rising too high and thus causing the formation of the monosulphide. This explanation is evidently erroneous, and the rôle of the ammonium salt is rather a chemical one; for the author finds that in order to obtain a satisfactory product it is necessary to have the tin present in a volatile form, or to add to the other ingredients some substance which shall cause the metal to assume this shape, such as mercuric chloride, bromine, hydrochloric acid, or ammonium chloride.

The following formulæ give good results:—For a pale yellow mosaic gold, 50 grms. of crystallised stannous chloride and 25 grms. of flowers of sulphur; for a reddish shade, 50 grms. of 50 per cent. tin amalgam, 25 of crystallised stannous chloride, 35 of ammonium chloride, and 35 of sulphur. The materials are finely powdered and are ignited gradually in a glass retort covered with asbestos in the flame of a large blowpipe, which should be moved about periodically in order that the charge may be equally heated all through. During the operation the colour of the mass should be dark brown; if it become black owing to the formation of stannous sulphide, the flame should be lowered. The yield is about 57 per cent. of the theoretical, calculated on the amount of tin employed.—F. H. L.

Driers and their Effect on Linseed Oil. M. Weger. Zeits. angew. Chem. 1897, 401, 542, and 560.

The first portion of this article consists chiefly of a criticism of Amsel's paper (this Journal, 1897, 685).

Inasmuch as a substance can only act on linseed oil as a drier if it be in a state of true solution, the chief advantages that the resinates and linoleates of lead and manganese possess over the metallic oxides formerly employed, lie in their ready solubility at lower temperatures, a phenomenon which evidently demands that they shall be compounds and not simply mixtures. At the present time there is no means of deciding whether the resin and linseed acids found on analysing these driers are present as metallic salts, or whether they merely exist in the free state or as esters, side by side with uncombined inorganic bases; and the suggestion recently made by Amsel (*loc. cit.*) that the point may be settled by a simple determination of the total mineral matter is obviously quite valueless. Until, therefore, some new analytical process has been worked out which will throw more direct light on the constitution of these bodies, it is necessary to estimate the soluble inorganic matter (*i.e.*, the lead or manganese soluble in such liquids as oil of turpentine, &c., because combined with resin or linseed

acids) and, by means of the combining proportions already given (this Journal, 1896, 728), to calculate this either into resinate or linoleate, according to the average acid or saponification number of the whole sample. As an instance, a fused mangano-lead resinate, contained 9·90 per cent. of soluble Pb, and 1·43 per cent. of soluble Mn, other metals being absent. The material was therefore composed of $(9 \cdot 9 \times \frac{100}{24} =)$ 41 per cent. of lead resinate and $(1 \cdot 43 \times \frac{100}{5 \cdot 3} =)$ 27 per cent. of the corresponding manganic salt, or 68 per cent. of active ingredients in all; and the amount of free resin (colophony) must have been 30 or 35 per cent.

Amsel's plan of estimating the moisture in the soluble driers is not of much use, for the resinates of lead and manganese obtained by precipitation, generally contain about 6 per cent. of water in their air-dried condition: and it never rises to 20 or 33 per cent. Linoleates prepared in a similar manner are very difficult to dry, often retaining water in distinct drops, whence it follows that they are of little practical value, and they are, indeed, seldom if ever met with commercially. Determination of the moisture, in fact, only serves to distinguish between fused and precipitated resinates, which cannot always be done with certainty by means of the microscope.

The author has investigated the action of a number of other compounds of manganese on linseed oil, including the monoxide, sesquioxide, and dioxide, each in the hydrated state, ordinary black oxide, chloride, sulphate, nitrate, chromate, borate, carbonate, oxalate, succinate, citrate, tartrate, benzoate, formate, and acetate. The salts of organic acids just mentioned, with the exception of the acetate, appear unsuited for the manufacture of varnishes, as they offer no advantages commensurate with their high prices. The acetate yields a well-drying varnish on heating with the oil for two hours at 120°; the nitrate is decomposed between 120° and 140°, and the resulting oxide dissolves completely at 150° to 170°; the black oxide involves a temperature of 250°. The hydrated oxides all dissolve between 170° and 220°, but before being taken into use they should be examined as to the amount of metal and available oxygen they contain, for they vary very much in purity, as is shown in the annexed table:—

Composition of Commercial Manganese Hydrates.

	Mn.	Available O.
	Per Cent.	Per Cent.
$MnO_2.H_2O$	38·41—53·67	10·14—10·87
$Mn_2O_3.H_2O$	44·05—52·63	5·55—11·05
$MnO.H_2O$	51·70	5·32

The proportion of manganese in the borate of commerce ranges from 5 to 22, being usually about 15, and the substance apparently consists of $2MnO.3B_2O_3.3H_2O$. Whilst in the case of an addition of $2\frac{1}{2}$ per cent. of manganese sesquihydrate agitated and intimately mixed in the cold by means of a current of air, with linseed oil, from 20 to 30 hours were required to show drying effect, and with 10 per cent. of manganese sulphate rubbed up for four hours (cold) with the oil, some 30 hours were required, yet in the case of 1 per cent. of manganese borate (containing 20 per cent. Mn) after rubbing up for one hour (cold), the time for drying was only 7 to 15 hours. Thus, whilst an undissolved drier is without action, a long-continued and extremely intimate mixture of drier with oil, can, even in the case of other than completely soluble preparations, to a certain extent replace any raising of the temperature. The ready solubility of borate of manganese presumably depends on the acidity of the oil, for the acid number of ordinary samples is about 2·5, and this represents a capacity for dissolving 0·12 per cent. of Mn (as monoxide), which is amply sufficient to yield a quick-drying varnish.

Turning to the influence of driers in general on linseed oil, it should be noted that an exact determination of the speed at which varnish dries is a problem of considerable difficulty, and two different observers working on one sample under apparently similar atmospheric and other conditions may find discrepancies amounting to 25 per cent. in the time required. The drying is affected by alterations in the temperature, moisture, and rate of motion of the air, and, notably, by the degree of light to which the glass plates carrying the films of oil are exposed. Variations of one-half to one hour may often be experienced between two trials of the same specimen of varnish at different times in one day, or of 50 per cent. between tests made in the day and at night; while a sample drying in two hours in the direct rays of the sun may take five or even eight hours if it is only placed in diffused daylight. For these and similar reasons it is impossible to express the value of any particular drier, or the speed of any definite varnish, in absolute terms; and it is indispensable, therefore, that all the materials which have to be studied shall be tested simultaneously, or that they shall be examined in juxtaposition to a standard varnish the behaviour of which is thoroughly understood.

To prepare a suitable standard, manganese resinate might be dissolved by heating to 150° in about four times its weight of a pure linseed oil kept specially for the purpose, the solution filtered, and diluted till it contains a convenient amount of metallic manganese, *e.g.*, 0·1 per cent. This may be rendered still weaker if necessary; but it is important to observe, for reasons indicated hereafter, that the varnish must be freshly made up each time it is employed.

It is usually believed in the trade that there is a well-defined limit to the proportion in which lead or manganese may be advantageously introduced into linseed oil, because above a certain point the drying power of the varnish is thereby diminished. Evidence of this retrogression the author cannot discover; and while the rapidity of drying at first increases with every addition of drier, provided always that the metal is actually dissolved, excess of the latter simply produces no effect on the speed. As the result of a large number of experiments, for full details of which the original communication must be consulted, Weger finds that for all practical purposes the maximum in the case of manganese resinate is 0·25 per cent. of metal, or, if a mangano-lead resinate be employed, 0·5 per cent. of Pb and 0·1 per cent. of Mn. Corresponding tests with the linoleates do not yield equally clear indications, but it is certainly useless to add more than 0·2 per cent. of Mn in the shape of its linoleate, and probably 0·1 per cent. would be preferable. Such quantities as these, moreover, must only be introduced into the oil when speed is the primary consideration; for it seems to be well recognised that the durability of the film is seriously affected by increasing the amount of foreign matter present, or, in other words, that linseed oil by itself yields the most permanent varnish of all.

Varnishes containing manganese linoleate, and occasionally those with the corresponding borate, do not attain their full drying power until they are eight days old; while such as are made with mixtures of lead and manganese, or with litharge and black oxide of manganese in the old-fashioned way, are apt to diminish in speed on keeping, more particularly during the first few weeks. This peculiarity does not of itself negative the idea that a varnish is improved by age, for rapidity of drying is manifestly only one of a number of properties sought after in these products.

Attempts were made to discover if there is any practical difference between the activity of a definite amount of lead or manganese in the form of the linoleate and the same quantity of metal as resinate; some of the results seem to be slightly in favour of the fatty salt, but the evidence is not yet conclusive. Other experiments show that it is immaterial whether the drier (Pb + Mn) is dissolved in the oil at ordinary atmospheric temperature or by heating them (drier and oil) together at 150° to 260° C.—F. H. L.

PATENTS.

Anti-fouling Composition or Paint for Ships, A New or Improved. F. Scott, Swansea. Eng. Pat. 25,549, Nov. 13, 1896.

One cwt. of tallow, two of sulphur, two of resin (colophony), 12 lb. of verdigris, and 1 gall. of turpentine are mixed together, and the composition is used either hot or cold as may be more convenient.—F. H. L.

Coating Ships' Bottoms to Prevent Fouling and Corrosion, Impts. in; applicable also to the Coating of Tanks and the like to Prevent Corrosion. A. C. Furse, London. Eng. Pat. 25,675, Nov. 14, 1896.

This consists of finely-divided metallic aluminium suspended in a vehicle composed of liquid celluloid, or shellac dissolved in a suitable solvent.—F. H. L.

Manufacture of [Red] Lead Pigments, Impts. in or connected with the. J. Fairlie, Glasgow. Eng. Pat. 26,322, Nov. 21, 1896.

The object of this patent is to treat red lead in such a way that it shall not settle or deposit from linseed oil after it has been ground therein; so that it may be put on the market in the form of a paste, as is done with white lead. To the dry pigment are added sodium chloride and sulphate, potassium iodide and bromide, magnesium chloride and sulphate, and calcium chloride in amounts not exceeding 1 per cent. altogether; and the whole is mixed, "levigated with water," and dried.—F. H. L.

Glaze Paint specially for Painting Ships' Bottoms, Process for the Production of. L. de Bruycker, Hamburg. Eng. Pat. 7128, March 18, 1897.

250 grms. of aloes are dissolved in 100 grms. of methylated spirit, 4,000 grms. of "thick turpentine," and 750 grms. of linseed oil added, and the whole boiled till no scum is formed. 950 grms. of linseed oil are boiled separately with 25 grms. of lead acetate, 10 grms. of white lead, and 15 grms. of litharge until the mass becomes dark brown, and it is then combined with the first mixture. Finally, the composition is thinned with 750 grms. of petroleum spirit and 250 grms. of oil of turpentine, the desired pigment is introduced, and the paint is ground in the usual manner.—F. H. L.

Wood-Stain, New or Improved. H. E. Junghans, Schramberg, Württemberg. Eng. Pat. 7540, March 23, 1897.

The application of a dye dissolved in water or alcohol to wood has the disadvantage of causing the grain to "rise"; and when the surface is sandpapered again to make it smooth, the stain is apt to be removed irregularly. To obviate this trouble, the inventor uses as a solvent for his colouring matter, benzene, turpentine, or the anthracene oil known in commerce under the name of carbolineum.
—F. H. L.

Anti-Fouling and Anti-Corrosive Paint, An Improved. R. Hingston and J. C. Rankin, Sydney, N.S.W. Eng. Pat. 8811, April 6, 1897.

The following substances are ground together:—Red ochre, 38 lb.; yellow ochre, 29; purple oxide of "zinc" ("purple brown"), 3; "double-boiled" linseed oil, 19, and terebene, 15 lb.; also turpentine and methylated spirit, of each 8 oz. Finally, 5 lb. of mercuric chloride, 1 lb. of copper sulphate, and 1 lb. of white arsenic are added in fine powder, and the whole is well stirred up.—F. H. L.

Insulating and Weather-, Acid-, and Fire-Resistant Material applicable as a Paint, or for other Purposes; An Improved. J. Stocker and H. Zander, Rathenow, Germany. Eng. Pat. 16,490, July 12, 1897.

Three parts of caustic alkali are dissolved in 20 of soda or potash water-glass, 10 of fireclay are added, and, after standing two hours, 2 of clay powder, 6 of "asbestos powder" or "wool," and the desired colouring matter are introduced, and the whole mixed together. For impregnating canvas, sail-cloth, &c., this composition is treated at 100° C., with 10 parts of earth-nut oil and 5 of hydrochloric acid; for use as a waterproof paint, it is ground with 5 parts of earth-nut oil alone; and for the preparation of an insulating mass or a substitute for hard rubber, it is mixed with oil as in the last case, but with the addition of 2 parts of isinglass. The plates, &c. made from it are moulded at 100° to 120° C. under hydraulic pressure.
—F. H. L.

Composition specially adapted for Printers' Inking Rollers and the Like, Improved Compound or J. W. White, London. Eng. Pat. 17,123, July 20, 1897.

This composition consists of gelatin, 1 lb.; golden syrup, 3 lb.; oxide of zinc, 1 oz.; glycerin, 1 gill; and water, *quant. suff.* The addition of the zinc oxide to the ordinary formula causes the rollers to have an opaque white surface; and it is, therefore, easy to see whether they are properly clean, before a pale-coloured ink is applied to them.
—F. H. L.

White Lead, Improved Apparatus for Use in the Manufacture of. A. H. Eyles, Pelhamville, New York, U.S.A. Eng. Pat. 19,030, Aug. 17, 1897.

This specification describes an improved carbonising machine for use in the manufacture of white lead by the precipitation process. A cylindrical vessel mounted on hollow, horizontal axles is provided on its inner surface with a number of bars of perforated angle-iron, bolted to the walls in a direction parallel to its axis, but divided into several short lengths, and so fixed that each length "breaks joint" or is "staggered" as regards its neighbours. These pallets or beaters act as agitators, and as the barrel revolves, its contents are thoroughly and uniformly exposed to the action of the gases present in the vessel. The inlet pipe is branched so that carbonic acid and oxygen can be introduced together; and both inlet and outlet are fitted inside the vessel with baffle plates.—F. H. L.

(*B.*)—RESINS, VARNISHES.

Varnish, Unboiled Linseed. H. Amsel. Chem. Zeit. 1897, **21**, 690.

An unboiled linseed varnish may be defined as a product which has been made by dissolving a metallic resinate or "oleate" in linseed oil at a temperature not exceeding 180° C., in contradistinction to one prepared by the original process of heating the oil with metallic oxides at or above 250°. The author has examined a large number of these materials—some of his own manufacture, others commercial specimens—with regard to their speed of drying and the character of the films they yield; and he now (this Journal, 1895, 814) concludes that they are at least equal in value to the old high-temperature varnishes. Whenever the samples do not dry satisfactorily on glass in 24 hours, he employs Kissling's process of determining the weight of oxygen they absorb in a definite period of time, either in the sun or in diffused light; but as both tests lead to the same result, and the latter takes much longer to carry out, he prefers the former method. "Ozonised" oils in general, dry more slowly than boiled varnishes, but they yield remarkably smooth and brilliant films which should render them specially suitable for many purposes.

To distinguish between resin oil and mineral oil in linseed oil varnish, the sample may be rubbed down with zinc oxide; if the mass thickens to a cheese-like consistency in 24 hours, the former is present, whereas mineral oil has no such effect. Ten or more per cent. of cotton-seed oil in a varnish can also be detected in a precisely similar manner.
—F. H. L.

Balsams, Resins, and Gum Resins, Rational Examination of. K. Dieterich. Chem. Rev. Fett u. Harz Ind. **4**, [17], 233.

See under XXIII., *page* 940.

PATENT.

Varnishes, Paints, and the like; Impts. in or connected with the Manufacture of. I. Goldblum, Lublin, Russian Poland. Eng. Pat. 18,519, Aug. 10, 1897.

As a partial or total substitute for linseed oil varnish, 50 parts of common resin (colophony) are dissolved in 100 parts of benzene or some similar solvent, 12 to 15 parts of crystallised sodium carbonate are added, and the whole finally filtered or decanted. "Washing" paints may be made by grinding pigments to a stiff paste with boiled oil, allowing the mixtures to dry, and then grinding the resulting powder with the above-mentioned vehicle.—F. H. L.

(C.)—INDIA-RUBBER, &c.

Gutta Percha, Composition of. J. A. Montpellier. Rev. Chim. Ind. 1897, **8**, 237. (See also this Journal, 1897, 815.)

Gutta percha consists of a mixture of three resins: *gutta*, $C_{20}H_{32}$; *fluavile*, $C_{20}H_{32}O$; and *albane*, $C_{20}H_{32}O_2$. The first is the body to which the material owes its industrial value, and it forms from 40 to 85 per cent. of the whole. At 10° to 20° C. it is white, soft, but not elastic; at 45° it becomes yellow and pasty; between 100° and 110° it softens; and at 130° it melts, boils, and is decomposed into an oil and a gaseous hydrocarbon. Gutta is soluble in chloroform, toluene, and carbon bisulphide; insoluble in dry alcohol, and also ether—at least, after treatment with the former. It oxidises in the air, and is decomposed by hot nitric acid, formic and hydrocyanic acids being formed. Hydrochloric acid converts it into a dark brown substance. Fluavile is a yellow, brittle, amorphous resin, softening about 50°, and melting at 100°—110°. It is heavier than water. It dissolves cold in dry alcohol and ether, benzene, toluene, oil of turpentine, carbon bisulphide, and chloroform. Strong alkalis, weak acids, and concentrated HCl are without action. Albane is a white resin, crystallising from hot alcohol, softening at 160°, and melting, without decomposition, at 175°—180°. Its solubility resembles that of fluavile, except that alcohol only takes up 5·1 per cent. in the cold and 54 per cent. at the boiling point. In presence of acids and alkalis it also behaves like fluavile. Heated to 130 it loses one molecule of water.

Attempts to value the gutta percha of commerce should be based on the determination of the proportion of pure gutta, moisture, and other impurities.—F. H. L.

XIV.—TANNING, LEATHER, GLUE, SIZE.

Canaigre, Cultivation of. W. Eitner. Der Gerber, **23**, [554], 232.

E. Andreis, of Turin, in a pamphlet entitled "Il Canaigre," gives a very full description of its properties, and also advocates the cultivation of the root in Italy.

This has been already undertaken in Arizona and New Mexico by several companies. The commercial results are yet unknown, but there is little doubt that in America, canaigre extract will to a great extent replace gambier. The author has made several attempts to grow canaigre in Vienna and in Bosnia, latitude 43°, but without success; the cultivation from seeds is rarely successful. A later trial in Smyrna has been successful on a small scale, but as the first year's roots never contain much tannin, it will be some time before the experiment can be pronounced a success. It is questionable from these trials whether canaigre will grow in a latitude much north of its original habitat. The author, however, is of opinion that it will be successfully grown in the Levant.—J. T. W.

Palmetto Extract. W. Eitner. Der Gerber, **23**, [550], 184.

This is the extract of the root of a kind of Palmetto palm (*Corypha Palmetto*) which is grown in Georgia and Florida. An analysis of the extract (30° B.) gave—

	Per Cent.
Tanning matters	18·01
Sol. non-tannin	20·06
Water	49·96
Insoluble	2·71
Ash	9·26

The amount of tannin is not high for an extract, while the non-tannin is considerable and easily fermentable, so that the liquors soon become sour.

The ash consists principally of potassium chloride, which appears to be due to the roots having been treated with potash, and afterwards neutralised with HCl.

Used alone, palmetto extract produces a hard brittle leather, of a dirty reddish-grey colour; with other materials, however, it makes a good sole leather. It is best used in the final processes, and not in the colouring pits.—J. T. W.

Fermentation Phenomena in Tan Liquors. F. Andreasch. Imp. Research Laboratory, Vienna. Der Gerber, **23**, [541], 69; **23**, [543], 95.

A supply of nitrogenous substance is absolutely necessary for the proper production of acid. The author shows that this is furnished by the hides themselves. Practical fermentations were conducted in tan liquors from different sources, the nitrogen in the fermented liquors being estimated, and also its mode of combination.

Old tan liquors contain albumoses and peptones along with amido acids and alkaline products of putrefaction. Coriin, hide fibroin, and semiglutin are also present; the latter, or a very similar body, occurring in large quantities in limed hide. Bated skins introduce different decomposition products of albuminoids by bacteria. The nature of the nitrogenous compounds was examined (after freeing the liquid from tannin by gently heating with calcined magnesia) by means of the following tests:—

1. The xantho-protein reaction. 2. Millon's reagent. 3. Precipitation with $NaClO_3$ and HCl. 4. The Biuret reaction.

The following table (A) gives some nitrogen analyses of tan liquors in the tannery:—

Table A.

Number of Colouring Pits.	Per Cent. N in 100 parts Dry Extract.	Grms. N in 100 c.c. Liquor.	——
I. (weakest)	0·5621	0·0036	From a series of 9 sole-leather colouring pits. Pine bark liquors.
III.	0·4202	0·0138	
VI.	0·3233	0·0144	
VIII.	0·2508	0·0109	
IX.	0·2113	0·0082	
I. (weakest)	0·3240	0·0051	Very old calf colouring liquor. Oak and pine barks.
II.	0·3612	0·0064	
III.	0·3811	0·0068	
IV.	0·3481	0·0065	
V.	0·3202	0·0053	
VI.	0·2459	0·0048	
I. (weakest)	0·3292	0·0045	From a series of 10 sole-leather colouring pits. Pine liquors.
III.	0·2429	0·0043	
V.	0·2500	0·0045	
VII.	0·1917	0·0046	
IX.	0·1514	0·0035	
X.	0·1467	0·0025	

It will be noticed (Table B, this Journal, 1897, 748) that the acidity of the liquors increases with the amount of nitrogen, though not always proportionately: this is owing to the irregular absorption of acids by the hides, besides which the nitrogenous bodies, even in the same series of liquors, are not always of similar nature. Comparing the N in the used liquors (Table A, above) with that of the original materials (This Journal, 1897, 748) an increase of N is shown, although there is less tannin, and this increase can only come from the hides.

Fermentations in presence of hide showed conclusively that the increased acidity depended solely on the nitrogenous substances present, and further, that not only is the soluble nitrogen utilised, but that the fresh or slightly tanned hides directly contribute towards the fermentation in a similar way to that in which coagulated albumin or precipitated casein in cheese, influence the formation of lactic acid.

The hide used in the experiments was sterilised in the following way:—Pieces of unhaired hide were shaken for several hours in a fresh sterilised lime liquor. The lime was then removed partly by washing in sterilised water and partly by passing CO_2 through the vessel. The hide then remained for three weeks in water saturated with CO_2 in the incubator at 25°, those pieces which gave no development of bacteria on a gelatin plate being used.

The following tables give the results of these experiments. The first table (B) gives fermentations in a solution of glucose 3 grms. per 100 c.c., with the necessary mineral salts, but no other nitrogenous material than the sterilised hide.

Table B.

	Grm. Lactic Acid in 100 c.c. after 3 Weeks at 30° C. Inoculated with Lactic Acid Bacterium from Tan Liquors.			
	Original Solution.	Original Solution. 4·2 grms. Hide per 100 c.c.	8·5 grms. Hide per 100 c.c.	17·0 grms. Hide per 100 c.c.
Total lactic acid	0·0153	0·1470	0·2600	0·5009
Lactic acid due to hide..	..	0·1017	0·2147	0·4556

Table C gives the results of fermentations of the most generally used materials. Pine-bark and myrabolan liquors were also employed after removal of the tannin, in order to ascertain its influence on the fermentation.

Limed cow-hide and calf skin, sweated cow-hide and bated calf skin were used. The figures refer to the weight of dry hide.

Except where there is a great excess of hide in comparison to the fermentable non-tannins, the author shows that the following law holds good :—The formation of lactic acid depends on the previous treatment of the hide, and with the same hides, is proportional to the weight of hide substance present.

Table C.

Fermentation with Lactic Acid Bacterium of Tan Liquors for 20 Days at 30° C.

Tanning Material.	Hide.	Per Cent. Tannin.	Per Cent. Non-Tannin.	Total Lactic Acid in 100 c.c. of Tan Liquor. Original Liquor.	Original Liquor + Hide. 4·5 Grms. per 100 c.c.	Original Liquor + Hide. 9·10 Grms. per 100 c.c.	Original Liquor + Hide. 18·25 Grms. per 100 c.c.	Lactic Acid per 100 c.c. due to Hide. 4·5 Grms. Hide.	9·10 Grms. Hide.	18·25 Grms. Hide.
Pine bark..........	Limed cow hide ..	0·9652	1·000	0·0520	0·1219	0·1975	0·3289	0·0699	0·1455	0·2769
		..	1·000	0·0430	0·1583	0·2781	0·5008	0·1153	0·2351	0·4578
	Sweated cow hide.	0·9652	1·000	0·0520	0·2711	0·4934	0·6413	0·2191	0·4414	0·5893
		..	1·000	0·0430	0·3152	0·5870	0·6951	0·2722	0·5440	0·6521
	Bated calf skin ...	0·9652	1·000	0·0520	0·2243	0·4016	0·7210	0·1723	0·3496	0·6690
		..	1·000	0·0430	0·2521	0·4612	0·7035	0·2090	0·4182	0·6415
Myrabolans	Limed cow hide...	1·9304	1·000	0·1340	0·2060	0·2753	0·4120	0·0720	0·1413	0·2780
		..	1·000	0·1285	0·2351	0·3440	0·5589	0·1066	0·2155	0·4304
	Sweated cow hide..	1·9304	1·000	0·1340	0·2853	0·4421	0·7342	0·1513	0·3081	0·6002
Sumac.............	Limed calf skin....	1·1650	1·000	0·1583	0·3054	0·4631	0·7253	0·1471	0·3048	0·5670
	Bated calf skin.....	1·1650	1·000	0·1583	0·3387	0·5234	0·7503	0·1804	0·3651	0·5920
Oak bark	Limed cow hide....	1·3710	1·000	0·0500	0·1345	0·2025	0·3483	0·0845	0·1525	0·2958
Hemlock bark	Ditto	1·4370	1·000	0·0563	0·1115	0·1683	0·2800	0·0552	0·1120	0·2237
Oakwood extract....	Ditto	1·6053	1·000	0·0611	0·1410	0·2343	0·3852	0·0799	0·1732	0·3241

—J. T. W.

Dégras, Artificial, Manufacture of. C. Baron. Rev. Chim. Ind. 1897, **8**, 225.

See under XII., *page* 922.

Dégras, Genuine and Artificial, Distinction between. F. Jean. Rev. Chim. Ind. 1897, **8**, 227.

See under XXIII., *page* 937.

PATENTS.

Tanning Hides and the like, Impts. in the Method of and Means for. J. Forster, Warrington. Eng. Pat. 22,103, Oct. 6, 1896.

The hides are suspended in the pits on frames mounted on wheels. The rails on which the wheels run consist of a series of inclined planes. A reciprocal motion being given to the frame, the hides are thereby lifted and lowered in addition to the horizontal motion. The motion is continued during the whole of the process.—J. T. W.

Leather, Artificial, or Leather Substitute, Improved Manufacture of. A. J. Boult, 111, Hatton Garden. From G. Brigalant, Barentin, France. Eng. Pat. 15,404, June 28, 1897.

Leather waste is shredded and made into a paste, textile substances such as hemp, flax, jute, &c. being added along with glue. The "fibroleum" thus prepared is made into sheets, compressed, dried, and rolled.

It may be used for any purposes for which leather or leather substitutes are employed.—J. T. W.

Leather or Skins, Washable, Impts. in or relating to the Manufacture of. J. L. Garcin, Grenoble. Eng. Pat. 16,477, July 12, 1897.

Skins are first tawed or partially tawed, and then tanned either by the usual process or by means of chrome.

Leather prepared in this way does not lose its suppleness and elasticity by the action of water; in short, it is claimed that a washable leather is formed.—J. T. W.

Manure, Manufacture of, from Waste Leather and Phosphate of Lime. W. E. Rowlands, Seacombe, Chester. Eng. Pat. 17,926, July 30, 1897.

See under XV., *page* 927.

Leather Substitute, Manufacture of and Apparatus for Use therein. Leon de Banville, J. F. Roulleau, and L. V. Rancelant, Paris. Eng. Pat. 24,885, Aug. 6, 1897.

The substitute consists of an intimate mixture of leather waste and rubber solution. The waste used is the fluff from the skins used in glove manufacture.

The mixture or paste is spread between two parallel webs by means of a special machine of which drawings are given. It is dried on the webs and afterwards embossed in imitation of Cordovan, &c.—J. T. W.

Tanning, Impts. in Apparatus for. E. Du Bois, Ralston, Penn., U.S.A. Eng. Pat. 19,874, Aug. 28, 1897.

The tan pits are fitted with frames the cross-bars of which are provided with a series of hooks above and below the centre of vibration, the upper series being in different vertical planes from the lower series on each bar; the frame is given a vertical motion within the vat, and the cross-bars of the frame an oscillatory motion by means of the mechanism illustrated, so that adjacent sides of leather are made to move in opposite directions. These motions thoroughly agitate the tan liquor and cause it to circulate freely between the leather "sides."—J. T. W.

XV.—MANURES, Etc.

Denitrification, Recent Experiments on. W. Somerville. Jour. Royal Agric. Soc., Eng., 1897, [3], **8**, 477—490.

The results of experiments made by Maercker and by Wagner, in which different plants grown in pots were variously manured, showed that in presence of sufficient

phosphoric acid and potash, less produce was obtained when dung was supplied than with nitrate of soda, and that dung in conjunction with nitrate of soda gave rise to a considerable decrease in the amount of produce as compared with nitrate of soda alone. It was further shown that horse dung may induce loss of nitrogen in bare soil. Garden soil (3·1 kilos.) containing potassium phosphate and nitrate of soda (N = 0·5 grm.) lost more than half the nitric nitrogen in six weeks after the addition of 600 grms. of dung. Assuming the pots to be about 6 in. deep, this would correspond with an application of about 125 tons of dung per acre mixed with the soil to the same depth.

There seems to be no doubt that the loss of nitrogen is caused by micro-organisms which occur in dung, in cereal straw, and, to some extent, in the soil. The power of liberating free nitrogen diminishes with the age of the dung, and Wagner thinks that the greater value of rotten, as compared with fresh manure is largely due to its diminished power of destroying the nitrates present in the soil. Addition of about 3 per cent. of superphosphate and kainite, respectively, to dung, increased its denitrifying power, both when the dung was turned over and when stored without turning over.

A large number of interesting results have already been obtained by the investigators already named, and by Stutzer, Pfeiffer, and others.—N. H. J. M.

PATENT.

Manure, Manufacture of, from Waste Leather and Phosphate of Lime. W. E. Rowlands, Seacombe, Chester. Eng. Pat. 17,926. July 30, 1897.

The leather is moistened, a "suitable ferment" added, and the material is piled in mass until it becomes disintegrated by fermentation. Five parts of fermented leather are then mixed with 8 parts of crude phosphate of lime in a mixing machine along with sufficient H_2SO_4 to decompose the phosphate.—J. T. W.

XVI.—SUGAR, STARCH, GUM, Etc.

Indeterminate Losses in Cane-Sugar Factories. H. Pellet. Bull. Assoc. Chim. 1897, **14**, 1159—1162.

According to the theory of Prinsen - Geerligs (this Journal, 1897, 688), that in ripe canes the whole of the reducing sugar consists of dextrose, the direct polarisation would indicate too much sugar by 0·50 to 0·60 per cent., and as by the analysis of molasses in the ordinary way less sugar is found than really exists, the double error would amount to very high indeterminate losses.

The author has made tests in various factories during the past four years, and finds that with Egyptian canes of the first and second year, frequent analyses being made during the whole period of manufacture, the direct polarisation is the same as the polarisation by inversion, or slightly less, but never higher. The lower or older, and therefore riper internodes, show both less reducing sugar and less levulose, but levulose is always present. The indeterminate losses under proper control reach a maximum of only 0·20 per cent.

It would be interesting to know if the canes worked in Java contain more dextrose in proportion to reducing sugar than the red canes of Egypt, and on what this difference depends.—L. J. de W.

Molasses, Incipient Carbonisation or Combustion of. J. T. Crawley. J. Amer. Chem. Soc. 1897, **19**, 538—542.

The molasses in question was boiled to what is known as "string proof," at a temperature of 71° to 74°, and run into an underground cistern from time to time, until the material was within six or eight inches of the top of the cistern, when the walls cracked, and the molasses began to leak out. It continued to leak for about one and a half months, during which time five or six feet was thus lost. That which remained seemed to be in a perfect condition. Five days later, however, it was found flowing over the sides of the cistern showing signs of incipient carbonisation. The temperature at the point where it flowed over was 92°, but in the cistern, and especially where the crack was located, this boiling seemed to be more violent. This flowing continued for about 12 hours, but the molasses continued boiling within the cistern until there was left only a few feet of charred mass. The fumes given off affected the eyes considerably, and coming into contact with the metal roof, formed a salt.

The following substances were detected in the sample of char, the percentage being given where it was estimated:—Organic acid, distilled, 3·8 per cent., calculated as acetic acid. Furfural and furfural-yielding substances, minute quantities; glucose-yielding substances, small quantities, and caramels large quantities. Reducing sugars, 11·20; nitrogen, 0·87; phosphoric acid, 0·30; potash, 2·27; insoluble in water, 38·20; and moisture, 15·00 per cent. The sample of salt from the metal roof was almost pure zinc acetate, with a little formate.

The author attributes the phenomenon to incipient carbonisation. It is known that if molasses of low grade be boiled at a high temperature and run into coolers, it is very apt to froth and run over the sides of the containers; moreover, successive runnings of hot molasses into the coolers will often result in a burning of the product. Although the cause of this is not positively known, the author is of opinion that in the majority of cases it is due to decomposition of the non-sugars.—D. B.

Sugar Content of Some Tropical Fruits, Determination of. H. C. Prinsen-Geerligs. Chem. Zeit. 1897, **21**, 719.

See under XXIII., *page* 939.

Beetroot Juice, Effect of Grease on the Filtration of Carbonated. R. Schiller. Bull. Assoc. Chim. 1897, **14**, 1178—1181.

An average of the working of several factories gave 58 litres of diffusion juice per 50 kilos. of beetroots, and a consumption of grease equal to 0·0026 per cent. Adhering to these proportions a series of tests were made using 50 kilos of roots in each case. The greases tried were palm nut grease, rape oil, Russian mineral oil, and Dalmenharst woolgrease. The differences in the rate of filtration were so small as to be absolutely insignificant.—L. J. de W.

Beetroot Juice, Flask for the Polarisation of. D. Sidersky. Bull. Assoc. Chim. 1897, **14**, 1170.

See under XXIII. *page* 935.

PATENTS.

Starch and Gluten, Improved Process and Apparatus for the Production of, out of Cerealia and Legumines. J. Keil, Halle, Germany. Eng. Pat. 17,444, July 24, 1897.

The cereal and leguminous flours are mixed with water as usual, to which about 0·2 per cent. of hydrated oxide of calcium is added, and kneaded in a receptacle provided with S-shaped paddles and stirring paddles inclining upwards towards the shaft, a plate being attached to the same at the top to prevent the dough rising above this height. The reservoir surrounding the paddle arrangement is provided with a delivery device.

A quantity of from 25 to 50 kilos. is worked through within 30 to 45 minutes, the paddles turning with increasing speed, after which time the mass forms a salve-like mixture. This substance is then centrifugalised, when the starch separates in an absolutely pure firm state (prime starch), and the gluten is paste-like and contains all the nutrient matter of the flour, its consistency rendering it suitable as an addition to human food.—L. J. de W.

Crystallisable Liquids [Sugar Juice]. Impts. in the Granulation of, and especially of the By-products of Sugar Manufacture. J. Grosse, Kiew, Russia. Eng. Pat. 23,318, Oct. 20, 1896.

The contents of the vacuum or granulating pan are heated, in the lower layers only, by a coil of piping through which steam or other heating agent is caused to pass, and a mechanical stirring device in the interior of the pan conveys the particles of the mass from the lower parts into the upper colder layers, effecting a continuous rapid evaporation, so that crystallisation is considerably accelerated.

According as finer or coarser crystals are desired, further quantities of the by-product are drawn into the apparatus in larger or smaller quantities. After the pan is filled to about seven-eighths of its capacity, heating is discontinued, but the mass is still further agitated in the vacuum until the requisite cooling and the final crystallisation are effected.—L. J. de W.

Starching Preparation, An Improved, and Process of Making the same. R. Ansorge, Halle-on-Saale, Germany. Eng. Pat. 586, Jan. 8, 1897.

The present invention allows of starch being directly employed for laundry purposes in a cold state without previous boiling.

The starch is mixed with a suitable amount of zinc-white, or like filling material, and then borax and stearin, stirred up in hot water, are poured into it, and the whole is stirred together, and the starch dried.

The proportions preferably employed are :—30 grms. of white oxide of zinc, 15 grms. of stearin, 300 grms. of borax, and 1 kilo. of starch. The filling may be omitted.

Manufacturers can advantageously use the still wet starch obtained in the course of manufacture without previously drying it. A modification consists in the use of wheat flour instead of, or in addition to, raw starch.

—L. J. de W.

XVII.—BREWING, WINES, SPIRITS, Etc.

Wort, The Influence of the Malt Husk on the Extract, Colour, and Taste of the. L. Freis. Wochenschr. für Brauerei, 1897, 502.

Rothenbuchler's method for mashing malt flour, the husk being previously separated, has long been known to be impracticable, owing to the difficulty of obtaining a bright wort; but this led the author to inquire as to what effect the husk produces in the mash. He investigated this in the laboratory, and found that a pale malt gave 17·7 per cent., and a dark malt 18·7 per cent. of husk. The husk yielded 13 per cent. of extract; and as the malt yielded 71 per cent. of extract, 2·4 per cent. of this was due to the husk. The husk wort possessed a large proportion of the colour of the malt, the wort from the malt flour being very pale. The husk wort is sweet and insipid; about 50 per cent. is fermentable.—A. L. S.

[*Beer.*] *Albuminoid Matter in two Brewings on the Wahl and the Windisch Systems.* M. Henius and G. Thevenot. La Bière, **5**, [7], 100—102.

The two brews were prepared from equal quantities of materials, but the initial mashing temperatures differed, that in the Wahl method being 41·25° C., whilst in the Windisch method it was 75° C. The resulting worts also differed, containing respectively: extract, 12°·06 and 11°·68 Ball.; maltose, 8·61 and 8·68 per cent.; and a maltose ratio of 1:0·40 and 1:0·70. The Wahl wort broke exceedingly well, and, after fermentation, contained 4·688 per cent. of actual residual extract, and 3·8 per cent. of alcohol, the actual attenuation being 61·1 per cent.; whilst the other broke badly, and was very turbid when cold; and the figures for the above data were respectively 6·254, 2·87, and 46·5 per cent.

On subjecting the beers to analysis and precipitating the coagulable nitrogenous matter, the peptones, and albumoses, it was found that the Wahl process yielded a larger quantity of each than the Windisch process, *viz.*, total N, 5·03 (4·08); true peptones, 0·76 (0·44); albumoses, 0·87 (0·84) per cent. of the extract. Hence the authors conclude that if the production of "head" be, in fact, due to bodies of such nature, the end will be more satisfactorily attained by means of a low initial mashing temperature than by the converse method.—C. S.

Albuminoid, Structureless, Present in Admixture with Bottom-Fermentation Yeast. [*Brewing.*] H. Will. Zeits. für d. ges. Brauwesen, **20**, [35], 447—449; [36], 459—462; [38], 485—488.

On thoroughly shaking up fresh bottom-fermentation yeast in a test tube with ether, a gelatinous mass is formed, which after standing (closed) for some days, separates into two layers: a lower stratum of pale yellow liquid and an upper grey-brown vitreous layer. The difference is more sharply accentuated if the ether has been previously stained with alkannin, the gelatinous layer in that case, absorbing the colouring matter. When this portion is examined under the microscope, it is found to contain, in addition to yeast cells and fragments of grains, hops, &c., a network of gelatinous matter binding the whole together and also enclosing drops of ether, many of the latter being surrounded by an integument which shrivels up as the ether evaporates. This skin answers to all the reactions characteristic of albumin, and is rendered particularly conspicuous when treated with Millon's reagent or by the Raspail method (concentrated sugar solution and sulphuric acid).

On the other hand, the gelatinous matrix is not stained by iodine or by aniline dyes, but, when treated with water, it swells up and dissolves, liberating the yeast and other embedded substances; sulphuric acid causes it to thicken, as also Millon's reagent does. Fehling's solution gives no precipitate. From tests performed on the washings from several yeasts, this gelatinous matrix is believed to consist mainly of gum.

The capacity of yeast for permanently absorbing ether was also examined by the author. He finds that it varies in different yeasts, and also in the different strata—*e.g.*, "core" yeast and superficial layer—of one and the same yeast, the average absorption per 6 c.c. of yeast of normal constitution ranging from 10 to 12 c.c. of ether for the "core" yeast, and from 13 to 18 c.c. for the upper layers. In proportion as the yeast is freed, by washing, from the substances assisting in the formation of bubbles of ether, the capacity of permanent absorption is reduced, but is restored by the addition of mucedin and peptone, these bodies gelatinising under the ether treatment. The amount of water present is a measure of the distension of the albuminoids, which factor in turn governs the absorption of ether.

When the washings from the yeast were united and shaken up with ether, a supernatant stratum of thick emulsion, containing bubbles of ether, separated out. The amount of this emulsion in the individual washings progressively decreased, and at about the tenth washing ceased to form.—C. S.

Red Wines, Fermentation of Deeply Coloured; Influence of Colouring Matters on the. P. Carles and G. Nivière. Comptes Rend. 1897, **125**, [12], 452—453.

The authors arrive at the following conclusions:—(1.) The incomplete transformation of sugar in highly coloured worts is due to the colouring matter and not to the acidity, since decoctions of elder, whether acidified or not, have given the same result. (2.) The colouring matter, allied to tannin, acts as an antiseptic on the micro-organisms which produce fermentation.

Tartaric acid, added to red wines, has no influence on the fermentation, or, if it acts at all, it is only indirectly in preventing the precipitation of the colouring matter, which itself acts on the ferments.—J. S.

Lactic Fermentation, Contribution to the Study of. H. Pottevin. La Bière, **5**, [9], 136—140.

The object of the investigations undertaken by the author, was to ascertain whether any close optical relation exists between a sugar and the lactic acid resulting from its fermentation. The ferment employed was obtained, by dilution, from the juice of onions self-inoculated in air. It grows best at 35° C. and in a medium of onion juice, to which 0·5 per cent. of peptone has been added.

The acid obtained from the fermentation of *lactose* was optically *inactive* so long as the proportion of peptone taken remained above 1 per cent., but when this ratio was reduced, a mixture of inactive and sarcolactic acids resulted, without the ferment losing its activity. *Saccharose* yielded *inactive* lactic acid, whilst *maltose* yielded the *inactive* acid in presence of 1 per cent. of peptone, but the *dextro-rotatory* acid with 0·6 per cent. of peptone. The results obtained with *glucose*, *invert sugar*, *galactose*, and *mannose* were on similar lines, whereas *mannite*, *dulcite*, and *glycerin* (which

offer greater resistance to the ferment) all furnished *active* (dextro-) acid. With these latter bodies, fermentation proceeded very slowly, and was not hastened by an increase of peptone. Calcium malate was attacked by the ferment, but only alcohol and volatile acids resulted. Neither lactate, succinate, nor tartrate of calcium is, however, acted upon.

The author concludes that a lactic ferment may, without prejudice to its qualities as a true lactic ferment, produce active lactic acid equivalent to over 80 per cent. of the sugar eliminated. Also that the optical properties of the acid are independent of the composition and properties of the original carbohydrate, but that a number of factors come into play, the diminution of the nitrogenous nutriment, an increase in temperature, the addition of antiseptics and the greater resistance of the hydrocarbons, all tending to the production of dextro-rotatory acid.—C. S.

Mead, The Natural Ferments of. E. Kayser and E. Boullanger. La Bière, **5**, [8], 113—121; [9], 129—135.

The conclusions arrived at by the authors from their extensive researches, may be summarised as follows:—

The honey worts should be prepared in such a manner as not to indicate a greater strength than 24—25 per cent. of sugar by the Guyot glucometer (13° B.) for dry meads, or 26—27 per cent. Guyot (14°—14·5° B.) for sweet meads. Filtration is always advisable especially when working with selected yeasts; and is essential to the production of good mead in the case of waxy honey.

To ensure the normal and rapid progress of fermentation, diminish the risk of infection by injurious organisms, and produce a liquor of excellent quality and endowed with good keeping properties, the addition of one of the following yeast foods is necessary:—A.: Maltopeptone 1·5 c.c., potassium bitartrate 1 grm. per litre; B.: Maltopeptone 1·5 c.c., potassium bitartrate 1·5 grm., ammonium phosphate 1·0 grm. per litre; C.: Spongy peptone 0·12 grm., potassium bitartrate 1·5, ammonium phosphate 1·5 grm. per litre; X.: Calcium biphosphate 1 grm., ammonium phosphate 2·0, potassium bitartrate 2·0, magnesium sulphate 0·1 grm. per litre. The nutritive mixture is dissolved in a few litres of the wort and then well mixed up with the bulk.

In starting the fermentation, it is highly desirable to prepare the yeast by pitching 1 or 2 litres of sterilised wort, containing nutritive material, and leave it to ferment for a couple of days, after which it is transferred to the main body of wort. This procedure is of particular service (in cases where no yeast food is used) for developing a vigorous pitching yeast.

Selected wine yeasts will give excellent results and improve the quality of the product, their behaviour being superior to that of other yeasts when the fermentation temperature is high. Pollen yeast may be recommended, provided it has been subjected to the preparatory treatment named above.

Provided the temperature be favourable, the brisk primary fermentation should not last more than a month. Rousing is desirable when the fermentation slackens before the proper quantity of sugar is consumed.

Secondary fermentation is a protracted and delicate operation in the case of meads, and should continue for several months in order that a good product may result. Dry meads should be transferred to a cellar when primary fermentation is completed, and there left for the yeast to finish its work slowly and to clarify. Sweet hydromels, on the other hand, should be racked to remove the greater part of the yeast, and then fined. This last operation will be easier in proportion as the fermentation has been brisk and regular.

It should take about six months to produce a good saleable article; but, as already stated, primary fermentation ought to be complete in one month, at the end of which time dry meads should indicate not more than 2—3 per cent. of sugar, and sweet meads a maximum of 4—6 per cent. by the Guyot instrument. The liquor must not be bottled until perfectly clear.—C. S.

Methyl Alcohol, Formaldehyde, Formic Acid; Determination of Small Quantities of. Nicloux. Bull. Soc. Chim. 1897, **17**, 839.

See under XXIII., *page* 941.

Plumbates, Alkaline Earth; Manufacture and Properties of. G. Kassner. Rev. Chem. Ind. 1897, **8**, 227.

See under VII., *page* 913.

PATENTS.

Fermented Liquors, The Manufacture of; Impts. in. J. Schneiole, New York, U.S.A. Eng. Pat. 25,852, Nov. 17, 1896.

This invention relates to fermented liquors, such as beer, cider, &c., but has special reference to the manufacture of lager beer. It is customary to transfer lager beer, after fermentation, from the fermenting tuns at a temperature of 3°·1—4°·3 C. to the storage casks, where, the cellar being maintained at about 1°·25 C., it gradually cools down and slowly deposits (six weeks or more) those albuminoid constituents which are coagulable at a low temperature. The improvement consists in cooling the beer to 0° C. before transferring it to store-casks. By this means, it is claimed, the albuminoids are immediately thrown out of solution and caused to settle in a comparatively short space of time. In its application to cider, &c., the process is similar, *i.e.*, the liquor is stored at a low temperature until brilliant.—H. T. P.

Yeast, The Manufacture of; Impts. in and relating to. (Apparatus.) H. H. Lake, London. From L. S. Langville and H. Tauszky, New York, U.S.A. Eng. Pat. 18,112, Aug. 3, 1897.

A process and apparatus for separating yeast from fermented liquids, in the manufacture of pressed yeast. The apparatus consists of a number of shallow troughs, connected in series and communicating by valves or slides. The liquid containing yeast in suspension is allowed to flow through the troughs, and, being cooled during its passage by a refrigerating system arranged beneath, and in contact with the troughs, rapidly deposits its yeast. If desired, a separation of the yeast into two or more grades ("strong" and "weak") may be secured, by reason of the fact that the strong cells are deposited at an earlier stage than the weak ones.—H. T. P.

XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

(*A*.)—FOODS.

Fermentation, Quick versus Slow. [Bread Baking.] W. Jago. National Assoc. Rev., Aug. 1897, 187—493.

The conditions which accelerate fermentation are—strong yeast, soft flour, use of potatoes and saccharin extract (which act as stimulants), slackness of the dough (water), aeration, and primarily a high fermentation temperature. Fermentation is retarded by the reverse of these conditions and also by the use of large quantities of salt. In order to determine by which method the souring of the bread was best held in check, the author made experiments in which 50 grms. (1·8 oz.) of flour and 200 grms. (7 oz.) of water were fermented with 2·5 grms. (say $\frac{1}{10}$ oz.) of distillers' yeast and 10 grms. (say $\frac{4}{10}$ oz.) of brewers' yeast respectively. The fermentation was continued until about 350 c.c. of gas had been evolved, and the acid produced reckoned as grms. of lactic acid.

——	Time Taken.	Acid produced.
	Hours.	
Distillers' yeast, 75° F.	10½	0·219
" " 95° F.	3½	0·115
Brewers' " 75° F.	11	0·196
" " 95° F.	6	0·214

Repeats on Second Day.

Distillers' yeast. 75° F.	11½	0·225
" " 95° F.	4¾	0·180
Brewers' " 75° F.	11	0·522
" " 95° F.	5¾	0·513

In the author's opinion these results prove that, for the same amount of fermentation, the amount of acidity produced at a comparatively high temperature is "at least not higher" than that produced at a much lower temperature; and, from a similar series of laboratory experiments, he also draws conclusions in favour of the quick fermentation process.

Further experiments on a larger scale in the bakery are described, which seem to prove that from 3½ to 3¾ hours at 91° F. is the most suitable time for a quick fermentation. When the working time is shortened to three hours, the results are unsatisfactory, even when the temperature and amount of yeast are increased. With a quick fermentation process, less plant is required, and the dough does not remain so long exposed to atmospheric conditions, and while more yeast is required, there is an increased yield of bread. On the other hand, the sponges and doughs must be taken at exactly the right time, or the whole batch may be spoiled.—C. A. M.

Earth-Nut, Detection of, in Chocolate. Bilteryst. Bull. de l'Assoc. belge des Chim., March 1897, 447; through J. Pharm. Chim. 1897, **6**, [1], 29.

See under XXIII., *page* 937.

PATENTS.

Aeration and Preservation of Milk, and Apparatus therefor; Impts. in the. R. G. Nash, Dublin. Eng. Pat. 14,209, June 26, 1896.

The milk is heated to a temperature of 156° to 160° F., whereby it is sterilised, and then cooled. When thus treated, it is then charged with sterilised gas, decanted, and heated in a closed vessel to 150°–212° F. On cooling, a sterilised, aerated beverage is obtained. The gas is sterilised by passing it through a coil maintained at a red heat, thence through a coil submerged in cold water, and then through sterilised water or other purifying liquid. An improved method of flavouring aerated liquids is claimed, consisting in adding the flavouring material to the washing water employed in sterilising the gas, whereby the flavour is imparted to the gas. To prevent the milk curdling, an alkali and sugar are added. A creamy "head" on the milk is obtained by the addition of a suitable essence. —W. P. S.

Horse and Cattle Food, A New Manufacture of. W. C. Forsyth, Leytonstone, Essex. Eng. Pat. 25,344, Nov. 11, 1896.

The claim is for a food consisting of a mixture of linseed, oats, locust beans, and maize, together with small quantities of spices and salt.—W. P. S.

The Preservation of Organic Matters [Foodstuffs], Impts. in. J. J. Hazard, London. Eng. Pat. 899, Jan. 12, 1897.

The process claimed is one for preserving organic substances, such as carcases of beef or mutton, poultry, game, fruit, furs, eggs, &c., by subjecting them to the action of dry carbonic acid under pressure, and afterwards storing them in hermetically closed chambers, the atmosphere of which is kept dry by means of a suitable drying agent and agitation. It is usually found sufficient to expose the body in the pressure chamber for 20 to 40 minutes at a pressure of from 2 to 5 atmospheres.—W. P. S.

Milk, Impts. in Treating. A. Bernstein, Boston, U.S.A. Eng. Pat. 14,336, June 12, 1897.

The object of this invention is to prepare an edible product from skimmed or separated milk. For this purpose, a quantity of wheaten or other flour is added to the milk, which is then coagulated by the addition of rennet. After draining off the whey, the curd is obtained as a soft mass, which may be dried to a powder, and mixed with flour for baking purposes.—W. P. S.

Edible and Soluble Tablets, and the Method of Producing the same, Impts. in. H. Horlick, Racine, Wisconsin, U.S.A. Eng. Pat. 16,088, July 6, 1897.

The tablets consist of pasteurised milk combined with the extract of crushed barley malt and the extract of pulverised cereals, prepared as described below, and pressed into a dry solid form.

Equal quantities of coarsely broken or crushed barley malt and wheaten or other flour are macerated in cold water. The temperature is then raised to about 160°–165° F., and the mash kept in agitation until it becomes thin enough to filter. After filtering, an equal quantity of cows' milk is added to and thoroughly mixed with the extract. The resulting product is pasteurised, and then evaporated *in vacuo* until it is practically dry. It is then removed and pressed into moulds. Before the mixture is quite dry, 5 to 15 per cent. of either cocoa, chocolate, or beef tea may be added, and the drying continued as before. —W. P. S.

Preservation of Lobsters and other Crustacea packed in Metal Cans or other Receptacles, Impts. relating to the. L. Wurzburg, London. Eng. Pat. 17,914, July 30, 1897.

The method or process claimed, consists in dipping the crustacea-meat, and the metal cans or other receptacles, into a solution of boric acid. Each pint of preserving solution should contain two ounces of boric acid. After dipping, the tins are sealed up as soon as possible and put through the usual bath.—W. P. S.

(*B.*)—SANITATION; WATER PURIFICATION.

Acetylene, Poisonous Action of. U. Mosso and F. Ottolenghi. Ann. di Chim. e di Farmacol. **25**, 163.

The authors have studied the poisonous action of mixtures of acetylene and air. Small amounts of acetylene are dangerous to animals. Inspiration of ½ litre of the pure gas poisons dogs in a few seconds, and powerful artificial respiration is need to restore them. A 20 per cent. mixture of acetylene and air kills dogs in an hour or less; the disturbance to the system is so great in these slow actions, that the dogs often die after they have been brought into fresh air and have apparently recovered. Large amounts of the gas affect the respiration; smaller quantities produce irritation, followed by a period of paralysis and weakening of the heart and lungs.—J. T. D.

PATENT.

Putrefaction of Bodies [Corpses]; Improved Method of and Apparatus for preventing the; also applicable for Disinfecting Mattresses, Articles of Dress, and the like. F. de Rechter and G. de Rechter, Brussels. Eng. Pat. 15,368, June 26, 1897.

This specification describes an arrangement of apparatus for disinfecting by means of formalin. The apparatus consists of a large disinfecting, and a smaller generating chamber, both of which can be suitably heated. The formalin solution is delivered from a drop-feeding cup on to the surface of stretched plaited lamp wick or other absorbent material, contained in the generating chamber, where it evaporates and by means of top and bottom flues connecting the two chambers, one of which contains a revolving fan, the vapour is caused to circulate. An air-pump is provided, by means of which the atmosphere of the apparatus can be rarefied, and the disinfecting chamber contains a perforated tube filled with calcium chloride for drying the air. When disinfection is judged to be complete, the connecting flues are closed by means of shutters; and the calcium chloride tube is replaced by another tube containing absorbent material saturated with a neutralising liquid, such as ammonia, which absorbs the residual aldehyde vapour and renders the atmosphere respirable when the chamber is opened. —L. A.

(C.)—DISINFECTANTS.

Alcohol as a Disinfecting Agent. F. Epstein. Proc. Inst. Civil Eng. 1896-97, **129**, [3], 31; Zeits. für Hyg. 1897, 1.

The author found that absolute alcohol had no disinfecting action, but that its solutions were more or less active, according to the degree of concentration. 50 per cent. solutions gave the best results when alcohol was used alone; the disinfecting action was diminished when more concentrated or more dilute solutions were employed. Antiseptics which, when dissolved in water, were more or less active, lost their disinfecting action entirely when dissolved in highly concentrated alcohol; but, on the other hand, solutions of corrosive sublimate, carbolic acid, "lysol," and thymol in 50 per cent. alcohol. exerted greater powers than aqueous solutions of the same degree of concentration.—A. S.

PATENTS.

Disinfecting Purposes, Impts. in and relating to Materials and Apparatus for. R. Goehde and H. Oppermann, Berlin. Eng. Pat. 20,904, Sept. 22, 1896.

Formic aldehyde and menthol are dissolved in methyl alcohol, and the solution is placed in a dropping bottle, which is supported in an inverted position over a concave disc of asbestos fixed at the top of a sheet-iron chimney surrounding the flame of a lamp. The drops of solution falling upon the heated asbestos are rapidly evaporated and diffused throughout the surrounding air, which is thereby disinfected.

The solution is preferably saturated with formalin, but the proportion of menthol may vary from 1 to 50 per cent., the larger proportion being used in order to neutralise the action of the aldehyde on the mucous membrane, when the atmosphere is being respired by persons during the process of disinfection.—L. A.

Disinfecting, Impts. in, and in Apparatus Therefor. A. Zimmermann, London. From The Chemische Fabrik auf Actien vormals E. Schering, Berlin. Eng. Pat. 26,667, Nov. 24, 1896.

Dry paraformaldehyde in the form of pastils is placed in a metallic cup, which fits in the upper part of a chamber surrounding the flame of a spirit lamp, so constructed that the products of combustion are made to pass through orifices in the upper sides of the cup, and become mixed with the vapour of the aldehyde, which is in this manner largely diluted and mechanically diffused into the atmosphere of the place to be disinfected. The water vapour contained in the combustion gases is essential for the proper development of the disinfecting action of the aldehyde, and the rate of evaporation is such that 1 grm. of paraformaldehyde becomes diluted with 9 grms. of carbon dioxide, and about $3\frac{1}{2}$ grms. of steam.

Instead of paraformaldehyde, asbestos saturated with an aqueous solution of ordinary formaldehyde may be employed. —L. A.

Bursting of Bottles and other Receptacles while Substances enclosed therein are being Sterilised, Preventing the; Impts. in Methods of, and in Apparatus therefor. A. L. Rockstrom, Norberg, Sweden. Eng. Pat. 16,952, July 17, 1897.

The principle of the method is to maintain, during the whole time of heating and cooling, a pressure on the outside of the vessels equal to or slightly exceeding the pressure in the interior. This is accomplished by placing the vessels in a closed chamber, which is filled with a fluid, and by forcing into or withdrawing from the said chamber, water or a gas during the heating or cooling operation, an external pressure is maintained equal to or exceeding that which simultaneously exists in the interior of the vessels.—L. A.

Compounds of Formic Aldehyde for Preserving, Disinfecting, and Therapeutic Purposes; Process for the Preparation of. H. Oppermann, Berlin. Eng. Pat. 18,250, Aug. 5, 1897.

The patentee has found that by the agency of ammonia, formic aldehyde may be coupled with any substance capable of combining with or dissolving in ammonia solution. Crystalline compounds are thus produced (similar to the known compounds of aldehydes with bisulphites) in which formalin is united with substances having disinfecting, preserving, or specifically therapeutic properties. The bodies thus formed are fairly stable, easily soluble in water, and are powerful reagents. Thus, the compound of formalin with boric acid is said to be more efficacious than mercuric chloride; and the compound with phenol, when evaporated in small quantity, will disinfect a large space.

For the preparation of these bodies, the ordinary 40 per cent. solution of formalin, together with the required other substance, is dissolved in the requisite quantity of ammonia solution of 30 per cent. strength, with constant stirring and cooling. The compound either crystallises out, or is obtained by evaporating the liquid at a low temperature *in vacuo*. Substances mentioned are tartrates, boric and salicylic acids, and phenol; or triple compounds may be formed in which formalin is united with phenol and menthol, or with boric acid and tannin.—L. A.

XIX.—PAPER, PASTEBOARD, Etc.

Cellulose Carbohydrates, Quantitative Separation of, in Vegetable Substances. W. Hoffmeister. Landw. Versuchsstat. 1897, **48**, 401; through Chem. Zeit. 1897, **21**, 198.

See under XXIII., *page* 940.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

Ozone, Production of. Otto. Proc. Inst. Civil Eng. 1896-97, **129**, [3], 72—73. Mémoires et Comptes rend. des travaux de la Soc. des Ingén. Civils de France. March, 1897, 310.

The author considers that the low theoretical efficiency in output (rather less than 2 per cent.) of the Siemens and Halske and Andréoli machines for producing ozone, is due to the employment of too high potential and a bad choice of dielectric. He has designed machines for the production of ozone, which, with a periodicity of 80 per second, give a theoretical efficiency of 15 per cent.; the output was found to be greater as the periodicity of the working current increased, and also as the velocity of the current of gas through the machine was increased. The machines are of four types, but the general arrangement in all is one of plate conductors, protected either on one or both sides with dielectric, arranged alternately of opposite sign and with an air-space between, through which the gas to be treated is passed, and across which the electric discharge takes place. In some cases the gas is drawn in at the edges of the plates, and delivered through a central aperture on one side; and in others it is drawn in and delivered at central apertures, one on each side.—A. S.

Sulphur Precipitatum (American). T. D. Reed, M.D. Amer. Pharm. Assoc. 1897; through Pharm. J. 1897, **59**, [1421], 265.

An examination of trade samples of precipitated sulphur, obtained in Canada and in the United States, show that more than half (29 out of 55) contained calcium sulphate. —J. O. B.

Selenium in Commercial Sulphur. T. D. Reed. Amer. Pharm. Assoc. 1897; through Pharm. J. 1897, **59**, 261.

See under XXIII., *page* 935.

Turpentine, Action of Sulphuric Acid on Levo-rotatory. G. Bourchardat and J. Lafont. J. Pharm. Chim. 1897, **6**, 193—196.

Levo-rotatory French essence of turpentine was treated with 10 per cent. of sulphuric acid and the product heated at 150° with alcoholic potash in excess. On addition of water, a light oil was obtained, containing unaltered turpentine, camphene, terpilenes, and their solid and liquid polymers, together with a small quantity of levo-rotatory

borneol and its dextro-rotatory isomer melting about 50°. The aqueous solution contains two potassium salts of the formula $C_{10}H_{16}KHSO_4$: these were separated by fractional crystallisation from alcohol. The less soluble is a salt of the acid sulphuric ether of levo-rotatory borneol or of an isomer; it is itself levo-rotatory. The more soluble salt is dextro-rotatory, it is derived from dextro-rotatory fenchol.
—A. C. W.

Iodoform, Electrolytic Preparation of. K. Elbs and A. Herz. Zeits. f. Elektrochem. **4**, [4], 1897, 113—118.

An electrolytic process for preparing iodoform, bromoform, and chloroform from the corresponding halogen compounds of the alkalis and alkaline earths in presence of alcohol, aldehyde, or acetone, has been known and patented since 1884 (E. Schering, Berlin). The authors have studied more closely the nature of the electrolytic processes and the conditions which give a good yield. The following equation represents the reaction of iodine upon warm alcohol in the presence of an alkali or alkaline carbonate:—

$$CH_3.CH_2OH + 10I + H_2O = CHI_3 + CO_2 + 7HI \quad (1.)$$

The reaction can be conveniently carried out by passing an electric current through an aqueous solution of potassium iodide and soda mixed with alcohol—the current setting free from the potassium iodide the ions iodine and potassium. The hydriodic acid (or potassium or sodium iodide) formed by a secondary reaction is again decomposed by the current; thus the iodine not combined with carbon is for the most part regenerated continuously, and only a small part of it is converted at the anode into potassium iodate. Conditions might exist under which the oxidation might stop short of producing carbon dioxide, and might instead only produce formic acid—

$$CH_3.CH_2OH + 8I + H_2O = CHI_3 + HCOOH + 5HI \quad (2.)$$

But in the electrolysis carried out by the authors, no trace of formic acid was ever observed. The theoretical yield is therefore calculated according to equation (1), and amounts to 1·468 grms. of CHI_3 per ampère-hour. The actual yields are expressed as percentages of this. Five tables are given illustrating the influence of various conditions on the yield. Table A illustrates the effect of temperature: the yield is best when the temperature is between 60° and 70° C. Table B shows (other things being equal) that when the proportion of soda present is increased, the yield of iodoform slightly falls off, while there is a marked increase in the amount of iodic acid formed. When the soda is kept constant and the amount of potassium iodide is increased, the yield of iodoform increases and the amount of iodic acid decreases (Table C). Similar effects are produced when the soda and potassium iodide are kept constant and the amount of alcohol is increased. Table E illustrates the effect of varying current-density. The results show that the current-density should not exceed 1 ampère per square decimetre. 60° is a suitable temperature, and the electrolyte should be made up in the proportion of 5 or 6 grms. of anhydrous sodium carbonate, 10 grms. of potassium iodide, 20 c.c. of alcohol, and 100 c.c. of water. The precipitated iodoform should be removed hourly, and the electrolyte made up to the original composition by adding soda, potassium iodide, and alcohol. Iodic acid is only formed slowly, and is of secondary importance; it takes a long time before it accumulates sufficiently to interfere with the main reaction. The yield is excellent. In the third ampère-hour the yield amounted in one experiment to 97·3 per cent., and in another to 97·7 per cent. of the theoretical. The iodoform was crystalline, only very slightly coloured (yellow), and perfectly pure.—D. E. J.

Orthoform: A New Local Anæsthetic. A. Einhorn and R. Heinz. Munich Med. Woch. 1897, **14**, 931; through Pharm. J. 1897, **59**, [1422], 277.

This new synthetic body, which possesses marked anæsthetic properties, and is at the same time relatively nontoxic, and also antiseptic, promises to find a place as a therapeutic agent. Experiments of previous workers show that none of the products obtained by splitting up the cocaine molecule are capable of producing anæsthesia, and that this action is possessed only by those compounds, analogous to cocaine, in which the ecgonine esters are combined with certain acid radicles. In this respect, the group $COOHCH_3$ appears to play a very important part. This anæsthetic property was found to be possessed by all amido-oxy esters of the aromatic series, but the body from which these are all derivable — the amido-oxybenzoic ester—has this action in a still greater degree. After trial with a large number of these bodies, the *p*-amido-*m*-oxybenzoic methyl ester was found to give the most satisfactory results, and this is therefore introduced for therapeutic use under the name of orthoform.

It occurs as a white bulky crystalline powder, melting at 120° C., non-hygroscopic, and but slightly soluble in water, so that it is only slowly absorbed, and therefore produces a persistent anæsthesia. The hydrochloride is readily soluble in water, but as the solution has an acid reaction, it is not always applicable for hypodermic use.—J. O. B.

Cinchonine, The Transformations of, by the Action of Acids. Z. H. Skraup. Monatsh. für Chem. 1897, **18**, 411.

By the action of hydrochloric acid on cinchonine hydrochloride, 25—30 per cent. of the base is converted into an addition compound, the remainder into the isomeric bases—iso- and pseudocinchonine. If this addition compound be the intermediate stage in the transformation, according to the hypothesis of Wislicenus, if it be subjected to the conditions which produced it, then there will be formed the same proportions of the isomeric bases as above. This was not found to be the case. Hence the transformations do not take place through the addition compound.

Attempts to convert cinchotine into isomeric bases by the action of hydrochloric or sulphuric acid were unsuccessful. The sulphates obtained from the fractionally crystallised products had the same solubility and the bases the same rotation, though the salts differed in appearance. Now cinchotine probably differs from cinchonine only in containing an ethyl- in place of a vinyl-group ($-CH:CH_2$). Thus the transformations of cinchonine must be referred to changes in the vinyl-group.—A. C. W.

Retamine: A New Alkaloid. Battandier and Malosse. Journ. Pharm. Chim. 1897, **6**, (6), 211.

From the young branches and bark of *Retama sphærocarpa*, the authors have isolated an alkaloid, retamine, having the composition $C_{15}H_{26}N_2O$, which appears to be an oxysparteine. From a saturated solution in petroleum ether it crystallises in long needles; from alcohol, on cooling the saturated solution, it separates in lamellæ, by spontaneous evaporation from the same solvent in fine rectangular tablets. The taste is bitter, and the alkaloid is without marked physiological action. The crystals melt at 162° C., with alteration, and at a higher temperature, decompose, giving a crystalline sublimate, and evolving a pyridine-like odour. Retamine acts as a powerful base, colouring phenolphthalein, displacing ammonia, and precipitating the salts of iron and copper. It also acts as a powerful reducing agent; gold chloride and phosphomolybdic acid are immediately reduced by it, silver salts and potassium ferricyanide more slowly; mercuric chloride is transformed to calomel by its action. It gives all the alkaloidal reactions, but does not precipitate with platinum perchloride. Its salts crystallise very easily, with the exception of the nitrate. In absolute alcohol at 17° C. its solubility is 2·462 in 100. The specific rotation is $(\alpha)_D = 43°·1$.—J. O. B.

Cinnamic Acid from Storax and Benzoic Acid from Benzoin, Extraction of. E. Claassen. Pharm. Rev. 1897, **15**, 136; through Chem. Zeit. 1897, **21**, 199.

Storax is boiled for some time in a copper vessel with an excess of sodium carbonate solution. When cool, hydrochloric acid is added until the reaction is only faintly alkaline. The mixture is again heated to boiling and filtered, and the residue washed with water until exhausted. The mixed filtrates are precipitated with an excess of hydrochloric acid. The precipitate is collected, washed

with cold water, dried and extracted with hot benzene. On cooling, the greater part of the cinnamic acid separates out. The benzene is distilled off and used for further extractions as long as anything is dissolved. Finally, the benzene solution of the acid is shaken out with dilute sodium carbonate solution, and the acid re-precipitated from this with hydrochloric acid, washed with cold water on a filter, and dried. The acid thus obtained from the first extraction is brilliantly white, but subsequently is coloured yellowish; when this occurs, the coloured acid is redissolved in benzene and again extracted, and precipitated as above. The same process is applicable to certain varieties of benzoin, for the extraction of benzoic acid, and also for the separation of benzoic and cinnamic acid from Tolu balsam.—J. O. B.

Parathyroid Glandules, Presence of Iodine in. F. Gley. Comptes rend. 1897, **135**, 313.

The small parathyroid glandules which are found accompanying the larger true thyroid glands, are stated to be relatively richer in iodine in the case of dogs and rabbits than the latter bodies. It is evident therefore that, if the presence of a secretion containing iodine is to be considered distinctive of the thyroidal class of organs, the smaller parathyroid glands must be included with them. And further, that if this secretion be the active principle of the gland, that the parathyroid glandules must be, at least as potent, weight for weight, as the true thyroid.—J. O. B.

Bisobol Myrrh, Composition of and Distinction from Official Myrrh. Tucholka. Arch de Pharm. 1897, 290.

See under XXIII., *page* 938.

Eucalyptol [Tests for Purity of]. A. Schamelhout. Ann. Pharm. 1897, **3**, 257.

See under XXIII., *page* 937.

Peppermint Oil, Determination of Menthol in. C. Kleber. Pharm. Rev. 1897, **15**, 135; through Chem. Zeit. 1897, **21**, 198.

See under XXIII., *page* 940.

PATENTS.

Terebenthene Monohydrochloride, Impts. in the Manufacture and Application of. J. G. McIntosh, London. Eng. Pat. 21,031, Sept. 22, 1896.

In the process claimed, the mixture of solid and liquid hydrochlorides obtained by the saturation of turpentine with hydrochloric acid gas, is distilled with the addition of caustic alkali. The combined distillates are filtered and cooled to separate the solid from the liquid hydrochloride. The use of either of the hydrochlorides, or a mixture, as a substitute for natural camphor in the manufacture of celluloid, soaps, antiseptics, &c., is also claimed.—A. C. W.

Pharmaceutical Compound [Codeïne], Impts. in the Production of. Farbenfabriken vormals F. Bayer and Co. Elberfeld, Germany. Eng. Pat. 24,799, Nov. 5, 1896.

In the methylation of morphine by means of methyl iodide or methylsulphuric acid, considerable quantities of the alkaloid remain unchanged. The patentees claim the production of codeïne from morphine and diazomethane or such substances as are capable of yielding diazomethane, with or without the presence of alkalis, and they also claim the use of nitrosomethylurethane as the source of diazomethane in the process first mentioned.—A. C. W.

Piperazine Salicylate, Manufacture of. La Société Chimique des Usines du Rhône, Lyons, France. Eng. Pat. 25,905, Nov. 17, 1896.

Piperazine salicylate, $C_4H_{10}N_2[C_6H_4(OH)COOH]_2$, is claimed as a new product, and the processes for its manufacture, which consist in mixing boiling concentrated aqueous, alcoholic, or ethereal solutions of 1 mol. of base with 2 mols. of acid, or in melting the same ingredients together and recrystallising from one of the above solvents.—A.C.W.

Nucleins, An Improved Preparation of, and Process for Making it. J. Carnrick, New York, U.S.A. Eng. Pat. 17,610, July 27, 1897.

The process claimed, consists "in dissecting the lymphoid tissues containing the largest percentage of nuclein from the muscular and other coarse tissues, drying the lymphoid tissues at a comparatively low temperature, not exceeding 130° F., then reducing the material to a powder, and separating the nuclein cellular matter from the scaly fibrous matter by sifting or other means. The nuclein cellular powder is then treated with a solution of a suitable gum, as benzoin, for protecting and preserving it." The product is also claimed as a new article of manufacture.—A. C. W.

Acetone, Halogen Derivatives of; Impts in the Process for Preparing. L. Lederer, Munich, Germany. Eng. Pat. 17,692, July 28, 1897.

In the process claimed, iodine, ready formed or nascent, is made to react with acetonedicarboxylic acid, in the presence of substances which combine with or decompose hydriodic acid. The chlorine and bromine compounds are similarly produced with or without the presence of some substance to combine with the free acid produced. By using the proper proportions of iodine, per-, penta- or tetra-iodoacetone may be made.—A. C. W.

XXI.—PHOTOGRAPHY.

PATENT.

Photographic Paper, An Improved Sensitised. A. Schwarz, Gross Lichterfelde, Germany. Eng. Pat. 26,234, Nov. 20, 1896.

A "bromide" paper, prepared by coating transparent paper—instead of opaque paper as heretofore—with silver bromide.—H. T. P.

XXII.—EXPLOSIVES, MATCHES, Etc.

Nitronaphthalene, the Commercial Preparation of. W. H. Krug and J. E. Blomen. J. Amer. Chem. Soc. 1897, **19**, 532—538.

The manufacture of nitronaphthalenes has of recent years acquired considerable importance, owing to the fact that their presence in nitroglycerin renders the latter practically non-sensitive to concussion, whilst even a small admixture prevents dynamite from freezing. Nitronaphthalene is also said to possess the property of rendering dynamite "fumeless," but this has not yet been definitely decided. It considerably increases the solvent action of nitroglycerin on nitrocellulose.

When the nitronaphthalene is used in conjunction with nitroglycerin, its melting point is of minor importance, but it is desirable to have as high a degree of nitration as possible, since this will retard the momentum of the explosion to a less extent. When used with other explosives, such as picric acid, the melting point must obviously be below the point of decomposition of the explosive agent. When it is desired to use a higher nitronaphthalene in this connection, it may be accomplished by adding a small amount of mononitronaphthalene, as the latter acts as a solvent of the higher nitro-derivatives, and they together will dissolve picric acid, nitrocellulose, and the like.

The nitro-derivatives of naphthalene are generally obtained either by the action of nitric acid alone or mixtures of nitric and sulphuric acids on naphthalene, or for the highest nitro-derivatives, by renitration of the lower nitro compounds. The author has made a number of experiments with a view of ascertaining the practicability of the proposition to prepare the higher derivatives by the nitration of naphthalenesulphonic acid, similarly to the preparation of nitrophenols from phenolsulphonic acid.

α-Naphthalenesulphonic acid was prepared by heating naphthalene to 100° for about eight hours, with one, two, and three times the amount of sulphuric acid required by theory. A fourth preparation was made, using a large excess of sulphuric acid. A quantity of each of these acids, corresponding to 10 grms. of naphthalene, was treated in one series with 40 grms. of nitric acid of 36° B., and in

another series with 30 grms. of the same acid. The nitrations were effected on a water-bath and were considered complete some time after the red fumes ceased to come off. The product was melted under water and washed in this manner until free from acid. It was then carefully dried and weighed. The results showed that the two series corresponded quite closely in every respect, the yield decreasing proportionately to the extent of sulphonation. It is therefore impossible, under these conditions, to prepare nitronaphthalenes from α-naphthalenesulphonic acid, although a higher degree of nitration is obtained than with pure naphthalene.

With regard to the preparation of nitronapthalenes from naphthalene direct, the authors made a number of trials, which show that it is best to nitrate with a mixture of nitric and sulphuric acids, using about three times as much acid of 36° B., as of naphthalene. The amount of sulphuric acid to be used, will vary with the degree of nitration desired, and it ranges from 4:1 (nitric:sulphuric) for low melting points, to 3:2 for the higher derivatives.—D. B.

Gunpowders, Composition of Various. Merle. Moniteur Scientif. 1897, **25**; through Pharm. Centralh. 1897, **38**, [31], 503.

According to the author, the following is the composition of certain powders :—*Diorrexin:* Potassium nitrate, 42·78; sodium nitrate, 23·16; sulphur, 13·40; charcoal, 7·49; wood powder, 10·97; picric acid, 1·65; moisture, 0·55 parts. *Azotin* and *Haloxylin* also contain sodium nitrate, which, weight for weight, gives more oxygen than the potassium salt. *Amid.* contains 38 per cent of ammonium nitrate; it gives but little flame, but develops great power. *Saxifragin* contains barium instead of potassium nitrate; its energy is, however, but slight.

Among the blasting powders which derive a part of their oxygen from potassium chlorate, are :—*Himly Powder,* containing potassium chlorate, 45; potassium nitrate, 35; coal tar, 20 parts. The tar is dissolved in benzene, mixed with the salts, and the solvent driven off. *Michalewsky Powder* contains potassium chlorate, 50; manganese peroxide, 5; and bran, 45 parts. *American Comet Powder* consists of a mixture of potassium chlorate, 75; resin, 25 parts. *Augendre Powder* contains potassium chlorate, 49; sugar, 23; potassium ferrocyanide, 18 parts. *Janite* contains potassium nitrate, 70; sulphur, 12; bitumen, 18; picric acid, 0·4; potassium chlorate, 0·4; sodium carbonate, 0·3. *Amidogen* consists of nitre, 73; sulphur, 10; charcoal, 8; starch or bran, 8; and magnesium sulphate, 1. *Lithofracteur* contains sodium nitrate, 65; sulphur, 13; and guncotton, 22 parts.—J. O. B.

High Explosives, The Walker Method of Firing. J. Mein. Eng. and Mining J. 1897, **64**, [13], 364.

The object of this method is to avoid the danger incident to the ramming of shots when the detonator and charge of high explosives are placed together in the shot-hole. A paper tube, 6 in. in length and $\frac{7}{8}$ in. in diameter, is fixed in the charge, preferably during the process of manufacture. This tube forms a chamber for the reception of the detonator. The charge is placed in position in the shot-hole by means of a hollow brass or copper tube, of sufficient length to suit any hole, with an outside diameter of ½ in. and an internal diameter of $\frac{3}{8}$ in., and with one end slightly reduced in thickness, so as to admit of its being inserted into the paper tube. Then, after the ramming is finished, the detonator is pushed through the hollow tube by means of a copper rod. The detonator being held in position by means of a simple spring made of cane or some similar material, the copper rod and hollow tube may be simultaneously withdrawn, and the shot fired in the usual way. The total additional cost of this arrangement is said not to exceed 1 c. per shot.—A. S.

Plumbates, Alkaline Earth [$XPbO_3$]; Manufacture and Properties of. G. Kassner. Rev. Chim. Ind. 1897, **8**, 227.

See under VII., *page* 913.

PATENTS.

Mixing and Conveying Acids for Use in Nitroglycerine and other Manufactures, An Improved Method and Apparatus for. G. Kynoch and Co., Ltd., and A. T. Cockings, Witton. Eng. Pat. 22,717, Oct. 13, 1896.

Drawings and descriptions are given of the apparatus employed, which consists of two vertical closed iron cylindrical vessels, supported over a larger horizontal cylinder, with which they are connected by pipes and cocks. The vertical vessels contain the nitric acid and the sulphuric acid, and serve as measuring vessels, and can be put in connection with a vacuum pump; and the invention consists in "introducing the acids into their respective measuring vessels by means of reduced pressure therein," and, after allowing the acids to flow into the lower vessel, mixing and delivering them by means of compressed air.—W. M.

Nitroglycerin Explosives, Impts. in Manufacture of. C. E. Bichel, Hamburg. Eng. Pat. 17,367, Aug. 5, 1897.

This invention relates to the class of explosives in which nitroglycerin is absorbed in a suitable mixture of wood meal and saltpetre, and consists in the replacing of about 5 per cent. of the nitre by "chromates, bichromates, or permanganates of the alkalis."—W. M.

Matches, Impt. in Manufacture of. J. Craveri, Buenos Ayres. Eng. Pat. 18,563, Aug. 10, 1897.

The inventor proposes a composition for the heads of matches or tapers, which is entirely free from phosphorus. The following proportions are given :—

"Acid persulpho-cyanic, 2 parts; cyanogen persulphide, 4 parts; nitronaphthalene, 2 parts; xanthate of potassium, 1 part; charcoal, 1 part; chlorate of potassium, 20 parts; peroxide of lead, 50 parts; trisulphide of antimony, 10 parts; gelatin or fine glue, 10 parts."—W. M.

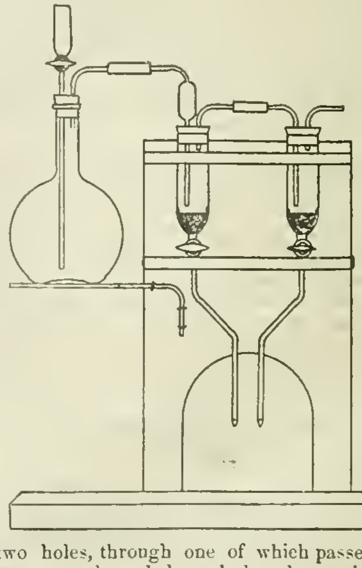
XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

Sulphur, Determinations in Iron and Steel, A New Apparatus for, and a Useful Form of Wash Bottle. R. K. Meade. J. Amer. Chem. Soc. 1897, **19**, 581—585.

The apparatus shown in Fig. 1, and drawn to one-eighth scale, consists of the following parts :—(1.) A 500 c.c. flask of the "Joliet" pattern, fitted with an india-rubber stopper

Fig. 1.



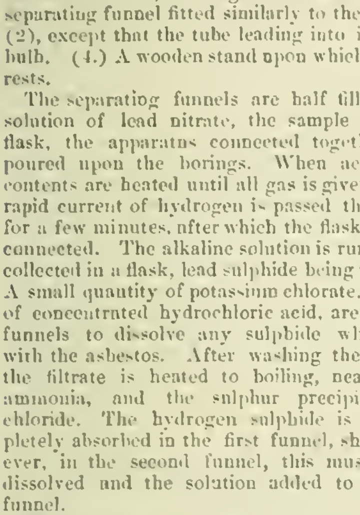
having two holes, through one of which passes a funnel with glass stop-cock, and through the other a piece of glass tubing bent at right angles about the middle point. (2.) A

1-oz. separating funnel fitted with a two-hole rubber stopper provided with tubes for the admission and exit of the gases. The tube leading from flask to funnel is blown into a bulb of 25 to 50 c.c. capacity. The funnel itself is indented in three places, equi-distant from each other, and about $\frac{1}{2}$ in. from the bottom, or just where the walls begin to narrow to meet the stop-cock. In the bottom of the funnel is placed a little glass wool, and upon this sufficient asbestos to reach level with the dents in the walls of the funnel. Over this, a thin perforated disc of platinum, made to fit the funnel closely, is pressed down under the dents, holding the asbestos in place. (3.) A separating funnel fitted similarly to the one described under (2), except that the tube leading into it is not blown into a bulb. (4.) A wooden stand upon which the entire apparatus rests.

The separating funnels are half filled with an alkaline solution of lead nitrate, the sample is weighed into the flask, the apparatus connected together, and dilute acid poured upon the borings. When action moderates, the contents are heated until all gas is given off. A moderately rapid current of hydrogen is passed through the apparatus for a few minutes, after which the flask and funnels are disconnected. The alkaline solution is run through a filter and collected in a flask, lead sulphide being retained on the filter. A small quantity of potassium chlorate, together with 20 c.c. of concentrated hydrochloric acid, are introduced into the funnels to dissolve any sulphide which may be mixed with the asbestos. After washing the funnels and filters, the filtrate is heated to boiling, nearly neutralised with ammonia, and the sulphur precipitated with barium chloride. The hydrogen sulphide is in most cases completely absorbed in the first funnel, should any form, however, in the second funnel, this must be collected and dissolved and the solution added to that from the first funnel.

Fig. 2 represents a wash-bottle of the usual type except that the shorter tube is made of very stout glass tubing. To this is fastened a piece of hard wood $\frac{3}{8}$ by $\frac{1}{4}$ by $\frac{3}{4}$ ins., notched with a V-shaped notch across its face and with a groove cut down its sides parallel to the neck of the flask. A piece of stout wire is bent with a loop at either end. The larger of these loops is made of sufficient size to admit the thumb comfortably and is covered with a piece of small rubber tubing. The smaller is bent over the rubber tubing and wood and is large enough to slide up and down freely in the vertical grooves with about half an inch play. The bottle is grasped by the last three fingers of the hand, and the thumb is passed through the loop in the wire, leaving the first finger free to guide the jet. A slight pull from the thumb is sufficient to bring the wire down upon the rubber, pressing the latter into the notch and effectually closing the opening. By doing this before ceasing to blow into the bottle, the fumes of the acid or steam or whatever volatile substance the bottle may contain, are prevented from passing back into the mouth and a steady stream is kept going from the jet for some moments or until the pressure of the thumb is relaxed, without further effort on the part of the operator.—D. B.

Fig. 2.



Beetroot Juice, Flask for the Polarisation of. D. Sidersky. Bull. Assoc. Chim., 1897, **14**, 1170—1171.

The custom of polarising juice clarified with 10 per cent. of basic lead acetate necessitates calculations which may be easily obviated.

The author has arranged a flask of 100—110 c.c. with a third mark on the elongated neck indicating 122·8 c.c. as given by the proportion $(5 \times 16 \cdot 29) : 100 = 100 : 122 \cdot 8$.

On filling the flask to the mark 100 with juice, adding basic lead acetate to 110, and completing to 122·8 c.c. with water, and polarising the filtered liquid, the polariscope reading with a 20 cm. tube will be five times the sugar content per 100 c.c. of juice, and multiplication by 0·2 presents no difficulty.

For the analysis of massecuites, 20 grms. in 100 c.c. of water is made up to 122·8 c.c. with basic lead acetate and water. The direct reading gives the sugar per cent. on the massecuite.—L. J. de W.

PATENT.

Gas [Oxygen, Carbonic Acid, &c.] Absorbing Apparatus for Ascertaining the Quantity of a Particular Gas in a Gaseous Mixture [Smoke-Gas, &c.] M. Arndt, Aachen, Germany. Eng. Pat. 25,762, Nov. 16, 1896.

The apparatus consists of an upper vessel for containing the gas, and a lower vessel for enclosing the absorbing medium, these vessels being connected end to end, but being normally separated by a movable closure or partition which carries or has connected to it, the absorbing medium. Solid absorbents, such as phosphorus rods for oxygen, are enclosed in a cage attached to the partition, whilst in the case of liquid absorbents, such as potash lye for carbonic acid, plates or fragments, &c. of glass, pumice stone, &c. are wetted or saturated with the liquid. The gas vessel is connected through a three-way supply cock to a measuring tube, the open lower end of which is immersed in a vessel of liquid. When the gas vessel has been filled with the gas, the partition and the absorbing medium are raised into the gas vessel until all absorption has taken place, the diminution in volume of the gas being indicated in the measuring-tube. The gas vessel may also be connected with a vacuum gauge, the pointer of which indicates the degree of rarefaction produced by the absorption.—R. A.

INORGANIC CHEMISTRY.—QUALITATIVE.

Arsenic, Antimony, and Bismuth: Separation from the Precious Metals by Hypophosphorous Acid. L. Vanino. Ber. **30**, [14], 2001—2003.

Platinum, iridium, and rhodium are not precipitated from strongly acid solution by hypophosphorous acid, and can thus be separated from ruthenium, palladium, osmium, gold, silver, mercury, arsenic, antimony, and bismuth, which are so precipitated. If to this precipitate, hydrogen peroxide be added in sufficient quantity, the last three metals redissolve, and can be separated from the rest by filtration, and then reprecipitated by warming after addition of more hypophosphorous acid, when the arsenic falls almost at once, the others after a longer time.—J. T. D.

Selenium in Commercial Sulphur. T. D. Reed, M.D. Amer. Pharm. Assoc. 1897; through Pharm. J. 1897, **59**, [1421], 264.

The author finds that there is no difficulty, in America, in obtaining sulphur free from selenium. The official test of the U.S.P. consists in boiling 0·5 grm. of the sulphur with 0·5 grm. of potassium cyanide in 5 c.c. of water and filtering, then adding excess of hydrochloric acid to the filtrate. In the presence of selenium a reddish coloration is obtained. In order to obtain more complete solution of the sulphur, it was found necessary to increase the amount of cyanide to 1·25 grms., and continue the boiling for half an hour. To determine the delicacy of the test, an experiment made with fused selenium showed that it would indicate $\frac{1}{300}$ of a grain. Care must be taken to avoid traces of iron, since that metal gives a coloration with the test closely resembling that of selenium.—J. O. B.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Water-Analysis, Colorimetric Estimation of Ammonia, of Nitrites, and of Iron. J. König. Chem. Zeit. **21**, [60], 599—601.

To avoid the labour of preparing continually fresh solutions for colorimetric comparison, and to provide a uniform set of tints, the author has had copied by a painter the tints produced in 100 c.c. of water containing known quantities of ammonia, of potassium nitrite, and of iron, by Nessler's reagent, zinc iodide and starch, and ammonium sulphocyanide solution respectively. These tints have been reproduced by lithography, and six graded slips, length and breadth corresponding to the height and diameter of a Nesslerising tube filled to the 100 c.c. mark, are arranged as the faces of a hexagonal prism, on a stand, so that they can be rotated round a vertical axis, and successively brought into comparison with a Nessler glass, containing the liquid for examination, which is supported on an arm of the same stand. These slips are marked in each case with the quantity of ammonia (or other substance) in 100 c.c., to which they correspond; but the author advises each analyst to make and test a series of standard solutions, and once for all to mark the corrections that may be needed for his personality. This done, the estimation of ammonia in a water is made by taking 300 c.c. of the water, adding to it in a stoppered cylinder, 2 c.c. of sodium carbonate solution (2·7 pure soda crystal in 5 parts of water) and 1 c.c. of caustic soda (1 part pure sodium hydroxide in 2 parts of water), shaking, allowing to settle, and pouring off 100 c.c. into the Nessler cylinder. If, after addition of the reagent, the colour be deeper than the deepest tint, 50 c.c. are poured away and 50 c.c. of pure water added. When the right tint has been struck, check experiments may be made by diluting to different extents successive quantities of the water, but this dilution must not be carried too far. For the estimation of nitrites, 100 c.c. of the water can be used directly, save in the case of muddy or coloured waters, when precipitation by sodium carbonate, sodium hydroxide, and a little alum is resorted to, exactly as in the case of ammonia. To 100 c.c. of the water, zinc iodide and starch are added and a little dilute sulphuric acid, and the tint struck compared with those on the appropriate comparison instrument. For iron, 200—500 c.c. are oxidised by boiling with a fragment of potassium chlorate and 1 c.c. of hydrochloric acid, allowed to cool, brought to the original bulk with distilled water, and 100 c.c. taken for the estimation.—J. T. D.

Total Ammonia in Gas Liquor, Estimation of. E. Donath and K. Pollak. Zeits. angew. Chem. 1897, 555.

By direct experiment the authors find that sodium hypobromite decomposes thiocyanogen, producing a certain amount of gas which contains carbonic oxide as well as nitrogen. The processes for estimating the ammonia in gas liquor, which depend on the measurement of the nitrogen evolved by the action of this reagent on the ammoniacal salts, are therefore not trustworthy, as they cause the samples to be valued above their true strength; and it is accordingly necessary to fall back upon the older methods of distillation with lime.—F. H. L.

Iron, A Modification of the Permanganate Method for the Determination of. H. P. Cady and A. P. Ruediger. J. Amer. Chem. Soc. 1897, **19**, 575—581.

The authors have found it possible to titrate iron with permanganate in the presence of hydrochloric acid, by adding an excess of mercury sulphate to the solution. The solutions are prepared in the following manner:—

Solution A. — A 5 per cent. solution of stannous chloride. This need not necessarily be freshly prepared, but it should give no precipitate when diluted with 20 times its volume of water and boiled. It may be kept in good condition by acidifying with hydrochloric acid and putting a few fragments of tin into the bottle.

Solution B.—Hydrochloric acid of sp. gr. 1·10.

Solution C.—This is made by adding to 200 grms. of mercuric sulphate, 80 c.c. of concentrated sulphuric acid, and mixing the resulting paste with 800 c.c. of water. (If a yellow precipitate be formed, more sulphuric acid is added.) To this is added 100 grms. of orthophosphoric acid dissolved in a small quantity of water, and the whole is made up to one litre.

Method 1.—Dissolve the substance in water with a varying quantity of hydrochloric acid, and heat to boiling after diluting to 100 c.c., reducing with stannous chloride until the solution becomes colourless, and potassium thiocyanate (sulphocyanide) ceases to give a coloration. Any excess of stannous chloride that may have been used is oxidised by potassium permanganate until the solution gives a faint but distinct colour with potassium thiocyanate, after which, one or two drops of stannous chloride are added very cautiously. This operation is carried on at a boiling heat. After cooling the solution, 50 c.c. of dilute sulphuric acid are added, and for every 10 c.c. of hydrochloric acid that has been used, 35 c.c. of the mercury sulphate solution; the whole is then diluted to 400 c.c., and titrated with potassium permanganate.

Method 2.—The substance is dissolved best in a small Erlenmeyer flask in 15 c.c. of hydrochloric acid, 2 c.c. of mercury sulphate are added, the mixture is heated, and stannous chloride added in small quantities at a time, until the solution becomes colourless. The contents of the flask are rinsed into a beaker and diluted to about 300 c.c., 50 c.c. of dilute sulphuric acid are added, and then 45 c.c. of mercury sulphate. The solution is then ready for titration without cooling, as the amount added should cool it sufficiently. Method 1 can be used in all cases, while method 2 (which is much shorter), can always be used where there is not too much insoluble residue, which might obscure the end of the reaction. The points requiring special care in this process are:—

1. Use stannous chloride which is in good condition.
2. Have the smallest possible excess of stannous chloride present after the reduction is completed.
3. Carefully adjust the proportions between the mercury sulphate and the hydrochloric acid. Less than three minutes, in the case of soluble salts, has been found sufficient time for solution, reduction, and titration.—D. B.

Iron and Aluminium, The Separation of, by means of Sodium Peroxide. C. Glaser. Chem. Zeit. 1897, **21**, 678.

Ferric oxide and alumina, in phosphates and other minerals, may be separated and estimated quantitatively by making use of their different behaviour towards sodium peroxide: for, while solutions containing aluminium are affected by this reagent exactly as by alkalis or alkaline carbonates, this is not the case with ferric salts. When a solution of a ferric salt is treated in the cold with excess of dry sodium peroxide, no precipitate is formed in the effervescing liquid, and any cloudiness observed disappears on adding excess of reagent. The iron is present as a ferrate, and on boiling is precipitated as hydroxide; and for quantitative estimation this precipitate should be dissolved in hydrochloric acid, and the iron reprecipitated with nitroso-β-naphthol or with ammonium sulphide, the latter in ammoniacal solution containing citric or tartaric acid. The precipitate is, in either case, converted into ferric oxide and weighed in this form.

A solution containing iron and aluminium in the absence of calcium phosphate is nearly neutralised with ammonia, and is then ready for the separation; if, however, calcium phosphate be present, it must be carefully removed by means of ammonium acetate or by E. Glaser's method, and the solution then nearly neutralised as before. To the cold solution, dry sodium peroxide is added until the solution appears perfectly clear. As a rule for 0·4 grm. of substance there is required 3—6 grms. of sodium peroxide. The clear solution is rapidly heated to boiling point, the precipitate allowed to settle, and the whole poured through a thick filter. In the filtrate, alumina is estimated by a usual method, preferably by precipitation with pure phosphoric acid in the presence of ammonium acetate. The ferric hydroxide is dissolved in hydrochloric acid and purified as described above. In the presence of silicates the results may be slightly too high, and the original solution should therefore be free from silicic acid.

The author considers the above separation more rapid than the ordinary fusion method, and free from the disadvantage of the latter that the platinum crucible employed is slightly attacked, and the apparent weight of the ferric oxide increased by traces of platinum.—R. B. B.

Zinc, Estimation of, by the Schaffner Method in Plumbiferous Minerals. H. Pellet. Bull. Assoc. Belge des Chim. **11**, [4], 126—130.

Having found that the ammoniacal salts, produced when ammonia is employed to neutralise the acid solution of the mineral before titrating the zinc with sodium sulphide, retain in solution the lead present, and so falsify the titration, the author substitutes potash in that operation, leaving the liquid just sufficiently acid to prevent the deposition of the zinc. The solution is then poured into ammonia, agitated, and filtered, the filtrate being titrated with standardised sodium sulphide, one drop of ferric chloride (20 per cent.) solution being added as indicator. By this means the method retains its accuracy even in presence of 40 per cent. of lead, whereas in the ferrocyanide method the lead has to be removed before titrating the zinc.

It is found that the volume of sodium sulphide solution required per unit of zinc increases slightly with the dilution of the liquid.

An important point is to pour the neutralised solution into the ammonia, and thereby obviate the deposition of zinc oxide, which would otherwise have to be redissolved.

In dissolving the mineral, the author prefers to oxidise the iron present by means of potassium chlorate instead of nitric acid.—C. S.

Nickel and Cobalt. Separation from Iron. O. Ducru. Comptes Rend. 1897, **125**, [11], 436—439.

If a solution of iron containing nickel be precipitated by excess of ammonia, part of the nickel dissolves, part goes into the precipitate. But if the solution with the precipitate suspended in it be now electrolysed, the whole of the nickel is deposited on the cathode, along with a trace of iron, which, in exact analyses, can be subsequently determined. In the process of analysis, the solution, in which the iron has been peroxidised, is evaporated to dryness with a slight excess of sulphuric acid, taken up with as little water as possible, 5 to 10 grms. of ammonium sulphate added, and the whole warmed till a clear solution is obtained. The liquid is transferred to Riche's apparatus (Ann. Chim. Phys. [5], **13**, 528), in which has been placed 60 or 70 c.c. of strong ammonia, and electrolysed with a current density of 25 to 45 milli-ampères per sq. cm. of the cathode. In less than four hours the electrolysis is complete. Figures are given both for known mixtures of iron and nickel or iron and cobalt and for nickel steels, which show that the method is very accurate, and that for ordinary purposes it is not necessary to take into account the iron deposited on the cathode. Silicon and carbon need not be separated in the first place, nor do the small quantities of manganese or phosphorus, or of chromium, interfere with the method. A small amount of chromic acid, however, prevents the deposition of the nickel; the author has this point under investigation.—J. T. D.

Nickel-Copper Alloys, Assay of. A. Riche. J. Pharm. Chim. 1897, **6**, [7], 300—303.

The minting of cupro-nickel with 15 or 25 per cent. of nickel has necessitated the introduction of a safe and rapid method of assay. It is only needful to determine one constituent of a binary alloy, and in this case, owing to the higher price and lower purity of nickel, this metal may be estimated, and the copper may be found by difference. Formerly the author had precipitated the copper electrolytically with an element giving an E.M.F. of 2 volts, and had deposited the residual nickel in ammoniacal solution with two such elements. Under these conditions the nickel solution rapidly grows warm, and the deposit may become grey, loose, and even partly oxidised. It is better therefore to use only 3 Daniell cells—a method which gives perfect results and demands no supervision. From 0·5 to 1 grm. or more of alloy is dissolved in the minimum of nitric acid, a little water is added after solution is complete, and the nitric acid is expelled by two consecutive evaporations with 5—6 drops of sulphuric acid. After redissolving in water with a few drops of sulphuric acid, the copper is deposited electrolytically until the solution gives no indication with bicarbonate and ferrocyanide. The cone with the copper is then washed into the containing vessel, and may be weighed. The residual solution is then mixed with an excess (usually 12—15 c.c. suffices) of ammonia, and is electrolysed with 3 Daniell cells. The results are quite concordant, but the analysis may show a deficiency of 0·8—0·9 per cent., due to impurities in the nickel, some of which are observable, forming a reddish flocculent precipitate in the solution, which, however, does not interfere with the accuracy of the nickel determination. It was formerly believed that a current with an E.M.F. lower than 2 volts would fail to deposit nickel from ammoniacal solution. By allowing a longer time (a whole night), the deposition may be effected completely with a current of 0·1 ampère from the battery employed for the copper electrolysis. The cone with the nickel deposit is washed successively with water and alcohol, and is dried and weighed. In the assay of coinage alloys, 0·475 grm. of good copper is deposited in 4 hours with a current which falls during that time from 0·5 to 0·1 ampère. The nickel is deposited in 4 or 5 hours, the ampère meter showing a current of 0·3 ampère at the beginning, and 0·1 to 0·2 at the end of the operation, when 15 c.c. of ammonia has been used.—W. G. M.

ORGANIC CHEMISTRY.—QUALITATIVE.

Dégras, Genuine and Artificial; Distinction between. F. Jean. Rev. Chim. Ind. 1897, **8**, 227. (See also this Journal, 1895, 281; 1893, 937; 1892, 639; 1891, 557.)

In a note appended to Baron's paper on artificial dégras (see page 922), the author states that on salting out the sodium soaps of true moëllon, and decomposing them with hydrochloric acid, a black, brittle, resin-like body called *dégragene* is produced, which contains nitrogen, and is soluble in alcohol and ether, but insoluble in petroleum spirit. Artificial dégras also yields a similar compound analogous to leucine, but it is accompanied by a large proportion of another substance of like appearance, which is free from nitrogen.—F. H. L.

Earthnut, Detection of, in Chocolate. Bilteryst. Bull. de l'Assoc. belge des Chim., March 1897, 447; through J. Pharm. Chim. 1897, **6**, [1], 29.

To detect the addition of arachis by microscopical examination, the chocolate is first freed from fat and sugar, and then examined in a medium of water and glycerin. The characteristic starch granules of arachis are thus shown very distinctly; they are larger than those of the natural starch of theobroma and each granule has a central nucleus.

The powder free from fat and sugar, is also macerated for 24 hours in a solution of 50 grms. of chloral hydrate in 40 grms. of water. The characteristic cells of *Arachis hypogæa* are then easily distinguished by their peculiar internal markings. Chemically, the adulteration is detected by the examination of the separated fat and the determination of the albuminoids. The fat is extracted by means of carbon tetrachloride, and its refractive index determined with the oleorefractometer. The figure for pure cacao butter is $-19°$, while for earthnut fat it is $+3°$. The author found the refraction index of the fat of a mixture of 5 per cent. of arachis with chocolate to be $-18°$, and of that from a 50 per cent. mixture $-7°$. This method, however, alone, will not reveal the presence of pressed marc of earthnut in chocolate. To detect this latter adulteration the determination of the albuminoids must be made. Cacao contains 18 per cent. of these; arachis, 20 per cent.; and the oil-free marc of earth-nut, 45 to 47 per cent. of albuminoids.

—J. O. B.

Eucalyptol [Tests for Purity of]. A. Schamelhout. Ann. Pharm. 1897, **3**, 257.

A useful reagent for the detection in eucalyptol of small amounts of eucalyptus oil and turpentine, consists

of a solution of 10 drops of bromine in 10 c.c. of pure chloroform. This reagent also serves to detect turpentine in eucalyptus oil. The reagent is added drop by drop. A yellowish-green colour is first produced, which changes on further addition to reddish yellow; and the number of drops necessary to bring about these colour changes indicates the purity of the sample. For example:—

	Drops of Reagent	
	For Yellowish-Green Colour.	For Reddish-Yellow Colour.
Pure eucalyptol	4	8
Rectified oil of turpentine	No coloration, even with 250 drops.	
Eucalyptus oil	25	95
Eucalyptol, with 5 per cent. eucalyptus oil	11	18
Eucalyptol, with 2·5 per cent. eucalyptus oil	7	12
Eucalyptol, with 2·5 per cent. turpentine	16	23
Eucalyptus oil, with 2·5 per cent. turpentine	..	135

—R. B. B.

Bisobol Myrrh, Composition of, and Distinction from Official Myrrh. Tucholka. Arch. der Pharm. 1897, 290; through Pharm. Centralh. 1897, **38**, [31], 500.

Six drops of a 1 in 15 petroleum ether extract of bisobol myrrh are mixed with 6 c.c. of acetic acid; then 3 c.c. of concentrated sulphuric acid are added, so as to form a lower layer. A rose-red colour is developed at the line of contact, and in a short time the whole acetic acid solution is coloured red, the tint being permanent for some time. If the petroleum extract be made stronger than 1 in 15, a brown colour is produced. Official myrrh treated in a similar manner gives only a slight red colour in the acetic layer, which does not intensify on standing; and the zone of contact is at first green, then brown, with a greenish fluorescence. Bisobol myrrh contains: gum soluble in water, 22·1; gum soluble in soda solution, 29·85; resin, 21·5; bitter principle, 1·5; volatile oil, 7·8; water, 3·17; vegetable and inorganic matter, 13·4 per cent.

The essential oil gave the above characteristic colour reaction very markedly; when treated with gaseous hydrochloric acid by Wallach's method, the terpene gave small, well-formed crystalline tablets of hydrochloride melting at 79·3 C. The terpene itself, which is named bisaboleue, had the abnormally high boiling point 259°—260°·3 C., and has not been identified with any similar body hitherto isolated. It is considered probable that it consists of a double molecule $(C_{10}H_{16})_2$. Besides bisabolene the oil probably contains alcoholic bodies and esters, since it reacted violently with benzoyl chloride on warming. The reddish-brown oil from which the crystalline bisabolene hydrochloride had been separated, fractionated between 230°—231° C., also gave the characteristic colour reaction. The resin soluble in alcohol was markedly acid in reaction; it contained two acids, giving soluble and insoluble lead salts respectively. The gum soluble in water is probably a calcium arabinate.—J. O. B.

ORGANIC CHEMISTRY.—QUANTITATIVE.

Hæmateïn, The Estimation of, in Logwood Extracts. Aglot. With Report by E. Jaquet and O. Michel. Bull. Soc. Ind. Mulhouse, 1897, 250—258.

A solution of the sample of a definite strength is prepared, a few drops of ammonia added, until a cherry-red colour just appears, and normal potassium chromate then added in sufficient amount to change the colour to a blue free from violet. By means of the author's optical apparatus a definite source of light is viewed through a layer of the liquid thus produced, and the thickness of the layer is adjusted until no light reaches the eye. The amount of colouring matter is inversely proportional to the thickness of the layer when absorption of light is complete. For the above test, 10 grms. of the sample are dissolved in a litre of water, 10 c.c. of this solution are made alkaline with ammonia, and 10 c.c. of a 3 per cent solution of potassium chromate added; the liquid is then boiled for 10 minutes, allowed to cool, and diluted to 50 c.c. A part of this solution is introduced into the apparatus and observed as described. According to the author's results, the presence of impurities, such as chestnut extract or molasses, does not cause a difference of 1 per cent. in the amount of colouring matter found, and he therefore concludes that this method may be used for the comparison of samples from various sources with each other and with pure hæmatoxylin.

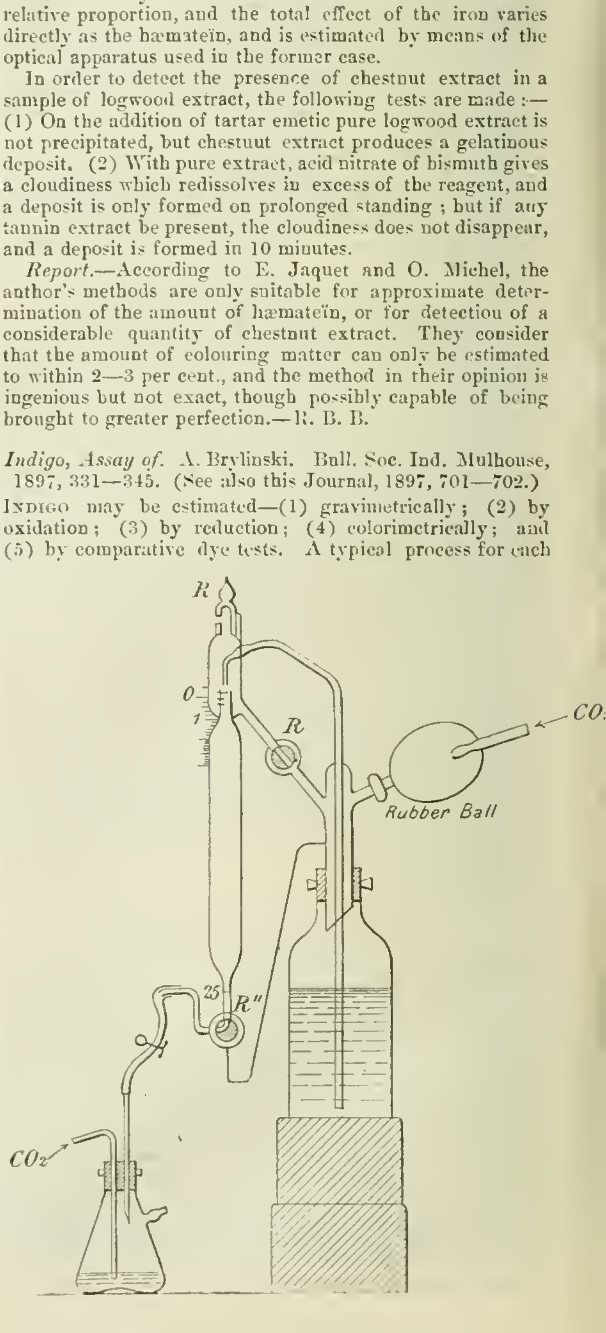
When testing different samples from the same factory, where approximately the same wood is always used and chestnut extract is not added, it suffices to precipitate portions of the solution with albumin and with acetate of iron, the former causing precipitation of tannin only, the latter of tannin, certain pectic matters, and hæmateïn. The wood will always contain tannin and hæmateïn in the same relative proportion, and the total effect of the iron varies directly as the hæmateïn, and is estimated by means of the optical apparatus used in the former case.

In order to detect the presence of chestnut extract in a sample of logwood extract, the following tests are made:—(1) On the addition of tartar emetic pure logwood extract is not precipitated, but chestnut extract produces a gelatinous deposit. (2) With pure extract, acid nitrate of bismuth gives a cloudiness which redissolves in excess of the reagent, and a deposit is only formed on prolonged standing; but if any tannin extract be present, the cloudiness does not disappear, and a deposit is formed in 10 minutes.

Report.—According to E. Jaquet and O. Michel, the author's methods are only suitable for approximate determination of the amount of hæmateïn, or for detection of a considerable quantity of chestnut extract. They consider that the amount of colouring matter can only be estimated to within 2—3 per cent., and the method in their opinion is ingenious but not exact, though possibly capable of being brought to greater perfection.—R. B. B.

Indigo, Assay of. A. Brylinski. Bull. Soc. Ind. Mulhouse, 1897, 331—345. (See also this Journal, 1897, 701—702.)

Indigo may be estimated—(1) gravimetrically; (2) by oxidation; (3) by reduction; (4) colorimetrically; and (5) by comparative dye tests. A typical process for each



of these methods is described, and their relative value discussed. The author concludes the method by reduction, as proposed by G. Engel, to be the best. The process is based on the fact that indigo can be reduced quantitatively by a solution of vanadyl sulphate.

When an acid solution of vanadium sulphate is reduced by zinc, two successive stages of reduction are observable. The originally yellow solution at first turns green, forming vanadous sulphate, which may also be obtained by reducing with a bisulphite. This vanadous sulphate reduces permanganate and ferric salts, but not indigo. On continuing the addition of zinc to the hot solution, the latter turns to a purplish-blue colour; this is the vanadyl sulphate of Würtz, recommended by Engel for the titration of indigo. The solution can, however, be only used in an inert atmosphere, as it absorbs oxygen from the air very rapidly, being reconverted into vanadous sulphate. The titration is performed, therefore, with a special apparatus in the presence of a current of CO_2. The vanadyl sulphate solution is run from a burette into a measured solution of indigo carmine until the colour of the latter has disappeared. The solution passes from a green to a pale yellow, the end reaction being indicated by a greenish-grey colour.

The details of the process are as follows:—10 grms. of ammonium vanadate are dissolved in 100 grms. of sulphuric acid of 66 B., with the aid of heat. The resulting red solution is poured into two litres of water of 50 C. To this are added by degrees 50 grms. of zinc powder until the solution shows a purplish-blue colour. Filter and allow to cool.

A measured quantity of indigo solution is put into an Erlenmeyer filtering flask, provided with a side tube. This flask is closed with a double perforated cork, through which two tubes pass, one of them being connected with the burette, and the other with a CO_2 generator, as shown in the drawing. At first a standard indigo is titrated, and then the sample to be assayed, and the value of the latter is then calculated.

To guard against the possibility of oxygen being carried into the apparatus by the CO_2, the latter should be purified by first passing it through a solution of vanadyl sulphate. The small quantities of ferric salts present in the solution may occasion a very slight error, which may in most cases be neglected. But if need be, the iron present may be determined in the ash of a separate portion, and the quantity of vanadyl sulphate used for its reduction determined.

—I. S.

Sugar Content of Some Tropical Fruits, Determination of. H. C. Prinsen-Geerligs. Chem. Zeit. 1897, **21**, [72], 719.

A quantity of the fruit was taken, and the amount of external covering, flesh, and stone determined. 100 grms. of the edible portion were triturated with calcium carbonate to neutralise organic acids or acid salts, and then extracted with alcohol. The neutral alcoholic extract was evaporated on the water-bath, the residue taken up with water, treated with neutral lead acetate, and made up to 100 c.c. with water. After filtration, the solution was polarised and the amount of cupric reducing sugars determined in an aliquot portion. 50 c.c. of the filtrate were inverted with 5 c.c. of hydrochloric acid, and after dilution to 100 c.c., the solution was polarised and the cupric reducing power determined. The difference between the amount of cupric reducing sugar before and after inversion, was considered as due to cane sugar; and the percentage of this sugar was calculated from that value, since in no case was there any evidence of the presence of maltose or other carbohydrates capable of hydrolysis. The specific rotation of the reducing sugars was deduced from the difference between the calculated polarisation corresponding to the amount of cane sugar found as above and that observed in the original solution. Since it was assumed no reducing sugars but dextrose and levulose were present, the amounts of these two could be calculated from the above-mentioned difference. The following table shows the results obtained by the author:—

Fruit from	Average Weight of a Single Fruit.	100 Parts of Fruit contain			100 Parts of Flesh contain			
		Flesh.	Skin.	Stone.	Cane Sugar.	Glucose.	Levulose.	Total Sugar.
Achras sapota [sapodilla plum or naseberry]	60 grms.	85	13	2	7·02	3·70	3·40	14·12
Ananassa sativa [pine-apple]	300 „	67	33	..	8·61	1·00	0·60	10·21
Anona muricata [sour sop]	800 „	75	20	5	2·53	5·05	4·04	11·62
„ reticulata [bullock's heart or true custard apple of West Indies].	500 „	72	22	6	..	6·20	4·22	10·42
Anona squamosa [sweet sop]	140 „	50	38	12	0·50	5·40	3·60	9·50
Arto carpus integrifolia [Jack fruit]	11 kilos.	26	66	8	3·70	1·14	..	4·84
Averrhoa carambola [Caramba]	80 grms.	95	5	..	0·82	5·50	3·70	10·02
Carica papaya [papaw]	600 „	65	10	25	0·85	2·60	2·10	5·50
Cicca nodiflora [Phyllanthus distichus]	5 „	80	..	20	..	0·33	1·00	1·33
Citrullus edulis	2 kilos.	59	37	4	2·13	..	2·75	4·88
Citrus aurantium [orange]	80 grms.	80	20	..	3·06	2·40	1·60	7·06
Durio zibethinus [durian fruit]	1·5 kilos.	20	60	20	8·07	1·80	2·20	12·07
Flacourtia Ramontchi	6 grms.	100	..	..	0·50	0·41	0·70	1·61
Garcinia mangostana [mangosteen]	100 „	28	64	8	10·80	1·00	1·20	13·00
Eugenia javanica	50 „	100	..	..	0·53	3·20	3·20	6·93
Lansium domesticum [Lansa]	20 „	51	25	24	9·98	1·67	2·50	14·15
Mangifera indica [sweet mango]	200 „	67	3	30	9·48	0·62	1·98	11·98
„ „ [sour mango]	300 „	75	3	22	3·60	..	1·90	5·50
Musa paradisiaca [plantain]	100 „	70	30	..	13·68	4·72	3·61	22·01
Nephelium lappaceum [ramboutan]	20 „	40	50	10	7·80	2·25	1·25	11·30
Persea gratissima [alligator pear]	140 „	67	8	15	0·86	0·40	0·46	1·72
Psidium guajava [guava]	65 „	85	12	3	1·66	2·00	0·50	4·16
Spondias mangifera [hog plum]	120 „	80	2	18	2·94	1·68	1·84	6·46
Tamarindus indica [tamarind]	6 „	41	30	29	..	5·81	2·51	8·32
Zalacca edulis	30 „	58	15	27	8·07	2·40	..	10·47

—J. L. B.

Beeswax, The Examination of. S. Weinwurm. Chem. Zeit. 1897, **21**, [52], 519—520.

In working Hübl's process the author shortens the saponification by heating the wax and alcoholic potash in a covered flask for 30 minutes on a water-bath, then removing the cover and completely evaporating the alcohol. For the detection of small quantities of paraffin or cerasin, he describes a method based on the fact that an aqueous solution of glycerin dissolves the normal unsaponifiable matter in beeswax, whilst paraffin and cerasin (or 5 per cent. of resin) are insoluble. 5 grms. of the filtered beeswax are saponified with 25 c.c. of semi-normal alcoholic alkali in the manner described above, and, after the addition of about 20 c.c. of glycerin the flask is heated on the water-bath until there is a clear solution and then for several minutes longer. On now adding 100 c.c. of boiling water, the liquid remains more or less clear and transparent in the case of genuine beeswax, and it should be possible to read printed matter when placed beneath the flask. But when 5 per cent. of cerasin or resin is present, the liquid is turbid and it is

no longer possible to read the print through it. When a wax gives normal or nearly normal figures by Hübl's process, and a clear solution is obtained in this way, the sample is either pure or contains not more than about 3 per cent. of cerasin or resin. A second determination is now made, after addition to the wax of 5 per cent. of cerasin (0·25 grm.), and if the solution is only slightly turbid it proves that no cerasin was originally present; if, on the other hand, there is a marked precipitate, about 3 per cent. was present. If the first test gives a turbid solution it points to the presence of about 5 per cent. of either cerasin or resin. 3 per cent. of cerasin is then added to the wax before saponification, and a second test is made. If the original impurity was cerasin, there is now a precipitate: if not, there is still only a turbidity. Since cerasin enters into the composition of nearly all wax substitutes, this method alone is often sufficient to detect adulteration, and when carnaüba wax and stearic acid are employed so much of the latter is required that the detection of the admixture is easy. The author has examined a large number of samples by this method, and in some has detected the addition of trifling amounts of cerasin and tallow.—C. A. M.

Cellulose Carbohydrates, Quantitative Separation of, in Vegetable Substances. W. Hoffmeister. Landw. Versuchsstat. 1897, **48**, 401; through Chem. Zeit. 1897, **21**, 198.

The substance is first extracted with ether to remove all fat; the fat-free residue is then extracted, in the cold, by maceration in successive quantities of dilute hydrochloric acid and ammonia alternately, bodies containing much starch being first treated with malt extract to render that substance soluble. When extracted as far as possible in this manner, the residue, without drying, is treated with a 5 or 6 per cent. solution of soda. It is macerated for 1 or 2 days with occasional shaking, then slightly diluted, allowed to deposit, the clear solution decanted, and the insoluble matter thrown on a filter and washed with hot water. The alkaline extract is neutralised with hydrochloric acid, and the hemicellulose precipitated by pouring into a large excess of alcohol. The precipitate is collected, dried, and weighed. The insoluble residue is then extracted in a similar manner with Schweitzer's reagent and the resulting extract poured into alcohol, as above, to precipitate the cellulose. The residue which remains after this treatment is quite resistant to all solvents, and is dried and weighed as lignin. In each case the final product must be washed with a slight excess of ammonia before drying, to remove any trace of adherent hydrochloric acid, which is difficult to remove by simple washing, and the presence of which, during drying, causes decomposition.

The following are some figures obtained by this method:—

——	Hemicellulose.	Cellulose.	Lignin.
Wheat bran	17·8	5·40	5·10
	15·54	4·79	6·28
Linseed cake	2·25	3·36	5·61
	2·4	3·55	6·05
	..	..	6·30
Rye bran	16·8	2·49	5·94
Rape cake	Traces.	2·87	8·62
Hemp cake	2·52	1·55	26·28
Sunflower seed kernels	Traces.	1·45	0·10

—J. O. B.

Peppermint Oil, Determination of Menthol in. C. Kleber. Pharm. Rev. 1897, **15**, 135; through Chem. Zeit. 1897, **21**, 198.

5 grms. of the oil are mixed with 5 grms. of acetic anhydride and boiled for 30 minutes in a flask under a reflux condenser. In the meantime, the same volume of the same acetic anhydride is titrated with normal soda solution, and phenolphthalein. When the boiled fluid has cooled, the reflux tube is washed with a little water, and this is added to the acetylised mixture, and titrated also with normal soda. The difference in c.c. between the two titrations, multiplied by 0·156, gives the amount of menthol contained in the oil. By this process it only takes about 50 minutes to complete the determination.—J. O. B.

Balsams, Resins, and Gum Resins; Rational Examination of. K. Dieterich. Chem. Rev. Fett- u. Harz-Ind. **4**, [17], 233—236. (This Journal, 1897, 829—830.)

Resins (continued). Dammar. *Acid number:* 1 grm. treated with 50 c.c. of petroleum spirit (sp. gr. 0·7) and left for 24 hours in presence of 20 c.c. of $\frac{1}{2}$N aqueous-alcoholic potash. Then titrated (without addition of water) with $\frac{1}{2}$N sulphuric acid, and phenolphthalein as indicator. *Ash:* 1 grm. incinerated and calcined till constant; weighed when cold.

Dragon's Blood.—Resin number: 1 grm. is treated with 50 c.c. of petroleum spirit and acted on for 24 hours by 25 c.c. of $\frac{1}{2}$ N aqueous potash; then mixed with 250 c.c. of water and 100 c.c. of alcohol and titrated with $\frac{1}{2}$ N H_2SO_4. *Saponification value:* Same procedure, except that $\frac{1}{2}$N alcoholic potash is employed for the first reaction, the aqueous potash being then allowed to act during a second 24 hours. *Dracoalban test:* 10 grms. powdered and extracted with 25 c.c. of hot ether, the extract concentrated to 30 c.c., and mixed with 50 c.c. of absolute alcohol. After an hour's rest a white flocculent precipitate comes down. (Characteristic of Palm-dragon's blood only). No free acid is present.

Resina Guaiaci.—Acid number: 20 c.c. $\frac{1}{2}$N aqueous-alcoholic potash allowed to react on 1 grm. for 24 hours, followed by addition of 500 c.c. of water and titration with $\frac{1}{2}$N, H_2SO_4. *Ash:* Same as for dammar.

Mastic.—Acid number: Same method as for dammar, but with addition of 500 c.c. of water before titration. *Ash:* Same as for dammar.

Sandarac.—Acid number and ash are determined in same manner as for dammar.

Resina Pini.—Acid number: 1 grm. is dissolved in 50 c.c. of alcohol and titrated with $\frac{1}{2}$N alcoholic potash and phenolphthalein until a red coloration ensues. *Saponification value:* 1 grm. heated with 30 c.c. $\frac{1}{2}$N alcoholic potash for an hour under a reflux condenser. Titrated with $\frac{1}{2}$N H_2SO_4, after cooling and adding 100 c.c. of alcohol. *Ester number:* By subtraction.

Storax.—Acid number: 1 grm. dissolved in 200 c.c. of alcohol and titrated with $\frac{1}{2}$N alcoholic potash. *Saponification value:* Same method as for R. pini. *Ester number:* By subtraction. *Loss at* 100° *C.:* 1 grm. dried at 100° C. till constant. *Alcoholic portion:* 10 grms. dissolved by warming in 100 c.c. of 90 per cent. alcohol, filtered, and washed with 50 c.c. of hot alcohol, the filtrate being evaporated and the residue dried at 100° C. Creeping over is prevented by floating the evaporating dish in a basin of hot water on the water- or steam-bath. The content of dirt and woody fragments is determined by weighing the dried beaker and filter.

Terebinthina Communis et Veneta.—Acid number, ester number, and saponification value: Same methods as for R. pini; no water should be added in the last-named determination.

Gum Resins. Ammoniacum.—Acid number: 0·5 grm. is treated with water, and a current of hot steam passed through the liquid, the flask being heated to prevent condensation. The receiver contains 40 c.c. of $\frac{1}{2}$N aqueous potash, which, after 50 c.c. of distillate have been collected and the still neck and tube rinsed out, is titrated back. *Resin and saponification values:* Two 1 grm. samples are treated with 50 c.c. of petroleum spirit and exposed to the action of 25 c.c. of $\frac{1}{2}$N alcoholic potash for 24 hours, with frequent shakings. The one sample mixed with 500 c.c. of water and titrated with $\frac{1}{2}$N sulphuric acid gives the *resin number,* whilst the other, after a further 24 hours' exposure to the action of 25 c.c. of $\frac{1}{2}$N aqueous potash, *plus* 75 c.c. of water, is diluted with 500 c.c. of water and titrated, the *saponification value* being thus ascertained. *Loss at* 100° *C.* is determined by drying. *Examination for Galbanum:* 5 grms. are boiled a quarter of an hour with 15 grms. of HCl (sp. gr. 1·19) and filtered through a wet double filter. On super-saturating the clear filtrate with ammonia, the characteristic blue fluorescence appears when galbanum is present.

Asafœtida.—Acid number: Same method as for R. guaiaci. *Saponification value:* 1 grm. of finely-ground average sample is treated as in the case of the last-named resin, except that 200 c.c. of alcohol are added previous to titration. *Ester number:* By difference.

Galbanum.—Acid number: Same treatment as for ammoniacum, except that 500 c.c. of distillate are collected in place of 50 c.c. *Resin and saponification values*, also determined as in the case of ammoniacum. *Ash and loss at* 100° *C.:* Determined by incineration and drying respectively.

Olibanum.—Acid number: Same method as for mastic. *Saponification value:* 1 grm. of finely-ground sample boiled for one hour with 20 c.c. of $\frac{1}{2}$N alcoholic potash under a reflux condenser, and titrated with $\frac{1}{2}$N, H_2SO_4, after adding 500 c.c. of water. *Ester number and ash:* Same as in other cases.—C. S.

Methyl Alcohol, Formaldehyde, Formic Acid: Determination of Small Quantities of. Nicloux. Bull. Soc. Chim., 1897, **17**, 839—840.

The author's method (this Journal, 1897, 473) for determining small amounts of alcohol, can be applied to methyl alcohol, formaldehyde, or formic acid, provided there be no other substance present in each case capable of reducing bichromate. The procedure is the same as for ethyl alcohol, 5 c.c. of pure concentrated sulphuric acid being added to 5 c.c. of the solution, which is then heated for one minute and allowed to stand for five minutes. The author suggests solutions of bichromate of 19 grms. in the litre (2 c.c. of this solution correspond to 5 c.c. of a solution containing 1 c.c. of alcohol per litre), 17 grms. in the litre (2 c.c. = 5 c.c. of aldehyde of a strength of 1 grm. per litre), and 11 grms. in the litre (1 c.c. = 5 c.c. of formic acid of a strength of 1 grm. per litre) respectively. It is advisable to work with a series of comparison tubes prepared from solutions of known strengths, the better to hit the exact tints.—J. T. D.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Active Oxygen. The Process of Formation of. W. P. Jorissen. Ber. **30**, [14], 1951—1953.

Engler and Wild (Ber. **30**, 1669) explain the fact that in cases of oxidation by active oxygen, the amounts of oxygen absorbed by the directly-oxidised body (A) and the body oxidised by the active oxygen (B) are equal, by supposing that a peroxide of (A) is formed from the oxygen molecule, and that this peroxide then oxidises B:—

$$A + O_2 = AO_2$$
$$AO_2 + B = AO + BO$$

in opposition to van't Hoff's view that the oxygen molecule splits up into electrically opposite atoms, which then effect the oxidation:—

$$O_2 = \overset{+}{(O)} + \overset{-}{(O)},\ A + \overset{+}{(O)} = AO,\ B + \overset{-}{(O)} = BO.$$

Where such peroxides as Engler and Wild assume can be isolated, and there is nothing in the conditions of the experiment to prevent their formation, the first view is perhaps correct: but such a peroxide may possibly be formed as a result of a previous splitting up of the oxygen molecule:—

$$O_2 = \overset{+}{(O)} + \overset{-}{(O)},\ A + \overset{+}{(O)} = AO,\ AO + \overset{-}{(O)} = AO_2.$$

Arguments as to the possibility of forming such bodies must not be based on the behaviour of ozone, which is a different thing from active oxygen. In a case just investigated by the author, in which oxygen, rendered active by sodium sulphite, oxidises sodium arsenite, the formation from sodium sulphite of a body functioning like a peroxide would seem to be out of the question.—J. T. D.

Silicon Carbide. L. Franck. Stahl und Eisen, **17**, 485.

A sample of cast-iron, in the reduction of which calcium carbide had been employed, was found to contain a noteworthy amount of silicon carbide. The iron was dissolved by Moissan's method, silicates destroyed by hydrofluoric acid, graphite removed, and the residue treated with aqua regia, hydrofluoric acid, and concentrated sulphuric acid. There remained microscopic well-formed octohedra, partly bluish, partly yellow or greenish, resembling diamond dust. They had a sp. gr. of 3·1—3·3, scratched rubies, and differed from diamond in their action on polarised light. Analysis gave Si 14·34, C 81·95. Further research has shown that this diamond-like silicon carbide is contained in calcium carbide itself, in greater or less quantity according to the amount of silicious impurity in the materials from which the calcium carbide is prepared.—J. T. D.

Corrosive Mine Waters. L. J. W. Jones. Eng. and Mining J. 1897, **64**, [13], 368.

The corrosive action of the water of the Stanley Mine at Idaho Springs is so great that ordinary iron pipe is not able long to resist it; indeed, when the exhaust steam of the pumps is discharged into the "sump," the piping will sometimes only last a week. The corrosive action is lessened by the use of compressed air instead of steam. A sample of the water contained a considerable amount of a muddy-brown precipitate, which consisted essentially of hydrated basic sulphate of iron. The filtered water contained 0·3 per cent. of salts, mainly metallic sulphates, in solution. Analysis of the filtered water resulted as follows: SiO_2, 0·0438 parts per thousand; NaCl, 0·01315; Na_2SO_4, 0·31172; K_2SO_4, 0·15548; $Al_2(SO_4)_3$, 0·019787; $ZnSO_4$, 0·12244; $MnSO_4$, 0·42714; $MgSO_4$, 0·46716; $CaSO_4$, 0·63629; $Fe_2(SO_4)_3$, 0·60336; $FeSO_4$, 0·009337; $CuSO_4$, 0·191801; total, 3·002065 parts per thousand. After trying various materials, bronze piping was found to satisfactorily withstand the action of the water. In the discussion on the paper, E. B. Kirby and P. Argall advocated the use of ordinary iron pipes, lined with pine staves, 1 in. thick. E. Le Neve Foster said that the action of corrosive mine water is greater on wrought-iron than on cast-iron, and that the action is greater, the higher the pressure.—A. S.

Passive Iron. J. S. de Benneville. Iron and Steel Institute, Autumn Meeting, 1897.

After a lengthy historical account of the researches on this subject and a discussion of them, the author mentions the following theories of explanation:—(1.) The oxide theory of Faraday and Schönbein. This is the most important, but the objections to it are action by shock, complete solution on activity, and hence failure to prove the insolubility of the oxide, and recovered activity on long standing in copper sulphate. (2.) The insoluble nitrate theory advanced by Braconnot. (3.) Gaseous theory of Mousson and Varenne. This does not explain the activity brought about by solutions of greater strength than those which maintain passivity. (4.) Tomlinson's mechanical theory, against which can be advanced Cailletet's experiments on the effect of diminishing pressure, also the action of strong nitric acid on heating, the passivity induced by contact, the effect of dilution of silver nitrate solutions, the activity induced by shock, and the effect of position in the circuit on passivity. (5.) The magnetic or electrical state theory of Wetzlar and Herschel, which does not conflict with the existence of allotropic iron. (6.) The "altered" or allotropic condition of the iron proposed by Keir and by Senderens.

The passive state being due to an allotropic change in the iron is based on the reaction with copper sulphate solution, and on the instability of the conditions brought about by chemical treatment of the iron. In the present paper an attempt is made to show that previous heat or mechanical treatment has not affected this reaction in the case of mild steel. β and γ iron do not influence, nor can they be identified with iron "altered" by treatment with strong nitric acid. Such change in the iron includes not only allotropic change, but the formation of oxide as shown by the specific gravities and by the volume of hydrogen obtained on solution in dilute sulphuric acid; chemical reactions, however, necessitate the presence of

allotropic iron. Passivity as induced by silver nitrate solutions differs in degree only from that induced by strong nitric acid.—A. W.

Blast-furnace Slag, Titanium Cyanide Nitride in. Léon Franck. Chem. Zeit. 1897, **21**, [52], 520.

Among the substances formed in the glowing interior of a blast furnace which had been for years uninterruptedly at work, the author found well-marked cubes of titanium cyanide nitride. These had a specific gravity of 5·28, were infusible at a high temperature, and insoluble in boiling nitric or sulphuric acid. On ignition in presence of steam, they yielded hydrocyanic acid, ammonia, and hydrogen, and left a residue of titanium oxide. They dissolved in a mixture of nitric and hydrofluoric acids, with the exception of a small amount, which, on treatment with an oxidising mixture, left a residue of about 2·5 mgrms. This residue had a specific gravity of 3·1 to 3·4 (in methylene iodide), was hard enough to scratch the ruby, and when ignited in oxygen left a very minute amount of ash, which, examined micro-chemically, showed all the properties of rutile. Hence the conclusion was arrived at that the specimen of titanium cyanide nitride contained a minute quantity of crystalline carbon with all the properties of the diamond, together with more or less titanium oxide in a different form of combination.—C. A. M.

Tetraphenyl Methane. M. Gomberg. Ber. **30**, [14], 2043—2047.

Triphenylbromomethane in ethereal solution gives, with phenylhydrazine, an almost quantitative yield of triphenylmethane hydrazobenzene $(C_6H_5)_3C.NH.NH.C_6H_5$, which readily oxidises to the more stable corresponding azo-compound $(C_6H_5)_3C.N:N.C_6H_5$. This, when heated to 110°, gives off nitrogen, and from the residue is separated a white crystalline body, the analysis of which and its molecular weight, determined by the freezing point of its solution in naphthalene, agree with the formula $C(C_6H_5)_4$. The substance melts at 267° C., is soluble in hot benzene, but not in ether, acetic acid, or petroleum spirit.—J. T. D.

New Books.

Chemiker-Kalendar, 1898. Von Dr. Rudolf Biedermann. 19ter Jahrgang. Mit einer Beilage. Julius Springer, Berlin, N. H. Grevel and Co., 33, King Street, Covent Garden, London. 1898. Price 4s. 6d. post free.

The 19th edition of the *Chemiker-Kalendar*, arranged in pocket-book form, has now appeared. It is strongly bound in cloth, contains almanack for 1898, and a table of frequently used atomic weights, and their multiples. A Chronological Table is added of the principal events in the history of the Sciences, more especially of Chemistry. A Note-book Calendar is also supplied, with blank pages for the different days of the year, wherein notes, data, &c may be recorded. Tables next follow of specific gravities of Gases and Liquids, Salts and Alkaloids. Tables also of solubilities and properties of Inorganic Chemical Substances; another set being devoted to similar data for Organic Substances. The remainder of the Pocket-book is filled with tabulated data, &c. on the subjects of Qualitative and Quantitative Chemical Analysis, Volumetric Analysis, Spectrum Analysis, Gas Analysis, and Mineralogy.

The Supplementary Volume, or "Beilage zum Chemiker-Kalendar, 1898," is a separate Pocket-book in paper covers, more especially designed for Technologists. After a series of data, statistics, &c. of mathematical, physical, and general interest, the following groups of subjects receive special attention:—I. Water. II. Assaying. III. Fuels. IV. Sulphuric Acid. V. Alkali. VI. Bleaching Powder. VII. Nitre and Explosives. VIII. Ceramic Arts. IX. Glass. X. Mortar and Cement. XI. Illuminating Gas. XII. Starch and Sugar. XIII. Fermentation Industries. XIV. Fats and Oils. XV. Tannins. XVI. Textile Fibres and Paper. XVII. Photography. XVIII. Colours and Dyes. XIX. Physiological-Chemical Analysis. A series of useful recipes follows, and this concludes the work.

The Analyst's Laboratory Companion. A Collection of Tables and Data for the Use of Public and General Analysts, Agricultural, Brewers', and Works' Chemists, and Students. By Alfred E. Johnson, F.I.C., &c. Second edition, enlarged and improved. J. and A. Churchill, 7, Great Marlborough Street, London. 1897. Price 5s.

8vo volume, containing Preface, Table of Contents, blank ruled sheet, 96 pages of subject-matter, alphabetical index, and eight blank ruled pages for notes. Interspersed throughout the body of the work also, there are similar blank ruled pages for the record of notes.

The work, as indicated in the title, is mainly a collection of tables and formulæ for analysts. They are as follows:—Atomic Weights. Notes on Indicators. Precipitating Powers of Common Reagents. Molecular Weights and Percentage Composition of commonly occurring Compounds. Weight of 1 litre of various Gases. Multipliers and their Logarithms required in Gravimetric and Volumetric Analysis. Notes on and Table of Logarithms. Useful factors, &c. Signs used in Medical Prescriptions. Useful Data. Tables required in Water Analysis, &c. Barometric Tables. Correction of Volumes of Gases for Temperature. Tension of Mercury Vapour. Tables for Beer Analysis. Specific Rotatory Power of the Carbohydrates; also their Cupric Reducing Powers. Phosphate Table. Ammonia and Albuminoid Table. Kjeldahl Process. Quinine. Chicory in Coffee, estimation of. Quinine Sulphate. Baume's Hydrometer. Alcohol Tables, &c. Butter and Milk Analysis. Glycerin. Correction of Standard Solutions for Temperature.

The Principles and Practice of Brewing. By Walter J. Sykes, M.D., F.I.C., &c., Editor of the *Analyst*. With plate and numerous illustrations. Chas. Griffin and Co., Ltd., Exeter Street, Strand, London. Price 21s.

This work is dedicated to Dr. Emil. Chr. Hansen. It contains 495 pages of subject-matter, list of principal books consulted, filling one page, and appendices, six pages. There are also the preface and alphabetical index, and the work is illustrated with 72 wood engravings.

The text is sub-divided as follows:—Part I.—i. Physical Principles involved in Brewing Operations. ii. Chemistry, with special reference to the Materials used in Brewing. Part II.—iii. The Microscope. iv. Vegetable Biology. v. Fermentation. Part III.—vi. Water. vii. Barley and Malting. viii. Brewery Plant. ix. Brewing. x. Beer and its Diseases. Bibliography. Appendix A. Solution Weight and Solution Factor. B. Specific Rotatory Power. C. The Law of Definite Relation. D. Alcoholic Fermentation without Yeast Cells. E. Fermentation in a Vacuum. On page 42, is a handsome plate illustrating the arrangement of the atoms of a molecule in space.

Some North American Coniferæ, being a series of papers reprinted from the *American Journal of Pharmacy*, January 1896 to July 1897. By Edson S. Bastin and Henry Trimble. *American Journal of Pharmacy*, Philadelphia, U.S.A. 1897.

This work is in pamphlet form and of 8vo size. It contains preface and 124 pages of subject-matter, and is illustrated with 58 wood engravings and two plates, one illustrating the *Pinus Strobus L.* and the other *A branch of Tsuga Canadensis*. The further extension of the work was brought to an untimely end through the death of Prof. E. S. Bastin. The principal points in the contents are as follows:—Classification and Distribution. Chemical Composition. Economics. Varieties of Pinus. Physiology of the Resins. Turpentine Industry. Tar. General Characters of the Spruces. Varieties of Spruce, &c. Abies, General Characters, &c. Tsuga, General Characters, &c. Hemlock Tannins.

Trade Report.

TARIFF CHANGES AND CUSTOMS REGULATIONS.

(*From the Board of Trade Journal.*)

New Customs Tariff of Canada.

(*Continued from page 836.*)

Schedule B.—*Free Goods*—(*continued*).

No.	Article.
522	Firebricks, for use in processes of manufacture, or for manufacturing purposes.
528	Foot grease, being the refuse of cotton seed after the oil has been pressed out, but not when treated with alkalis: and grease, rough, the refuse of animal fat, for the manufacture of soap and oils only.
531	Gums, viz.:—Amber, Arabic, Australian, copal, damar, elemy, Kauric, mastic, sandarac, Senegal, shellac; and white shellac in gum or flake, for manufacturing purposes, and gum tragacanth, gum gedda and gum barbery.
536	Hides and skins, raw, dry or wet, and raw pelts.
547	Lampblack and ivory black.
550	Lime juice, crude only.
556	Nickel; and ores of metals of all kinds; and silex or crystallised quartz.
558	Oils, viz.:—Cocoanut and palm, in their natural state; and carbolic or heavy oil; oil of roses and ottar or attar of roses, and olive oil for manufacturing soap or tobacco, or for canning fish.
559	Oil cake and meal, cotton cake and meal, and palm-nut cake and meal.
561	Oleostearine and degras.
564	Platinum wire and platinum in bars, strips, sheets, or plates; platinum retorts, pans, condensers, tubing, and pipe, when imported by manufacturers of sulphuric acid for use in their works in the manufacture or concentration of sulphuric acid.
565	Potash, muriatic and bichromate of, crude, caustic potash, and red and yellow prussiate of potash; also pot and pearl ash, in packages of not less than 25 lb. weight.
568	Quicksilver.
570	Rags, paper waste, and any waste other than mineral.
573	Rubber and gutta-percha, crude, caoutchouc or india-rubber, unmanufactured; powdered rubber and rubber waste; hard rubber in sheets, but not further manufactured, and recovered rubber and rubber substitute.
575	Rubber thread, elastic.
579	Salt, imported from the United Kingdom, or any British possession, or imported for the use of the sea or gulf fisheries.
584	Silver, nickel and German, in ingots, blocks, bars, strips, sheets, or plates, unmanufactured.
586	Soda, sulphate of, crude, known as salt cake, barilla, or soda ash, caustic soda; silicate of soda in crystals or in solution; bichromate of soda, nitrate of soda, or cubic nitre, sal soda, sulphide of sodium, nitrate of soda, arseniate, binarseniate, chloride, chlorate, bisulphite, and stannate of soda.
610	Tin, in blocks, pigs, bars, and sheets, tin plates, tin crystals, tin strip waste, and tin foil, tea lead.
618	Turpentine, raw or crude.
621	Ultramarine blue, dry or in pulp.
622	Varnish, black and bright, for ships' purposes.
624	Whiting or whitening, Paris white, and gilder's whiting, *blanc fixe*, and satin white.
632	Yellow metal, in bolts, bars, and for sheathing.
633	Zinc spelter and zinc in blocks, pigs, sheets, and plates; and seamless drawn tubing.
634	Molasses, second process, or molasses derived from the manufacture of "molasses sugar," testing by polariscope less than 35 degrees, when imported by manufacturers of blacking, for use in their own factories, in the manufacture of blacking, conditional that the importers shall, in addition to making oath at the time of entry that such molasses is imported for such use and will not be used for any other purpose, cause such molasses to be at once mixed in a proper tank made for the purpose with at least one-fifth of the quantity thereof of cod or other oil, whereby such molasses may be rendered unfit for any other use, such mixing to be done in the presence of a Customs officer at the expense of the importer, and under such further regulations as are from time to time considered necessary in the interest and for the protection of the revenue, and that until such mixing is done and duly certified on the face of the entry thereof by such Customs officer the entry shall be held to be incomplete and the molasses subject to the usual rates of duty as when imported for any other purpose.

Schedule C.—Prohibited Goods.

639	Oleomargarine, butterine, or other butter substitute.

(For Schedule D, see this Journal, June 1897, p. 573.)

In addition to the above schedules regulating the import duties, the following Act respecting export duties was also assented to on the 29th June last:—

2. The Governor in Council may, by proclamation published in the *Canada Gazette*, impose export duties, as under, upon the following ores and metals, and each such duty shall be chargeable accordingly after the publication of such proclamation; and the Governor in Council may, by proclamation published in like manner, from time to time remove and reimpose such export duties:—

(*a.*) On nickel contained in matte or in the ore, or in any crude or partially manufactured state, and upon copper contained in any matte or ore which also contains nickel—when exported from Canada—upon such nickel, an export duty not exceeding 10 cents per lb., and upon such copper, an export duty not exceeding 2 cents per lb.

(*b.*) On ores which contain copper or any metal other than nickel or lead, when exported from Canada, an export duty not exceeding 15 per cent. on the value of the said ores.

(*c.*) On lead ores, and on lead and silver ores, when exported from Canada to a country which imposes an import duty on lead in bars or in the form of pig lead in excess of the import duty on lead contained in lead ores or in lead and silver ores—an export duty on the lead contained in the ores so exported from Canada, to an amount per pound equivalent to such excess.

Canada.

Declarations as to Origin of Goods.

A report, dated 28th October last, has been received at the Board of Trade from the Secretary to the High Commissioner of Canada, stating that a telegram has been received from the Minister of Customs of Canada to the effect that declarations of the origin of goods may now be made before any witness. Prior to the modification of the regulations the declarations were required to be signed before a magistrate, justice of the peace, chamber of commerce, or notary public.

New Argentine Law in Regard to the Sale of Medicines.

Scientific American, Oct. 30, 1897, 275.

The Congress of the Argentine Republic is expected to pass a law creating a national board of health (or Department of Public Health, as it is called officially). The law will become effective in a short time. This board of health will have complete control as to what medicines or compounds shall be allowed upon the Argentine market, as will appear from the following two articles of the law:—

Article 36.—It shall be lawful to sell or to expose for sale in any pharmacy or apothecary's shop or store such specialties or compounds only whose component parts are clearly specified upon a visible part of the package thereof, setting forth also the doses of the active substances contained therein.

Article 37.—The Department of Public Health will authorise the sale of the medicines referred to in Article 36, when the required conditions have been fulfilled, without which authorisation such goods cannot be offered for sale.

Failure to comply with the requirements of these two articles will be punished by a fine of from 100 dols. to 200 dols.

It will therefore become necessary for American manufacturers exporting medicinal compounds and specifics to the Argentine Republic to obtain the required permit from the Department of Public Health, and to state the composition of the medicine on each package.

Changes in the American Patent Law.

Chem. Trade J., Nov. 13, 1897, 312.

Mr. J. C. Chapman, Fel. Chart. Inst. of Patent Agents, calls attention to some important changes in the American patent law which, by an Act of Congress, will come into force on the 1st of January next. Hitherto an inventor could always obtain an American patent if he applied at any time during the life of his English or foreign patent for the same invention, provided such invention had not been

in public use in America for more than two years before his application. This was a most generous law. Under the new Act, however, an inventor will not be able to obtain a United States patent for an invention unless he applies within seven months from the date of his application for his English or other first foreign patent for the same invention. Those who now have English or foreign patents granted for any invention, therefore, have still time to obtain, if they so desire, a United States patent for the same invention; but they must apply before January 1st, 1898, in order to protect the invention under the provisions of the present law. They will lose the right when the new law comes into operation. If, therefore, inventors here have not already applied, and desire protection in America, they must act at once.

EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

Report of the West India Royal Commission, with Subsidiary Report by D. Morris; and Appendices.

Government Blue Book, 1897, 1—171.

This Commission comprised Sir Henry Norman, Sir David Barbour, and Sir Edward Grey, the first-mentioned being the chairman. The report of the Commission is divided into three parts. Part I. deals with general questions affecting the economic conditions of all the West Indian colonies. Part II. deals with each colony in detail, and shows the great difference which exists between their several conditions. Part III. comprises the conclusions drawn from a consideration of the whole.

The total exports, excluding Jamaica, and gold from British Guiana, were 3,945,000*l.*, of which 2,951,000 (75 per cent.) were products of the sugar cane. The following table, furnished to the Commissioners by A. G. Bateman, of the Board of Trade, shows the average prices at which refined and unrefined sugar were imported into the United Kingdom in each of the last 16 years:—

Years.	Refined and Candy.		Unrefined.	
	Lumps or Loaves.	Other Sorts.	Beetroot.	Cane and other Sorts.
	Per Cwt.	Per Cwt.	Per Cwt.	Per Cwt.
1881	*s.* 28·93		*s.* 21·72	
	s.	*s.*	*s.*	*s.*
1882	29·14	28·03	21·15	21·09
1883	27·67	26·73	19·98	20·19
1884	21·79	20·15	14·16	16·41
1885	18·82	17·83	12·95	14·56
1886	17·67	16·34	11·96	13·85
1887	16·33	15·39	11·80	12·53
1888	18·58	17·20	13·05	13·84
1889	20·59	19·44	14·93	16·00
1890	17·22	16·08	12·25	13·33
1891	17·42	16·30	12·71	13·28
1892	17·97	16·83	13·11	13·18
1893	19·35	18·11	14·38	14·20
1894	16·97	15·18	11·21	12·22
1895	14·45	13·05	9·42	10·09
1896	14·75	13·33	10·34	10·85

Although, as shown above, the products of the sugar cane constitute the larger portion of the total exports, yet, in view of the fall in prices of sugar, the Commissioners conclude that cane cultivation is no longer profitable, and neither are there any good grounds for supposing that any marked rise in price is likely to occur in the immediate future; indeed, the prices for 1897 have ranged much below those of any previous year. The depreciation in the value of by-products—molasses and rum—has also contributed appreciably to the present depression.

The depression of the industry is due to the competition of other sugar-producing countries, such as the United States, Argentina, and Egypt, and in a special degree to the competition of beet sugar produced under a system of bounties. It is also affected by high protective tariffs and by the competition of beet sugar, the production of which is specially encouraged by the Governments concerned. The causes of the depression may be considered permanent, insomuch as they are largely due to the policy of foreign countries, and there is no indication that such policy is likely to be abandoned.

The evidence brought forward does not show that the depression arises from extravagance in management, to imperfection in the process of manufacture, or to inadequate supervision consequent on absentee ownership; and the removal of these causes, when existing, would not enable it, generally, to be profitably carried on under present conditions of competition. In many places in the West Indies—Barbados, St. Vincent, Dominica, Antigua, &c.—sugar is not manufactured according to the best and latest processes; but even the estates which have introduced the best machinery suffer from the depression, and the Commissioners consider that improvements would be more general out for the apparently well-founded conviction that even with the assistance of such machinery the working could not be profitably carried on. In places where, owing to local causes, large factories, equipped with the best machinery, cannot be established, it is doubtful if the sugar industry could, under any circumstances, be restored to a condition of permanent prosperity, except, perchance, in localities which enjoy very special advantages in soil, climate, and labour supply.

Owing to the crisis, many of the estates are being and have been abandoned, and the consequent distress among the labouring population will render it impossible for most of the islands to provide for their own government and administration without external aid.

In the event of the production of sugar being discontinued or largely reduced, there are no industries which could replace it in such islands as Barbados, Antigua, and St. Kitts, and be profitably carried on and supply employment for the labouring population. In most of the other islands other industries may be substituted for that of sugar, but only after the lapse of a considerable period and at the cost of much displacement of labour, and consequent suffering. The Commissioners consider that in all colonies where sugar can be replaced by other industries, the colonies in question will be in a much sounder position, both politically and economically.

The best remedy for the present state of affairs would be, if the continental nations could be induced to abandon the bounty system; but taking into consideration the great cheapening of the cost of production of beet sugar and the fact that many countries appear to have singled out the sugar industry as one which ought to be artificially stimulated in various ways, it is not clear that, even if the bounties were abolished, another crisis of a similar character might not arise in the West Indies at a future day. A remedy which was largely insisted on by witnesses interested in the sugar estates, and supported by the chairman, was the imposition of countervailing duties on bounty-fed sugar when imported into the United Kingdom. This course, however, was not approved by the majority of the Commission. The chairman has stated his views separately on this point.

The following are the special remedies which were unanimously recommended:—

1. The settlement of the labouring population on small plots of land as peasant proprietors. 2. The establishment of minor agricultural industries, and the improvement of the system of cultivation, especially in the case of small proprietors. 3. The improvement of the means of communication between the different islands. 4. The encouragement of a trade in fruit with New York, and, possibly, at a future time, with London. 5. The grant of a loan from the Imperial exchequer for the establishment of central factories in Barbados. The remedies in 2, 3, and 4 would amount to 27,000*l.* for 10 years, whilst the last-mentioned would cost 120,000*l.*, and no doubt involves the risk of loss. The smaller islands require a grant of 20,000*l.* a year for five years for ordinary expenditure of an obligatory nature; and immediate grants of 30,000*l.* are necessary for roads in Dominica and St. Vincent, to enable the settlement of the labouring population on the land to be carried out; and 60,000*l.* to clear off the floating debt in some of the smaller islands.

Appendix A comprises a report by Dr. Morris, in which he systematically discusses the islands and their industries.

He recommends the establishment of a department of economic botany and for agricultural instruction in the West Indies, and appends a scheme for this purpose.

Appendix B contains tables of statistics, with illustrative diagrams and a map of the West Indian colonies.—J. L. B.

Erratum.

SULPHATE OF COPPER.

(*This Journal, Oct.* 1897, 838.)

Replying to a special inquiry made by the United Alkali Co., Messrs. Rinau-Kessel and Co., of Cologne, state, in regard to the ruling prices of sulphate of copper during 1896 and 1897, that the average value in 1896 was about 16*l.* per ton, with a fluctuation to 18*l.* 10*s.* during March until June. In 1897 there was a similar average price, *viz.*, 16*l.* 5*s.*, with the exception of January to middle of April, when 17*l.* 10*s.* to 19*l.* was paid. The figures quoted from Foreign Office Report No. 1977 are therefore clearly wrong.

SWITZERLAND.

Tariff Decisions.

A despatch has been received at the Foreign Office from H.M. Minister at Berne, forwarding the following list of articles not previously mentioned in the Swiss tariff, but which are now included :—

No.	Article.	Rate of Duty.	
			Francs.
13	Extract of the kola nut	Per 100 kilos.	10·00
15	Nitrate of silver	"	8·00

UNITED STATES.

Tariff Decisions.

Phthalic anhydride or phthalic acid anhydrous is assessed for duty at 20 per cent. *ad valorem*, under paragraph 19 of the Tariff Act of October 1, 1890.

Tetrachlorphthalic anhydride is not an acid, and is dutiable at 20 per cent. *ad valorem* under paragraph 19 of the Tariff Act of 1890.

COMMERCIAL AGREEMENT WITH BULGARIA.

Bd. of Trade J., Nov. 1897, 514.

By an annex to a commercial agreement between Great Britain and Bulgaria, which was signed at Vienna in July last, the import duties on the goods specified below are not to exceed the rates scheduled as follows:—

1. Copper, in ingots, plates, and sheets, 10 per cent. *ad valorem.*

2. Caustic soda and soda of all kinds; potash: alums of all kinds; carbonate of ammonia: sal ammoniac: spirit of salt of ammonia and sulphate of ammonia; green and blue vitriol, 12 per cent. *ad valorem.*

PRODUCTION OF TARTARIC MATERIALS IN ITALY.

Bd. of Trade J., Nov. 1897, 591.

Mr. E. Neville Rolfe, H.M. Consul at Naples, in a report to the Foreign Office, dated 14th October, says that the total production of tartaric materials in Italy for the 12 months ended 30th September 1897, amounted to 9,342 tons, of a value of 366,500*l.* The exports to the United Kingdom were 2,988½ tons; to the United States, 3,470½ tons; to Germany, 509 tons; to France, 216 tons; and to other countries, 2,158 tons.

CALCIUM CARBIDE.

U.S. Cons. Reps., Nov. 1897, 331.

The Aluminium-Industrie-Actien-Gesellschaft, in Neuhausen, Switzerland, and The Electrochemische Werke G. m. b. H., in Bitterfeld, Germany, manufacture calcium carbide which averages 79 per cent. purity: from this percentage 3,118 kilos. (6,002·9 pounds) of calcium carbide are necessary to produce 1,000 kilos. (0·875 cb. m.*) of acetylene. It is impossible to ascertain the cost of the production of calcium carbide from either of the above-mentioned factories, but taking as a basis the lowest wholesale price of the carbide at 40 mks. per 1,000 kilos., then 1,000 kilos. (0·875 cb. m.) of acetylene will cost 1,247·20 mks.

* 1 cb. m. = 35·316 cb. ft.

It may be further added that, in addition to the above use of calcium carbide, it can be used advantageously in metal refining, especially for reductive purposes in the manufacture of ingot iron.

The following prices are charged by the Electrochemische Werke G. m. b. H., in Bitterfeld: On small orders, 60 mks. per 1,000 kilos.; large orders, 450 mks. per 10,000 kilos., and if a large and steady trade were created, the price would fall to 400 mks. per 10,000 kilos. These prices are for the calcium carbide, loaded in the ship at Hamburg, but do not, however, include packing. The steamship companies demand that the carbide be packed in iron cylinders or drums containing 200 kilos. These cylinders must not only be water-tight, but also encased in a tight box or barrel. The cost of packing is 8 mks. per 200 kilos.

Great difficulty has been heretofore experienced in the shipment of the carbide, but it has recently become easier and probably would become more so were the demand large.

The above-mentioned factory in Bitterfeld is the only one in Germany where calcium carbide has been manufactured to any large extent. They are now constructing a larger branch in Rheinfelden, near the Swiss frontier, where there is a very great water power. This new place will be finished in the early fall. As soon as their dynamos are run by water power their prices will become somewhat lower.

GENERAL TRADE NOTES.

BERLIN TRADE AND INDUSTRY, REPORT FOR 1896.

Ber. über Handel u. Industrie, Berlin, 1896.

METALS (pp. 170—172).—*Tin* further declined in value, rates—varying between 136 and 128 m. per 100 kilos.—sinking until in October they touched 120 m.; and, though the closing prices revived to 128—130 m., the average for the year was the lowest on record. An increased consumption and decreased production—many mines being closed on account of unremunerative prices—have reduced stocks, but a change for the better is believed to be imminent.

The consumption of *copper* increased everywhere, except in America, so that the increased shipments—109,000 tons against 65,000 in 1895—to Europe from that country were rapidly taken up. This notwithstanding, European stocks in sight at the close of the year had shrunk by 11,000 tons. Electrical appliances, in which Berlin does a large trade, the growing brass industry, and copper sulphate for treating vines, were mainly responsible for the improved demand.

Speculative purchases forced Berlin rates for the metal from 93—100 m. up to 117—112 m. in June, and after receding to 104—108 m. in August, they closed at 109—112 m. per 100 kilos., this rise being chiefly due to the improved demand. Mansfeld copper was saleable in increased quantities at equal rates with English and American sorts.

Antimony regulus was in better demand, though the quantity used is small; and rates varied between 68 and 60 m. per 100 kilos., with an upward tendency at the close.

Lead was in good demand throughout the year, and in the last quarter there was a slight general advance in rates due to improved economic conditions in America and the scarcity of local stocks. Except for special purposes, American lead forms the main article in Berlin trade, being usually 4 per cent. to 5 per cent. cheaper than the German grades (23—24 m. against 24—26 m. per 100 kilos.). The outlook is considered favourable, but higher prices are necessary to enable German producers to make at a profit.

Crude Zinc.—The year was a very favourable one, and prices rose steadily, with but slight interruptions, from 32 m. per 100 kilos. in January to 38—39 m. in June. In anticipation of an over-production, cheaper offers were made in the third quarter for October delivery, and, in

fact, prices receded 3 m. transiently in September, only, however, to again rise to 38 m. at the end of the year, the increased production being rapidly taken up by consumers, and makers were well sold forward for delivery in the first quarter of 1897. It is, therefore, assumed that rates will remain firm, and perhaps even advance.

Zinc plate was also in a constantly increasing demand, prices rising from 38·50 m. to 45 m. by June and remaining firm for the rest of the year, in spite of a partial weakening of London quotations and rates for crude zinc. Makers were full of orders.

In the *tinplate industry* prices were forced down by the low offers of English manufacturers. Large contracts had to placed in England, the imports increasing from 1,400 tons in 1895 to 10,400 tons in 1896. In the second half of the year German makers again came on the market, and, the South Wales strike having raised English prices, the outlook for 1897 became more favourable. The consumption of tinplate in Berlin and Brandenburg is steadily growing, but the profits of the wholesale trade are as steadily decreasing.

Drugs (pp. 193—195).—The position of the export trade is satisfactory, owing, it is said, to the commercial treaties. Russia is still Germany's best customer, but the exports to Central and Southern America, as well as Eastern Asia, have increased, though the trade with the United States has declined, as is also the case—from political considerations—with Turkey, Cuba, and the Philippines; whilst the Scandinavian connection is still maintained.

Camphor, the use of which for technical purposes is increasing, has become an object of speculation, and a London syndicate forced prices up from 260 m. to 480 m. per 100 kilos., but they afterwards subsided, and closed at about 275 m.

Quinine suffered a decline in rates from 50 m. to 35 m., through the increased production in Java, which country shipped some 2 million lb. more than in 1895.

Cocaïne.—The cultivation is extending in Peru and Bolivia, and semi-purified cocaïne is now exported. Germany received some 2,000 kilos. of crude cocaïne and 5,000 bales of coca leaves. The pure drug declined in price from 520 m. to 410 m. per kilo.

Fish Liver Oil.—The poor catch in the cod fishery caused prices to go up from 130 m. to 200 m. per 100 kilos., but the restricted consumption and the introduction of inferior oils into the market produced a relapse to 160 m.

Lactose, though extending in use for infants' food, is not made so largely in Germany as formerly, America being now a large exporter, instead of buyer, of the article.

Edible olive oil rose 10 per cent. in price in consequence of the poor and late crop and the increased demand for preserving the unusually large catch of sardines.

A reduction of 20 per cent. was made in the price of *salicylic acid* to compete with foreign producers and prevent the erection of new factories.

Spirits followed the rapid rise in the price of potatoes, but receded on the crop being found to exceed expectations.

Vanilla held its ground against the competition of vanillin, without any serious depreciation of values.

Dyestuffs (pp. 196—197):—*Indigo.*—Notwithstanding the large crop (162,200 factory maunds) in the Bengal Provinces, the anticipations of lower prices were not realised, and early purchase of Oudh and Benares indigo showed an advance of 10 and 30 rupees per maund, 340 r. being paid for good marks. However, values subsequently receded to from 20—25 r. above 1895 averages. Notwithstanding the rise in exchange the continent continued to import largely, and although a good deal of 1895 stock was left over, trade was brisk, especially when unfavourable reports were spread with regard to the new crop, until it was found that the production would reach 142,000 maunds, whereupon stagnation set in. The quality of the 236,200 bazaar maunds produced in the Madras Provinces was inferior, and prices ruled lower than for Calcutta sorts. Java produced 680,000 kilos. (76,000 kilos. increase) of good quality, and the crop turned out better than that of India. The Guatemala crop was small, and the high price restricted consumption.

Dyewoods.—An excess of imports caused prices to fall, especially towards the end of the year. Supplies of good Laguna-Campeachy logwood were scanty, but medium and inferior grades were in greater quantity than usual, as was also the case with Yucatan and Campeachy sorts, shipments of these latter mostly turning out well, particularly the Cuyo and pure Yucatan qualities. Domingo and Haiti logwoods are out of favour in this market. Saint Marc turned out satisfactory, and Aux-Layes supplied good quality, Montechristi roots being better known than usual. Jamaica trunks were scarce, but roots enjoyed a good share of attention.

Fustic.—Shipments from Savanilla and Carthagena were short, and from Maracaibo altogether lacking. Corinto exported but very little, and prices might have recovered, but for the large consignments from Mexico (Vera Cruz).

Redwood.—The shipments from Vera Cruz greatly increased, and prices ruled low.

Quebracho.—Prices fluctuated until summer, but then became firmer. Higher freights, large contracts for Argentina, and the more settled state of the trade after the rejection of the Quebracho tariff by the Reichstag, acted favourably on this article.

Dyewood Extracts.—A large turnover was again done, but prices fell in sympathy with those of the dyewoods.

Catechu.—A few large contracts were made, but prices receded at the close of the year.

A large turnover was done in *terra japonica* blocks and cubes, prices falling in summer, so that closing quotations were lower than at the end of 1895.

Myrabolams.—The low rates of 1895 experienced a further declension. Little trade was done, and only very recently has the market hardened, in consequence of epidemics in the producing centres, and higher freights and exchange.

Petroleum (pp. 197—198).—The imports of refined oil into Berlin totalled 72,733,468 kilos., an increase of about 3,700,000 kilos. for the year, and almost equal to the figures for 1894; and as 2,800 barrels of naphtha and benzine were also received, the total consumption amounted to about 401,270 barrels of 180 kilos. gross. It is thus evident that the competition of the incandescent gas light has little effect on the petroleum trade. Prices fluctuated from 22·50 m. per 100 kilos. in January, to a minimum of 19·40 m. in April—May, rising again steadily to 22·20 m. by the middle of November, but afterwards receding to 20·80 m.

Three-fourths of the total oil used was "water-white," but sundry complaints arose with regard to the burning quality.

Favourable reports were received from consumers on the mixtures of Russian and American oils introduced as an experiment.

Bones and other Fertilisers (pp. 198—199).—The price of *bones*, after falling, early in the year, to a level seldom attained, remained steady, and a good business was done.

Fertilisers again receded in value through the action of agricultural associations, keen competition, and increased production.

Bone meal was in small request, and prices fell to about half the rates current a few years back; and had it not been for the increased value of bone gelatin, many works would have had to close.

Superphosphate.—Although the dividends paid by producing companies in 1895 only averaged 3·75 per cent., prices experienced a further decline, owing to the competition of Thomas-slag meal, coupled with the increased production of superphosphate, in order to consume the large amounts of sulphuric acid lacking use, on account of the substitution of electrical processes in the manufacture of soda, chlorine, &c. Ocean freights falling caused a better inquiry for Florida phosphate as raw material, at the end of the year, and the market weakened once more.

The consumption of Thomas-phosphate meal remained at its old figure.

The large shipments and low offers of *nitrate of soda* depressed values of all nitrogenous manures. Prices ranged between 7·50 and 8·50 m. per centner, free Hamburg, and 535,250 tons were received at that port and Harburg.

Extracted Bone Fat.—The demand was low, and prices fell to below 30 m. per 100 kilos., without prospect of recovery.

Bone Gelatin.—A good demand prevailed, both at home and abroad, throughout the year, and prices improved considerably.

Degelatinised bone meal sold at 5—5½ m. per 100 kilos., chiefly for shipment to Sweden and England, whilst *undegelatinised* meal, selling at about 7 m. per 100 kilos., was difficult to dispose of, owing to foreign competition.

Hornmeal, Bloodmeal, Fleshmeal, and Leathermeal all suffered from the competition of mineral nitrogenous manures, and met with merely a restricted application.

Oils and Fats (pp. 199—290).—The official figures of imports to Berlin were:—Olive and edible oil, 86,540 kilos.; denatured olive oil, 841,685 kilos.; linseed oil, 1,177,856; cotton-seed oil, 794,581; palm- and cocoa-nut oil, 4,866,658; rape and other oils, 171,272; fish oil and blubber, 180,688; tallow and animal fats, 2,543,814 kilos.; all—except the first two items and palm oil—being inferior in quantity to the previous year's trade. Business was dull and prices weaker throughout.

Linseed oil was chiefly derived from the Harburg and Breslau crushers, the supplies drawn from England and Holland being much smaller than formerly. Prices for good, clear oil ranged from 52—42 m. per 100 kilos.

Cotton-seed oil was shipped largely from England and America, the very white grades from the latter country increasing in favour. Rates for pale, double-refined varied from 45 to 39 m.; extra pale, 2 to 4 m. higher.

Hemp oil is now scarcely ever used. Prices, 70—67 m.

Olive Oil.—This was chiefly the cheap Levant oil, Spanish oil not being offered later than autumn, owing to a deficient crop. Prime Smyrna was quoted at 69 m., falling to 62 m. and recovering 1 m. later: Malaga, 66—63 m.; green sulphur oil, 48—45—42 m.

Palm oil receded in sympathy with tallow, but short supplies in autumn sent rates up again. Prime Lagos, after falling from 50 to 43 m., recovering to 49—48 m.

Imports of *cocoa-nut oil* are small, the Harburg and Magdeburg coprah crushers being able to meet the demand. Cochin oil cost 64—63 m., and inland coprah oil 53—51 m.

Palm-kernel oil, also home-crushed, ranged from 47 to 42 m.

Tallow was dull throughout the year, the large stocks of 1895 shipments in London preventing any rise beyond a transitory advance in autumn due to speculative dealings. Inland candle tallow fell from 58 to 48 m., and soap tallow from 50 to 40 m.; Australian from 50—56 m. to 42—48 m.; and the small quantity of Russian lubricating tallow imported, from 90 m. to 80 m.

Olein touched very low prices, owing to the cheapness of tallow and the slack demands of the textile industry. Prices ruled: for saponified olein, 54—48 m.; distilled, 38—41, 47—51 m.; St. Petersburg Newsky, 51—48 m.

Fish oils rose in price until June, the better news from the seal fishery and the slow demand then causing a relapse. Greenland seal oil, 48—49—47 m.; Berger liver oil, 50—51—48—44 m.

Fats were plentiful, and prices declined, natural bone fat falling from 43 to 35 m., benzine fat from 36 to 30 m., and fuller's grease from 34 to 28 m., for medium qualities.

Mineral Oils.—The supplies from America and Russia were regular, at prices varying from 19 to 28 m. for low grades, 30—42 m. for medium, and 50 to 80 m. for heavy cylinder oils.

The Berlin Chemical Industry (pp. 201—207).—In general, the 1896 trade was equal to that of the previous year, the volume of business being even greater. As a result of restrictive tariffs the export of tartar emetic to Austria, and of salicylic acid, salts of bromine and iodine, &c. to Italy, was prejudicially affected, and attempts were made in vain to procure a reduction in the Russian duty on lithopone. In the export of aniline colours to Spain, German makers are still at a disadvantage as compared with French and Swiss producers.

The chemical industry continues to be hampered by the conditions of the regulations affecting the granting of permission to found and extend industrial establishments.

Nitrogenous raw materials were all depressed, the market being overloaded. *Sulphate of ammonia* continued to fall until the price touched about 15 m. per 100 kilos. at works, owing to the unusual activity of the coke factories (necessitated by the demands of the iron industry) and the large stocks of the article in England. *Ammonium carbonate* also receded in price to 60 m., and *ammonia* was unable to resist the downward tendency, technically pure (sp. gr. 0·910) ammonia declining from an initial value of 30 m. to a closing price of 24·50 m. Increased competition forced white crystalline *salammoniac* down to 50 m. per 100 kilos.

A brisk trade was done in 96—98 per cent. *potash*, and prices remained firm, the quotations for Stassfurt crude potash salts contributing thereto. The same applies to *oxalic acid* and *oxalates*, owing to the limitation of output by the international associations.

Chlorate of potash fell to the record low price of below 70 m. per 100 kilos., as a result of new cheap electrolytic methods of production.

Calcined Glauber salt was in very active demand, and the large stocks were all cleared out, a rise in values being the result. In *soda crystals* the competition of makers brought prices down to a rate barely remunerative for many works, since calcined soda remained unchanged.

Water-glass (Alkali Silicate).—A continuance of over-production caused the suspension of two works. Home consumption was normal, but the increased Russian tariff has killed the former large trade with that country, and Austro-Hungary buys less than formerly, the high duties having encouraged the erection of works for producing this article.

In *sulphuric acid* and *nitric acid* there was a large trade at but barely remunerative prices, and little profit was made on *hydrochloric acid*.

Methyl Alcohol.—Increased competition forced prices down. A new undertaking made large forward sales at low rates and thus unsettled the market, but at the end of the year was not yet in a position to turn out any merchantable product. The expected rise in the price of *calcium acetate* in America was not realised, and consequently *acetic acid* quotations fell still further.

Carbon bisulphide was in such good demand that there was difficulty in meeting all requirements. Values rose in sympathy with sulphur.

Carbon tetrachloride first came into prominence during the past year, and, being innocuous, has a satisfactory future before it, since the price is capable of reduction by the better utilisation of the by-products.

Sulphur chloride was in regular demand at firm prices, and the same applies to all preparations of *manganese*, such as the crystallised dehydrated chloride.

Liquid carbondioxide had an increased consumption among beer retailers and aerated water makers, the volume of trade in Berlin being 11 per cent. better than in the previous year. The exports from Berlin to northern countries diminished through the competition of makers in the Baltic ports, and the same effect was produced in Austria and Russia by high tariffs. The Rhineland monopolises the trade with western countries, owing to the cheaper and better natural dioxide produced there, and the cheap water carriage.

The favourable situation of the *tar industry* was maintained, great activity prevailing in all branches, with satisfactory financial results, and an improvement in the rates for oils for painting and steeping purposes.

The *benzene* industry also pursued an unexpected course, the demand, for colour making, being so large that, notwithstanding the considerable extension of the production, prices rose from 50 m. at the outset to 120 m. per 100 kilos. by autumn. Although values have since receded to 80 m., it is hoped that the increasing demand will keep the price up to a moderately high level.

Toluene, xylene, and solvent *naphtha* followed, to some extent, the rise in benzene; and the *phenols*, particularly *naphthalene*, also rose in price. On the other hand,

anthracene suffered a serious decline in value, English stocks being still large, despite the large shipments of this article to Germany.

Pyridine remained unchanged until the close of the year, but then jumped up 25 per cent. in value, through a report that the English Government intended to sanction the pyridine process for denaturing spirit.

Intermediate Products for the Tar Colour Industry.—The consumption equalled that of 1895, but foreign competition kept prices down, though the raw materials experienced a rise in values.

Aniline Oil and Aniline Salt.—A good demand prevailed at prices proportionate to that of benzene.

Business was active in the *tar colour industry*, but the high price of benzene on the one hand and low selling prices on the other reduced profits. The unsettled state of the currency question and tariff in America, and the famine and plague in India, restricted the exports to those countries.

Pigments and Mineral Colours.—The trade with Russia, Austria, and Switzerland was crippled by restrictive tariffs. Japanese trade did not improve, and that with the United States declined.

The conditions were also unfavourable in the *varnish* trade, English firms being able, by reason of the low German tariff, to rule the market in varnishes and raw material, especially in copal, although most of this material originates from German colonies. It is therefore considered advisable to attempt to secure the sale and shipment of copal to German ports direct.

Unfavourable conditions prevailed in the *fat-extracting industry* by reason of apathy in the consuming industries and of the greatly increased shipments of Australian tallow and Indian linseed. This reduced the price of palm-kernel oil from 44 m. to $38\frac{1}{2}$ m. per 100 kilos. *ex* Hamburg, and of linseed oil from 48 m. to 37 m. *ex* Hamburg. The large stocks and slackness of inquiry for feeding cakes and meals (*linseed cake, palm-kernel meal*) kept prices low, but a revival set in after the unfavourable potato and fodder harvests.

Cerasin.—The Galician ozokerite monopoly influenced this industry, and the increased price of the raw material (from 28—30 fl. to 34—36 fl. per 100 kilos. *ex* mine) led to the formation of a syndicate of German cerasin makers to raise the inland rate for the manufactured article.

Pharmaceutical and Photographic Chemicals.—A continuance of the favourable reaction that set in during 1895 is recorded.

Ether and alcohol remained fairly steady until the unfavourable potato crop sent prices up considerably in autumn.

The use of *sulphuric ether* in the textile industry experienced an extension.

Bromine and Bromine Salts.—In the former the associated producers in Stassfurt did not introduce any alteration in prices, but the competition among the manufacturers of bromine salts sent the profits on the latter down considerably.

Borax and Boracic Acid.—The volume of trade was satisfactory, but no settlement of prices could be arrived at, the associated producers having continually to face new competitors.

Carbolic Acid.—In anticipation of large State contracts, English prices for 39°—40° crystallised rose from $6\frac{1}{4}d.$ to $8\frac{1}{4}d.$ per lb., but afterwards receded to $7\frac{1}{2}d.$

Diphtheria Antitoxin.—The increased favour enjoyed by German serum was evidenced by the larger exports to England and America.

Iodine and Iodine Preparations.—In the latter articles the influence of the Association of European producers increased, but the associated iodine producers were embarrassed by the competition of shipments of crude iodine from extra-European lands, and the growing production in Japan crippled the export trade to that country.

Glycerin.—After a long period of settled prices, the unusually large purchases by America for 1896 delivery and the improved demand for dynamite-glycerin gave an impetus to rates, which was maintained until autumn, when, in consequence of accumulated stocks and the competition of foreign refiners, the market weakened.

Lactic acid (technical, 50 per cent.) found increased employment for textile purposes as a substitute for oxalic and tartaric acids.

In *tannin*, prices remained unchanged, owing to the great scarcity of gall nuts.

The use of *formalin* is extending, as is also the case with the preparation (*glutol*) of this substance and gelatin for the rapid healing of wounds.

The new industry (in Berlin) of *photographic materials* developed satisfactorily, the dry plates and papers made there being largely used.

Raw and Dyed Silks (pp. 214—215).—A bad year has to be reported in both branches. The demand for silk for passementerie and fancy goods was small, and the prices current early in the year, owing to the unfavourable weather for the cultivation of the silk worm, had subsequently to give way on the realisation of a good crop, and now rule unusually low. Chappe silk moved downwards in sympathy; but tussah, being produced only in small quantities, found willing buyers at normal rates. Sewing silks experienced unfavourable conditions, being thrown on the market in quantities, in excess of the demand, at the lowest conceivable prices.

The average quotations for raw silks for the year, per 100 kilos., in Crefeld, less 5 per cent. for cash, or nett at nine months' date, were as follows:—*Italian:* organzine, 18/20, 42·33 m.; trame, 24/26, $36\frac{1}{2}$ m.; grege, 12/14, $36\frac{1}{2}$ m. *Japanese:* organzine, 22/24, 39·33 m.; tram, 34/40, $37\frac{1}{2}$ m. *Chinese:* tram, 36/40, 33·67 m.; these rates being from 1 to 4 m. below the 1895 average.—C. S.

Production of Beer in Japan.

Bd. of Trade J., Nov. 1897, 604.

The director and manager of the Osaka Beer Brewery Company, of Osaka, Japan, has, according to the *Journal of Commerce of Victoria*, informed an American journal that the beer production of his country is about 100,000 barrels per annum. The demand is extending, and his mission to the States was to supervise the manufacture of a new plant for brewing lager beer on the American system, and he intimated that, although he learned the brewing of lager in Bavaria, and has hitherto used that method, he finds American beer preferable. Further, that he proposes to use Japan and American barley and Californian hops. Australasian hops do not appear to have received his attention. Incidentally it was mentioned that the consumption of "saka," the native drink, made from rice, is as much as 5,000,000 barrels annually.

Export of Medicinal Tinctures.

Chem. and Druggist, Nov. 20, 1897, 801.

During the first half of the present financial year—from April 1 to September 30 last—exporters of tinctures, medicinal spirits, &c., have had repaid to them the duty and allowances on 31,994 proof gallons of spirits exported in these articles, as against 25,619 proof gallons during the same period of 1896. This very substantial increase of 6,375 proof gallons would seem to indicate considerable activity in this section of the export drug trade during the six months in question.

Chinese Quicksilver.

Chem. and Druggist, Nov. 20, 1897, 801.

The richest deposits of quicksilver known in the world are in the province of Kwei-Chow, in South-western China. Until now this province has been closed to foreign trade, but, by a treaty between Great Britain and China, which took effect on June 4 last, the West River, which taps this region, was thrown open to commerce for all nations, and on the evening of the same day the steamer "Wing Tong," of the China Navigation Company, left Hong Kong for a trading journey up the West River.

Imported Chemicals in Japan.

Chem. and Druggist, Nov. 13, 1897, 750.

The Bureau of Commerce of Japan supplies us with the following data concerning the import of chemicals. Ger-

many has it all her own way in the important branch of aniline dyes. It was so in 1893, when the order stood thus:—

Germany.	Switzerland.	France.	United Kingdom.	Belgium.	Holland.
Yen.	Yen.	Yen.	Yen.	Yen.	Yen.
332,422	40,943	14,959	8,393	6,523	1,800

It is so now. Germany, indeed, has improved her position, and increased her already considerable output in greater proportion than her competitors. Englishmen, the inventors of aniline dyes, have meantime fallen off and given up the fourth place on the list to Holland, which really means Germany, as aniline dyes are not made in Holland, but only shipped through Dutch ports. Here are the latest figures:—

Germany.	Switzerland.	France.	Holland.	United Kingdom.	Belgium.
Yen.	Yen.	Yen.	Yen.	Yen.	Yen.
592,945	59,533	21,654	4,050	2,280	1,543

In alizarin dyes Germany conducts practically the whole of the trade. France commenced a slight trade in 1894. The same holds good with alcohol, except that Great Britain is this time the slight competitor. In paint in oil Great Britain led in 1893, and has increased her lead in a striking manner. She now does practically the whole of the trade. Great Britain still leads in chlorate of potash. Germany and France have, however, doubled their output somewhat at her cost. The latest figures are:—

	Yen.
Great Britain	366,850
Germany	41,996
France	10,202

In salicylic acid Germany does all the business. Great Britain and France are simply nowhere. Great Britain is safe in the manufacture of amorphous phosphorus. France just tries to compete:—

	Yen.
Great Britain	248,983
France	2,315

The same holds good in caustic soda. America entered into competition in 1893, while Germany fell out:—

	Yen.
Great Britain	331,147
America	2,486
Germany	66

Bicarbonate of soda comes from Great Britain to the value of 102,917 yen. So with quinine. Great Britain sends it to the value of 129,391 yen. The total value of carbolic acid imported amounts to 176,190 yen, chiefly from England. Bismuth subnitrate comes from Germany to the value of 108,459 yen. The total value of liquid gold amounts to 110,522 yen. This is chiefly from Great Britain.

Australian Kieselguhr.

Chem. and Druggist, Nov. 13, 1897, 749.

Kieselguhr of varying quality is obtained at several places in Victoria, one of the best grades coming from Avoca. It is shipped to Britain and Germany. It has been recommended to the Queensland Railway Commissioners for packing strong acids, which the Queensland Government will not carry on the railway in small quantities by ordinary trains. The suggestion that kieselguhr should be used for absorbing acids, so that they could be carried without being packed in jars, has been tried, but has not been found to answer in practice. The acids absorb water from the atmosphere so rapidly that impregnated kieselguhr gets converted from an almost dry powder to a pulp.

Petroleum in Newfoundland.

Imp. Inst. J., Nov. 1897, 338.

In a recent number of the *Imperial Institute Journal* (Vol. III., p. 154) a short account was given of the mineral wealth of Newfoundland, in which it was stated that petroleum had been found on the west coast of the island, but no particulars were given of the extend and quality of the supply. The following details are furnished by an article in *The Canadian Engineer* for September (p. 125):—

The oil-producing district appears to extend over an area of about 250 square miles, and, in geological formation consists of limestone, and conglomerates thereof, shales, and sandstones.

An analysis of the crude oil, gathered from surface depressions, gave the following results:—

	Per Cent.
Specific gravity	0·885
Water	Traces.
Gasoline	None.
Naphtha	"
Illuminating oil	14·5
Lubricating oil	82·5
Solid residue	3·0
Sulphur	0·068

The oil obtained from a depth of 1,000 ft. was, however much lighter, and yielded a higher percentage of illuminating oil. The crude oil was dark olive green in colour, of aromatic odour, and had a specific gravity of 0·844 at 60° F. On distillation it yielded:—

	Per Cent.
Naphtha	7
Illuminating oil	56
Lubricating oil	34
Coke	3

The products of distillation have a pleasant odour and are very easy to refine. The illuminating oil is water-white, of high flashing point, and burns with a brilliant flame. The crude oil is very free from grit and other foreign substances, and, consequently, the heavy oil, after suitable treatment, makes a very good lubricant. Tha quality of the oil compares very favourably with the best American oils.

All the wells hitherto sunk belong, with one exception, to the Newfoundland Oil Company, with whom an English company is stated to be negotiating for the acquisition of the property. These wells are close to the sea, and only six to ten miles of pipe will be required to convey the oil to the water.

The Indian Cinchona Plantations.

Imp. Inst. J., Nov. 1897, 338.

The reports of the Madras and Bengal Governments on their respective cinchona plantations have recently been issued. The salient features of these reports are mentioned in articles in the *Chemist and Druggist* (October 2nd, 1897), and the *Indian Pharmacologist* (September 1897). In the case of the Madras plantations, there has been an increase of 47,257 rupees over the officially estimated revenue of 100,000 rupees. This increase is said to be mainly due to improvements in the extraction of the alkaloid quinine, introduced by the superintendent, Mr. Standen, whereby the yield of sulphate of quinine has been increased from 1·5 per cent. to 3·3 per cent. The subjoined table shows the yield of cinchona products for the past five years:—

—	1892-93.	1893-94.	1894-95.	1895-96.	1896-97.
	Lb.	Lb.	Lb.	Lb.	Lb.
Bark worked	171,717	175,000	196,600	323,800	238,100
Yield of quinine	4,933	2,000	4,700	3,600	7,891
" febrifuge	3,139	5,775	1,756	2,284	3,360
" total alkaloid.	8,072	7,775	6,526	5,884	11,241

The Indian Government does not compete in the matter of sale of bark in the European market; all that is produced is sold to the Indian Medical Stores, or to native customers; quinine at 18 rupees, and febrifuge at 16 rupees per lb. A considerable quantity, in packet form, is disposed of to the native populations, being thus sold by native postmasters, at 1 pice each packet. The amount distributed in this way in 1896 was 2,000 lb.

In the Bengal cinchona plantations, a similar progress is reported. The stock of febrifuge-yielding trees (red bark) is being gradually decreased in favour of quinine-yielding trees. There are now in the Bengal cinchona gardens, 2,683,451 trees, of which only 185,000 are febrifuge-yielding.

The year's crop of dry bark amounted to 679,222 lb., while the quinine produced was 10,672 lb., and febrifuge, 3,452 lb.

The net profit on the year's working was 9,767 rupees, as against 4,598 rupees in the preceding year.

The amount of quinine sold in pice packets in this province during the year was 3,330 lb., an increase of 130 lb. over the previous year.

All restrictions on the sale of quinine to other governments and administrations have now been removed, and the superintendent has been instructed to comply with any demands he may receive.

Indian Hemp Products.

Imp. Inst. J., Nov. 1897, 337.

In a "Supplementary Note" to the Excise Board of India recently made by Dr. Prain, of the Royal Botanic Gardens, Sitpur, Calcutta, some interesting information is given as to the method of storing hemp products in order to prevent the deterioration which always takes place after they have been kept for two or three years.

The various forms of Indian hemp are exciseable articles in India, and their storage in bond has occasionally been a source of loss to the Government.

It had already been suggested by Dr. Pain that storage in hermetically sealed tins might overcome the difficulty, but in an experimental batch so treated it was found that the water present in the hemp had produced fermentation, and the "ganja," as the pressed tops of the hemp plant are called, had developed "a strong rancid odour," and had become "quite unfit for smoking."

It has now been found that if the "ganja" be kept in dry deal perforated boxes, it can be stored for two or three years without fermenting or losing its narcotic properties, and Dr Prain recommends this method of preservation to the Excise Board. Appended to this supplementary report is a translation of a paper by Mr. L. Lapin, of the Dorpat Pharmaceutical Institute, who has recently investigated the Indian hemp plant with a view to the isolation of the true active constituent. He has obtained a substance, called "Cannabindon" by him, which appears to have all the physiological properties of the plant. From the description of this substance given by Mr. Lapin, it seems to be identical with the "cannabinol" obtained by a much simpler process from "charas" (Indian hemp resin) by Messrs. Wood, Spivey, and Easterfield (Imp. Inst. J., March 1897). It is very probable that the various substances which have from time to time been regarded as the active constituents of Indian hemp may have been merely impure specimens of "Cannabinol," and this is borne out by the fact that the various preparations of Indian hemp which have been generally regarded as physiologically active are either purified alcoholic or ethereal extracts of the drug, as the following tabular arrangement of the best known of these preparations shows:—

Name of Preparation.	Character of Preparation.	Name of Investigator.
Cannabin............	Alcoholic extract of the previously purified drug.	Smith.
Cannabin............	(Method of preparation not known.)	Merck.
Cannabin............	Alcoholic extract of previously washed drug.	Decourtive.
Cannabinon..........	Purified alcoholic extract.	Fischer.
[illegible]..........	(Method of preparation not known.)	Merck.
Haschisch-purum.....	Alcoholic extract purified by treatment with alkaline solutions.	..
[illegible] tannicum	(Method of preparation not known.)	Merck.
Cannabindon.........	Purified ethereal extract..	Lapin.
Hasch[illegible]...........	Purified alcoholic extract.	..

Dr. Prain, however, regards it as unlikely that an extremely stable substance such as "cannabinol" should be the active constituent of a drug which so readily loses its physiological properties. The matter is one which still requires further investigation.

Production of Quicksilver in Russia.

Eng. and Mining J., Oct. 23, 1897, 490.

The production of quicksilver at the mines of A. Auerbach and Co., near the Nikitowka station (on the Kursk-Kharkov Railway) in the Bachmut district of the government of Ekaterinoslav, from the beginning has been as follows:—

Year.	Average Yield of Ore.	Production of Metal.	Year.	Average Yield of Ore.	Production of Metal.
	Per Cent.	Kilos.		Per Cent.	Kilos.
1887	1·00	64.062	1893	0·46	200,990
1888	0·76	164,815	1894	0·46	195,987
1889	0·51	167,109	1895	0·69	434,070
1890	0·73	292,137	1896	0·71	491,465
1891	0·80	323,865	*1897	0·79	406,224
1892	0·65	342,768			

* To Oct. 1st, 1897.

These are the only quicksilver mines in Russia. The metallurgical practice at the works, which employ the Auerbach improved Schernia furnaces, is very good, and ore yielding 0·4 per cent. of quicksilver is said to cover the cost of production.

The Nothberg Coke-Oven Plant.

Eng. and Mining J., Oct. 22, 1897, 484.

On a visit by the members of the Aachen section of the Verein Deutscher Ingenieure to the Nothberg Colliery, owned by the Eschweiler Bergwerks Verein, Herr Welcke read a paper on the coke-oven plant of that colliery, observing that two years ago the company was induced to put up a bank of 60 ovens, with plant for recovering the by-products, on account of their affording the advantages which are thus summarised by the *Colliery Guardian.* (1.) Higher yield from the small coal charged in, with equal quality of coke, while the coking is effected with complete exclusion of air. (2.) Considerably higher output per oven, on account of the larger charge of small coal, from which the gases are soon driven off by uniform heating by means of gas from the outside; and (3.) Recovery of the valuable by-products ammonia, tar, and benzol, from the gases of the small coal. Against these advantages, however, must be mentioned the greater first cost and higher current expenses, with a smaller amount of heat available for firing the boilers, although the advantages predominate, so that by far the largest number of new ovens are arranged for by-product recovery. In the discussion which followed the reading of this paper, Herr Othberg stated the reasons why two different types of oven had been adopted at Nothberg. On the one hand, it was considered advisable, while recovering the by-products, to utilise the gases as far as possible for firing the boilers, and from this standpoint the Ruppert oven was preferred. On the other hand, however, the question arose of employing, in addition to the Nothberg bituminous coal, the non-bituminous of the Anna Colliery, and from the latter standpoint the Otto oven recommended itself, because permitting the use of coals containing little gas, as well as the recovery of the by-products.

The Supply of Asbestos.

Engineering; through Imp. Inst. J., Nov. 1897, 339.

The French manufacturers of asbestos goods are supplied from four sources:—(1.) Canada, whence the asbestos is white, silky, very unctuous, having supple fibres from 5 to 25 mm. in length; of all varieties it is that which spins the most easily. (2.) Siberia, whence the mineral is yellowish, some species being of a straw yellow. The fibre is less flexible, and more woody, but stronger, than the Canadian,

which it resembles in length; large masses of long-fibre mineral being, however, rarer. (3.) The Cape of Good Hope asbestos has a characteristic blue colour. It occurs in larger masses than either the Canadian or Russian, and its fibre is generally longer and stronger. In spinning or other manipulation, however it is difficult to handle, a good deal of the fibre being reduced to powder. (4.) In Italy there are different kinds of asbestos, but generally they are little adapted to spinning. There are some long silky fibres of little resistance, employed for gas furnaces; others are very short and fit only to make heat-retaining coverings.

Platinum in Ontario.

Eng. and Mining J., Oct. 23, 1897, 484.

The *Canadian Mining Review* reports the discovery in this province of a deposit of sand carrying considerable quantities of platinum and an unusually large proportion of osmium and iridium.

English Arsenic.

Chem. and Druggist, Nov. 6, 1897, 736.

The output of arsenic in England is being gradually reduced as the quantity of tin diminishes. In Cornwall last year (1896) 1,366 tons of arsenious acid, or white oxide of arsenic, were produced, the value of which at the mines was estimated at 14,997*l.* Devonshire produced, in 1896, 2,250 tons of arsenic, the value being 30,186*l.* On the whole, therefore, the output has fallen from 4,798 tons, of the value of 52,198*l.*, last year, to 3,616 tons, value 45,183*l.*, this year. On the other hand, the quantity of arsenical pyrites extracted under the Metalliferous Minerals Act and sold for the manufacture of white arsenic has made an extraordinary leap. In 1895 only 2,951 tons of this mineral were sold in the two southern counties, fetching the sum of 2,785*l.* In 1896 the value of the 8,808 tons of pyrites was no less than 8,007*l.*, an increase in one year's output of 5,222*l.* With the exception of the three years 1883–85, however, the 1895 production was the smallest on record. Indeed, the outputs have varied enormously in different years. For instance, in 1891 the returns showed that 14,321 tons were extracted, and in 1892, 12,564 tons. This was followed by the production of under 2,000 tons in each of the subsequent three years.

BOARD OF TRADE RETURNS.

Summary of Imports.

Articles.	Month ending 31st October	
	1896.	1897.
	£	£
Metals	1,708,863	1,853,430
Chemicals and dyestuffs	404,570	329,470
Oils	709 394	735,969
Raw materials for non-textile industries.	4,548,091	5,241,185
Total value of all imports	39,574,830	38,943,763

Summary of Exports.

Articles.	Month ending 31st October	
	1896.	1897.
	£	£
Metals (other than machinery)	3,139,132	3,053,570
Chemicals and medicines	651,412	708,696
Miscellaneous articles	3,166,584	2,909,626
Total value of all exports	20,683,457	19,283,452

Imports of Metals for Month ending 31st October.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
Copper :—			£	£
Ore .... Tons	10,289	10,264	40,375	80,464
Regulus .... „	8,271	9,016	211,780	235,560
Unwrought ..... „	5,572	4,862	268,778	247,260
Iron :—				
Ore .... „	381,553	410,797	263,837	301,223
Bolt, bar, &c. ... „	5,981	7,521	49,182	58,854
Steel, unwrought.. „	1,713	1,844	15,892	20,049
Lead, pig and sheet „	13,532	14,124	144,649	185,269
Pyrites .... „	78,276	53,330	63,761	90,516
Quicksilver .... Lb.	9,218	25,510	922	2,350
Silver ore....... Value £	..	..	115,186	155,508
Tin .... Cwt.	75,547	58,372	222,137	181,093
Zinc .... Tons	8,297	5,165	132,867	89,316
Other articles ...Value £	..	..	179,497	206 053
Total value of metals	..	..	1,708,863	1,853,430

Imports of Oils for Month ending 31st October.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Cocoa-nut......... Cwt.	24,130	33,593	26,093	35,218
Olive.............. Tuns	424	1,286	11,678	46,951
Palm .... Cwt.	90,003	102,937	104,038	108,272
Petroleum .... Gall.	17,169,471	19,795,715	316,246	347,869
Seed .... Tons	3,316	2,284	72,943	52,419
Train, &c. .. .. Tuns	2,484	3,314	40,011	48,980
Turpentine .... Cwt.	29,774	11,552	29,802	12,724
Other articles .. Value £	..	..	108,583	83,336
Total value of oils...	..	..	709,394	735,969

Imports of Raw Material for Non-Textile Industries for Month ending 31st October.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Bark, Peruvian .. Cwt.	1,430	2,543	2,288	4,724
Bristles.......... Lb.	372,407	339,298	51,499	37,410
Caoutchouc ...... Cwt.	36,933	35,330	463,135	388,375
Gum :—				
Arabic......... „	1,192	2,755	3,145	5,840
Lac, &c. .... „	5,184	10,590	24,868	35,062
Gutta-percha .... „	4,474	5,060	44,862	61,490
Hides, raw :—				
Dry............ „	31,113	31 889	74,995	72,729
Wet........... „	59,943	59,298	123,164	126,961
Ivory............ „	1,003	949	15,130	39,280
Manure :—				
Guano .... Tons	257	1,312	1,475	5,248
Bones .... „	7,987	5,121	29,418	14,780
Nitrate of soda... „	2,383	3,245	19,058	24,194
Phosphate of lime „	18,448	23,052	25,857	36,540
Paraffin.......... Cwt.	83,692	67,483	79,186	57,880
Linen rags....... Tons	1,579	1,809	16,508	18,359
Esparto.......... „	14,125	12,690	61,446	49,314
Pulp of wood .... „	30,588	33,474	165,423	172,839
Rosin............ Cwt.	35,064	56,103	9,327	14,240
Tallow and stearin „	135,844	106,543	140,779	102,180
Tar ....Barrels	16,809	3,093	13,089	2,163
Wood :—				
Hewn .... Loads	203,177	301,939	410,943	525,603
Sawn .... „	721,412	924,127	1,613,349	2,139,004
Staves......... „	13,494	15,924	56,286	49,087
Mahogany ..... Tons	7,778	7,921	68,641	67,898
Other articles....Value £	..	..	1,044,000	1,180,026
Total value	..	..	4,548,091	5,241,185

Besides the above, drugs to the value of 79,450*l.* were imported, as against 89,152*l.* in October 1896.

Imports of Chemicals and Dyestuffs for Month ending 31st October.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Alkali............ Cwt.	12,024	11,416	8,359	7,161
Bark (tanners', &c.) „	24,611	17,637	7,708	5,400
Brimstone .... „	28,830	34,093	6,387	7,593
Chemicals...... Value £	..	..	116,641	114,434
Cochineal .... Cwt.	1,081	350	7,290	2,063
Cutch and gambier Tons	1,339	1,311	26,645	22,705
Dyes:—				
Alizarin ....Value £	..	..	24,559	17,107
Anilin and other „	..	..	44,555	42,189
Indigo .... Cwt.	1,440	353	17,817	4,623
Nitrate of potash . „	17,714	15,079	14,399	12,115
Valonia .... Tons	1,618	1,732	16,444	17,141
Other articles... Value £	..	..	113,766	76,939
Total value of chemicals	..	..	404,570	329,470

Exports of Metals (other than Machinery) for Month ending 31st October.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Brass............ Cwt.	9,586	8,682	42,485	49,714
Copper:—				
Unwrought.... „	27,706	44,559	68,798	115,576
Wrought ...... „	19,251	31,583	61,003	95,371
Mixed metal... „	27,525	26,060	65,443	61,466
Hardware...... Value £	..	..	199,045	189,576
Implements ..... „	..	..	128,725	115,587
Iron and steel ... Tons	344,615	340,915	2,200,345	2,161,693
Lead .... „	2,808	3,521	35,453	49,524
Plated wares ... Value £	..	..	48,238	47,328
Telegraph wires . „	..	..	162,054	47,746
Tin .... Cwt.	11,567	9,243	36,417	29,976
Zinc .... „	12,274	13,721	9,256	10,293
Other articles .. Value £	..	..	81,867	88,720
Total value	..	..	3,139,132	3,053,570

Exports of Miscellaneous Articles for Month ending 31st October.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Gunpowder...... Lb.	1,008,800	556,900	23,830	14,022
Military stores.. Value £	..	..	244,137	115,754
Candles.......... Lb.	2,066,100	2,138,100	30,400	27,931
Caoutchouc ..... Value £	..	..	116,279	116,959
Cement.......... Tons	32,593	35,512	53,117	59,440
Products of coal Value £	..	..	150,422	143,139
Earthenware ... „	..	..	167,846	149,759
Stoneware ...... „	..	..	15,219	16,372
Glass:—				
Plate........... Sq. Ft.	172,144	146,243	9,916	8,542
Flint........... Cwt.	9,680	9,004	22,514	23,028
Bottles......... „	78,172	80,445	41,102	37,979
Other kinds.... „	24,755	21,803	19,494	16,801
Leather:—				
Unwrought .... „	12,031	16,451	107,964	138,077
Wrought ..... Value £	..	..	41,088	39,390
Seed oil.......... Tons	5,668	4,287	99,925	73,866
Floorcloth ..... Sq. Yds.	1,734,900	1,743,700	75,984	75,839
Painters' materials Val. £	..	..	138,302	136,841
Paper .... Cwt.	100,177	90,189	152,171	132,511
Rags............ Tons	4,660	5,718	25,193	28,199
Soap .... Cwt.	60,374	63,295	63,372	67,253
Total value	..	..	3,166,584	2,909,623

Exports of Drugs and Chemicals for Month ending 31st October.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Alkali........... Cwt.	447,743	483,891	113,849	120,487
Bleaching materials „	104,753	126,310	34,299	39,653
Chemical manures Tons	26,624	34,374	116,635	145,828
Medicines....... Value £	..	..	97,615	106,436
Other articles... „	..	..	289,014	296,292
Total value	..	..	651,412	708,696

Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—PLANT, APPARATUS, and MACHINERY.

Applications.

23,947. R. L. Walker. Improvements in refuse or garbage furnaces. Oct. 18.

24,014. P. E. M. Jamain. Improvements in apparatus for compressing powdered substances. Oct. 18.

24,041. R. G. Ferguson and A. M. Elkus. Improvements in apparatus for mixing liquids with gases. Complete Specification. Oct. 19.

24,084. G. Barker.—From L. Rissmuller, Germany. Centrifugal separators. Oct. 19.

24,201. J. Klein. Improvements in and connected with apparatus for the condensation of steam and vapours. Complete Specification. Oct. 20.

24,227. T. Christie. An apparatus for raising liquid. Oct. 20.

24,342. A. J. Bell and P. A. G. Bell. Improvements in apparatus for the filtration of water, sewage, and other fluids. Oct. 21.

24,698. M. Deville Improved centrifugal apparatus for sorting or separating ores. Complete Specification. Oct. 25.

24,922. G. T. Tugwell. Improvements in apparatus for the production of hydrogen gas. Oct. 27.

24,933. H. H. Lake.—From O. Brzostowicz, Belgium, and R. Nitzsche, Germany. Improvements in carburising apparatus. Oct. 27.

25,084. G. Hawkins, H. Hawkins, and S. H. Hawkins. A new or improved hydrogen gas generator. Oct. 29.

25,265. G. Helps. Improvements in and relating to regenerative furnaces for the heating of gas retorts and the like. Nov. 1.

25,379. G. A. Nussbaum. Improvements in devices for absorbing, dispensing, or conducting heat, applicable also for separating and superheating steam. Nov. 2.

25,611. D. Black. Improvements in filter presses and similar filtering apparatus. Nov. 4.

25,634. E. Theisen. Process and apparatus for absorbing, extracting, distilling, or evaporating liquids and gases or vapours. Nov. 4.

25,654. The Reeves Patent Filters Company, Ltd., and W. Reeves. Improvements in or connected with filtering apparatus. Nov. 4.

25,793. C. Delecroix. New or improved hamper for bottles containing acids, essences, and the like. Nov. 6.

25,867. E. T. Bousfield. Improvements in vaporisers. Nov. 6.

25,898. E. Beckton. A portable spout to attach to drums containing ammonia or other strong or obnoxious chemicals. Nov. 8.

26,002. G. Watson. Improvements in furnaces for the destruction of town refuse. Nov. 9.

26,451. H. A. House, jun., and R. R. Symon. Improvements in apparatus for regulating the supply of combustible liquid or vapour to liquid fuel burners. Nov. 12.

26,525. J. T. Bentley. Improvements in apparatus for cooling liquids. Nov. 13.

Complete Specifications Accepted.*

1896.

27,009. P. H. Rosenkranz. Diaphragms. Oct. 27.

28,289. S. Sherratt and W. A. Richards. Method and apparatus for filtering, washing, and drying crystalline, granular, and other materials. Nov. 3.

29,000. P. J. E. E. Chambost. Apparatus for heating or evaporating by means of hydrocarbon liquids of any density. Nov. 3.

29,318. W. Stagg, R. T. Glover, and J. G. Glover. Pressure or vacuum gauges. Nov. 27.

1897.

2,219. G. Zschocke. Hurdles or grids for gas purifiers and cooling, drying, and graduating apparatus. Nov. 3.

17,099. C. H. Pieper and A. F. Pieper. Rheostats. Oct. 27.

II.—FUEL, GAS, and LIGHT.

Applications.

23,977. W. Johnson. Improvements on apparatus for the production and use of acetylene gas. Oct. 18.

24,292. A. J. Boult.—From L. Meneveaux, France. Improvements in or relating to apparatus for the manufacture of acetylene gas. Oct. 20.

24,301. J. McConechy. Improvements in apparatus for the production of acetylene gas. Oct. 21.

24,328. J. Wilson. Improvements in and in the manufacture of burners for incandescent gas lighting. Oct. 21.

24,435. H. A. Kent. Apparatus for the production of hydrogen gas and air. Oct. 22.

24,488. R. Schlatter. Improvement in acetylene gas generators. Oct. 22.

24,602. G. Mahnke and C. Kraft. An improved means of protection of incandescent gas light mantles. Complete Specification. Oct. 23.

24,611. A. Coze. Improvements in apparatus for charging inclined gas retorts. Oct. 23.

24,707. C. Guy. Improvements in apparatus for the production of acetylene gas. Oct. 25.

24,815. H. Ottenbruch. Improvements in mantles for incandescence gas lighting. Complete Specification. Oct. 26.

24,816. G. Fürbringer. Improvements in apparatus for generating acetylene gas. Oct. 26.

24,822. A. Syssoyeff and L. Braly. Improvements in auto-luminous compositions. Complete Specification. Oct. 26.

24,860. H. Wilson and T. Mitchell. Improvements in or relating to the generation of acetylene and other gases. Oct. 27.

25,002. L. de Ravel. Improvements in apparatus for the production of acetylene gas. Oct. 28.

25,023. W. B. Hartridge. Apparatus for manufacturing perforated fuel blocks. Oct. 28.

25,287. R. Farrington and M. H. Hawes. Improvements in and relating to incandescent gas lighting. Nov. 1.

25,318. J. W. Grainger. Improvements in and relating to the manufacture of fire-lighters. Nov. 1.

25,623. F. J. J. Bagley. Improvements in the manufacture of fire-lighters and fuel. Nov. 4.

25,627. W. R. Bell. An improved method or process and apparatus for the preparation of peat for fuel. Nov. 4.

25,632. R. E. Perraga. Improvements in method and apparatus for generating formaldehyde gas. Complete Specification. Nov. 4.

25,674. S. T. White. *See* Class XI.

25,750. J. Zimmerman. Improvements in gas-producing materials. Complete Specification. Nov. 5.

25,800. J. W. Scarth and W. A. Thornton. Improvements in or relating to lamps for burning acetylene gas. Nov. 6.

25,801. J. W. Scarth and W. A. Thornton. Improved apparatus for the generation of acetylene gas and like purposes. Nov. 6.

25,853. C. J. Lundström. Improvements relating to apparatus for developing acetylene gas. Nov. 6. Date applied for Aug. 6, 1897, being date of application in Sweden.

25,866. A. H. Pöppel. Improvements in apparatus for the manufacture of gas. Nov. 6.

25,870. A. H. Barthez. Improvements in apparatus for the automatic production of acetylene gas. Nov. 6.

25,949. C. Duval. Improvements in apparatus for generating acetylene gas. Nov. 8.

25,952. The Acetylene Light Syndicate, Ltd., and J. Gore. Improvements in apparatus for the manufacture of acetylene gas. Nov. 8.

26,095. M. Leray. Improvements in or relating to apparatus for use in the production of acetylene or similarly produced gas. Filed Nov. 9. Date applied for April 14, 1897, being date of application in France.

26,202. A. Martini. Improvements in and relating to incandescent mantles. Nov. 10.

26,269. G. E. Arkell, J. W. Bailey, and J. Clapham. Improved apparatus for use in the production of acetylene. Nov. 11.

26,311. J. Janz. Improvements in the manufacture of fabrics for incandescence mantles for illuminating purposes. Complete Specification. Nov. 11.

26,325. A. J. Boult.—From F. Alexandre, France. Improvements in or relating to apparatus for the manufacture and use of acetylene gas. Nov. 11.

26,381. A. M. Plaissetty. New incandescent mantle by gas, mineral oils, alcohol, and process of fabrication related thereto. Nov. 12.

26,435. J. Mücke and J. Mücke. Improved apparatus for generating acetylene gas. Complete Specification. Nov. 12.

26,484. C. A. Miller and F. J. Miller. Improvements in acetylene gas generating apparatus, principally for use in connection with cycle lamps and other lighting appliances generally. Nov. 13.

Complete Specifications Accepted.

1896.

26,191. E. T. Zohrab. Production of peat charcoal and apparatus therefor. Oct. 27.

27,212. J. Wetter.—From G. Meyer. Apparatus for generating acetylene gas. Nov. 3.

28,101. J. A. Sinclair. Means for lighting by electricity and incandescence. Nov. 3.

28,206. T. R. Fowler. Acetylene gas generator, purifier, and container. Nov. 17.

29,168. E. Richard-Lagerie. Apparatus for producing acetylene gas. Oct. 27.

29,597. A. Bandsept. Incandescent burners. Oct. 27.

29,603. J. Spurge. Gas-cleansing apparatus. Nov. 3.

29,763. W. H. Dargue. Apparatus for the production, drying, purifying, and storage of acetylene gas. Nov. 3.

1897.

501. R. Langhans. Manufacture of incandescing media for use in incandescent gas lighting. Nov. 17.

512. G. Lebran and F. Coronaille. Apparatus for producing acetylene gas. Nov. 10.

3013. W. P. Thompson.—From P. P. H. Mace and L. L. H. Gerard. Apparatus for the production of acetylene gas. Oct. 27.

3667. H. A. Kent. Inverted regenerated incandescent gas burners or lights. Nov. 17.

11,739. F. R. Foster. Incandescent mantles for gas-burners. Nov. 17.

* *See* Note (*) on previous page.

14,090. E. Jimeno. Apparatus for the production of acetylene. Nov. 10.

19,411. H. J. Bell and The Niagara Falls Acetylene Gas Machine Company, Ltd. Apparatus for production and storage of acetylene gas. Nov. 3.

20,464. A. Chauvel and G. Ménétrier. Manufacture of fuel cakes or blocks for industrial and domestic purposes. Nov. 3.

21,831. H. H. Lake.—From The Wizard Manufacturing Company, United States. Production of acetylene gas, and lamps for generating and burning the same. Oct. 27.

22,120. G. Hilgenstock. Improvements in coke ovens. Nov. 17.

22,730. A. Flöck. Apparatus for producing acetylene gas. Nov. 17.

23,198. F. Bosshardt.—From A. Molet, Argentine Republic. Automatic apparatus entitled "spirit," for the production of acetylene gas. Nov. 17.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Application.

25,501. T. F. Haldane. Improvements in obtaining products in distilling wood. Nov. 3.

IV.—COLOURING MATTERS and DYES.

Applications.

24,287. J. C. Mewburn.—From Chemische Fabrik von Heyden Gesellschaft mit beschränkter Haftung, Germany. Production of oxyphenyl-guanidines and their derivatives. Oct. 20.

24,410. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. The manufacture of new colouring matters of the safranine series. Oct. 21.

24,527. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of black diazo-colouring matters. Oct. 22.

24,644. C. E. Gassmann. A process for obtaining new mordant dyes. Oct. 25.

24,704. A. H. Goldberg, W. Siepermann, and H. Flemming. A process for the production of canarine. Oct. 25.

24,793. J. C. Mewburn.—From Chemische Fabrik von Heyden Gesellschaft mit beschränkter Haftung, Germany. Improvements in the production of phenolic carbonates. Oct. 26.

24,938. S. Pitt.—From L. Cassella and Co., Germany. Producing dyestuffs from amidophenols. Oct. 27.

24,964. I. Levinstein and Levinstein, Ltd. Production of colouring matters from tetrazo compounds of dichlorbenzidine. Oct. 28.

25,136. S. Pitt.—From L. Cassella and Co., Germany. Producing diamido-oxydiphenyl and like bases. Oct. 29.

25,234. S. Pitt.—From L. Cassella and Co., Germany. Production of direct-dyeing black cotton dyestuff. Oct. 30.

25,332. S. M. Angel. Improved dye for textile fabrics, feathers, and the like. Nov. 1.

25,590. W. E. Heys.—From A. Liebmann, Germany. The production of colouring matters on vegetable fibres. Nov 4.

25,756. O. Imray.—From The Fabriques de Produits Chimiques de Thann et Mulhouse, Alsace. Manufacture of para- and meta-nitroanisidine, and of colouring matters therefrom. Nov. 5.

25,787. A. Bang.—From G. A. Dahl, Germany. The production of direct-dyeing blue dyestuffs. Nov. 6.

26,419. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. The manufacture of mordant-dyeing diazo colouring matters. Nov. 12.

26,471. I. Levinstein and Levinstein, Ltd. Production of new sulphonic acids. Nov. 12.

Complete Specifications Accepted.

1896.

23,554. T. A. Myers. Manufacture and packing of blue for laundry purposes, liquid dyes, and other liquids. Oct. 27.

29,706. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of dyestuffs of the series known as patent blue. Oct. 27.

29,827. P. A. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Production of dyestuffs on fibre. Nov 3.

29,828. S. Pitt.—From L. Cassella and Co. Production of a brown colouring matter. Nov. 3.

1897.

765. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Manufacture of colouring matters and of new intermediate products. Nov. 17.

1320. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of alpha-naphthylamine in an easily diazotisable and stable form. Nov. 17.

10,810. J. Hauff. Preparation of orthotoluolsulpho acid and of orthosulpho benzoic acid by oxidation of orthothiocresol. Nov. 10.

19,793. R. Webb. Security inks. Nov. 10.

V.—TEXTILES, COTTON, WOOL, SILK, Etc.

Applications.

24,028. W. G. Heys.—From H. David, France. Improvements in and connected with the treatment of vegetable textile fabrics and materials with mercerising solutions. Oct. 19.

24,468. G. Ormondroyd. Improvements in the method of and apparatus for scouring hanks of yarn. Oct. 22.

24,528. C. Wetherwax. An improved process of treating flax and hemp for obtaining textile fibres therefrom. Oct. 22.

25,468. W. P. Ingham. Improvements in or connected with the manufacture of slag wool. Nov. 3.

26,044. A. J. Boult.—From J. Buffard, France. Improved process and composition for rendering fabrics waterproof. Nov. 9.

26,120. T. C. Taylor and J. E. Tolson. Improvements in apparatus to be employed in carbonising, neutralising, and bleaching fabrics and fibres. Nov. 10.

26,247. W. G. Heys.—From H. David, France. Improvements in and connected with the production of lustrous effects upon vegetable textile materials. Nov. 11.

Complete Specifications Accepted.

1896.

25,362. V. Bona. Manufacture of textile fabrics. Oct. 27.

26,485. J. S. Brown. Process and apparatus for preparing vegetable fibres for textile purposes. Oct. 27.

27,484. H. C. Longsdon. Means or apparatus applicable for use in the drying of wool and other fibrous substances. Oct. 27.

28,613. D. M. Sutherland and W. McLaren. Treating nitro-cellulose for the purpose of coating or impregnating textile and other fabrics. Nov. 3.

29,832. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. *See* Class VI.

1897.

12,541. C. L. Bachelerie. Treatment of textile plants to remove the fibre therefrom. Nov. 3.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

Applications.

24,506. S. Musgrave, H. Musgrave, and J. T. Musgrave. An improved method of and process for obtaining iridescent colours upon paper or other suitable material. Oct. 22.

24,533. C. L. Horack. Method of and apparatus for colouring yarns. Oct. 22. Date applied for April 15, 1897, being date of application in United States.

24,868. J. Stockhausen. *See* Class XII.

25,117. E. Lehmann. An improved process for marbling, mottling, or colouring paper or cardboard on one side in the paper machine. Complete Specification. Oct. 29.

25,162. H. R. Cassel. An improved method or process of bleaching pulp fibres, textile fabrics, and other materials. Oct. 30.

25,542. W. Shedlock. Improvements relating to the bleaching, drying, dyeing, and washing of textile materials and to apparatus therefor. Nov. 3.

25,564. W. H. Thorpe. Improvements in machinery for dyeing, scouring, and bleaching hanks or skeins of yarn. Nov. 4.

25,590. W. E. Heys.—From A. Liebmann, Germany. *See* Class IV.

25,598. H. J. McBride, H. W. McBride, and F. Duffin. Improvements in the process of bleaching linen union or cotton fabrics. Nov. 4.

26,538. A. C. Marot and A. Bonnet. An improved process for mordanting cotton for aniline black. Nov. 13.

Complete Specifications Accepted.

1896.

25,581. E. J. Stewart. Improvements in dyeing. Oct. 27.

27,109. A. Leven. Process for dyeing a thread in different colours. Nov. 17.

29,150. F. A. Gatty and Co., Ltd., and V. H. Gatty. Dyeing wool or other animal fibres in either the raw or manufactured state.

29,832. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning. Production of local, durable silk-like gloss effects on cotton or linen stuffs by means of printing. Nov. 3.

1897.

13,088. H. Zublin and A. Zuigg. White and colour discharge on enlevage printing of dyed para-nitraniline red and related substances. Oct. 27.

19,005. H. Seidel. Mordanting of fibrous materials preparatory to dyeing such materials. Oct. 27.

VII.—ACIDS, ALKALIS, and SALTS.

Applications.

23,916. W. Galbraith. The extraction or recovery of potash and soda salts, and of iodides and bromides from the waste liquors resulting from the extraction of ammonia from blast-furnace gases. Oct. 18.

24,077. A. J. Boult.—From R. Memmo, Italy. Improved manufacture of calcium and other carbides and by-products obtained thereby. Oct. 19.

24,515. C. C. Moore. Improvements in the treatment or purification of brine and other saline solutions. Oct. 22.

24,524. A. MacNab.—From W. A. Willock, India. An improvement in the manufacture of bay salt. Complete Specification. Oct. 22.

24,525. O. Imray.—From W. T. Gibbs, Canada. Improvements in the manufacture of potash salts. Oct. 22.

25,007. H. S. Elworthy. An improved process for the manufacture of carbonic acid and apparatus therefor. Oct. 28.

25,042. E. M. Taylor. Alkali phenate compound for bleaching, scouring, and stripping. Oct. 29.

26,172. M. Goldschmidt. An improved method or process of preparing oxalic acid and oxalates. Nov. 10.

26,176. J. C. Mewburn.—From Chemische Fabrik von Heyden Gesellschaft mit beschränkter Haftung, Germany. Improvements in the production of halogen compounds of silver. Nov. 10.

26,326. D. Lance and R. L. E. de Bourgade. A process for the economic production of cyanides, their derivatives, and other nitrogen compounds. Complete Specification. Filed Nov. 11. Date applied for April 12, 1897, being date of application in France.

Complete Specifications Accepted.

1896.

24,573. C. Hoepfner. Production of chloride of zinc and chlorine. Nov. 10.

28,891. G. Kynoch and Co., Ltd., and A. T. Cocking. Purifying and concentrating sulphuric acid in part applicable to the manufacture of nitric acid. Nov. 3.

1897.

381. A. K. McCosh. *See* Class X.

591. P. J. Worsley and the United Alkali Co., Ltd. Manufacture of caustic soda or caustic potash from carbonate of soda or carbonate of potash. Nov. 10.

17,313. H. J. Krebs. Process and apparatus for distilling ammonia. Nov. 17.

VIII.—GLASS, POTTERY, and ENAMELS.

Applications.

23,933. W. Burton and J. Burton. Improvements in the construction of potters' kilns. Complete Specification. Oct. 18.

24,015. A. J. Boult.—From A. Panigatti, A. Merenda, and F. Passadoro, Italy. Improvements in or relating to the ornamentation of glass. Oct. 18.

24,278. G. L. Siever. Improved process and apparatus for the manufacture of ceramic panels for decorative purposes. Oct. 20.

24,317. E. Swinden. Improved method of and composition for polishing glass. Complete Specification. Oct. 21.

24,505. A. C. Tulloch. Improved method of and means for the drying of china clay. Oct. 22.

24,729. The Chromo-Transfer Company, Ltd., and H. Wallace. An improvement in the decoration of pottery, china, and the like. Oct. 26.

26,384. F. Hancock and C. B. Winzer. Improvements in or applicable to kilns or muffles used for pottery and brick purposes. Nov. 12.

26,401. J. Davis, A. J. Davis, and A. J. Loftus. Improvements in the manufacture of lenses and other articles of ruby-coloured glass, and in appliances to be used in the said manufacture. Nov. 12

26,446. J. Clowes, T. Poole, and A. J. Poole. Improvements in kilns for firing pottery and other goods. Nov. 12.

26,537. A. Macairo and C. Scuvero. An improved process of and apparatus for printing on brittle surfaces, such as glass, crystal, porcelain, and the like. Complete Specification. Nov. 13.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, and CEMENTS.

Applications.

23,993. C. G. J. Frosell. An improved composition applicable for cement, artificial stone, and the like. Oct. 18.

24,179. C. E. Lee and C. F. Lawton. Improvements in the manufacture of cement. Oct. 19.

24,609. L. Brockmann. Improvements in the production of artificial stone. Oct. 23.

24,928. G. E. Gray and T. W. Pallin. An improved process for producing imitations of marble, granite, or other ornamental stones. Oct. 27.

25,685. A. Küpper. Improvements in manufacturing hollow building blocks or artificial stones, and in apparatus employed in their manufacture. Nov. 5.

25,861. D. H. Ferguson. Improvements in material for the construction and decoration of buildings. Complete Specification. Nov. 6.

26,213. L. Tebbutt. Improved removable floor for cement and other kilns. Nov. 11.

26,334. H. H. Lake.—From O. Leproux, France. An improved composition for preserving wood. Nov. 11.

Complete Specifications Accepted.

1896.

25,292. C. Craig and G. K. Craig. Building blocks and method of using same. Oct. 27.

26,306. G. Scott. Concrete building construction. Nov. 3.

27,975. S. R. Thompson. Composition or cement for making pipe and other joints. Oct. 27.

1897.

13,821. G. F. Leboida. Apparatus for drying and hardening wood. Nov. 17.

X.—METALLURGY, MINING, Etc.

Applications.

23,921. A. Gutensohn. Improved process for separating gold and other precious metals from pyritic auriferous ores and the like. Oct. 18.

24,121. W. P. Thompson.—From E. C. Ketchum, United States. Improved process for treating mixed sulphide ores. Complete Specification. Oct. 19.

24,267. P. E. Secrétan. A new or improved alloy of aluminium, and process for making the same. Oct. 20.

24,426. W. L. Wise.—From J. W. Comley, United States. Process and apparatus for welding metals. Oct. 21.

24,490. S. H. Byass. Improved means for facilitating the annealing of metal sheets. Oct. 22.

24,526. O. Imray.—From W. T. Gibbs, Canada. Treatment of zinc ores. Oct. 22.

24,696. B. Mohr. An improvement in the treatment of sulphide ores for separation of zinc therefrom. Oct. 25.

24,875. Sir T. G. F. Hesketh, Baronet. The hardening of copper and other metals by the action of a chemical agent. Oct. 27.

25,080. R. Wootton and G. B. Martin. Improvements in the mode and means for annealing metal. Oct. 29.

25,920. Sir T. G. F. Hesketh. A new method of hardening metals. Nov. 8.

26,030. W. L. Wise.—From J. W. Comley, United States. Process and apparatus for welding metals. Complete Specification. Nov. 9.

26,046. S. Ganelin. Method of treating lead sulphide ores. Complete Specification. Nov. 9.

26,063. A. Reynolds. An improvement in the manufacture of crucible steel. Nov. 9.

26,196. La Compagnie de Métallurgie Générale (Société Anonyme). Process of preparation and treatment of ores other than iron ores. Complete Specification. Filed Nov. 10. Date applied for Aug. 23, 1897, being date of application in Belgium.

26,290. R. Miller and J. Miller. Improvements in and relating to the manufacture of iron. Nov. 11.

26,406. C. H. Alldred. Improvements in the purification of iron and steel. Nov. 12.

Complete Specifications Accepted.

1896.

22,361. J. Armstrong. Extraction of lead, zinc, gold, silver, and other metals from sulphides, refractory or other ores or mattes. Nov. 10.

25,738. C. Hornung. Charging apparatus of blast furnaces. Nov. 3.

26,493. T. B. McGhie and E. G. Ballard. Beds or tests of cupellation furnaces, assay cupels, and the like. Oct. 27.

29,251. R. D. Burnie, and W. T. Lougher. Covering or coating steel and iron with copper or alloy of copper. Nov. 17.

29,709. W. Kirkham. Crucible melting furnaces. Nov. 3.

30,077. J. Burgess and W. A. Brodie. Producing castings in malleable iron, mild steel, and other metals. Nov. 17.

1897.

381. A. K. McCosh. Obtaining cyanides and other products from blast furnaces. Nov. 10.

14,438. W. G. M. Foote. Method of and apparatus for extracting metals from their ores or other compound bodies. Oct. 27.

16,808. F. Ellershausen. Treatment of complex sulphide ores and apparatus therefor. Nov. 3.

18,731. F. George. Process of soldering aluminium. Nov. 10.

19,480. E. H. T. Plant and S. Dellow. Amalgamating pan. Oct. 27.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

Applications.

21,051. R. Fabian. Improvements in electrodes for primary and secondary batteries in chemical cells. Oct. 19.

24,150. H. S. Lloyd. Improvements in electrodes for storage batteries. Complete Specification. Oct. 19.

24,257. F. O. Prince. Improvements in and connected with electric primary batteries. Oct. 20.

24,591. A. Heil. Improvements in galvanic cells. Oct. 23.

24,665. H. Wiggin and Co., Ltd., and G. A. Boeddicker. Improvements in anodes and in the electro-deposition of nickel and other metals. Oct. 25.

24,884. A. F. Harris. Improvements in electro-deposition apparatus. Oct. 27.

24,925. E. Giglio. Improvements in primary batteries. Oct. 27.

25,246. H. von der Linde. A process for the electrolytic separation of metals. Oct. 30.

25,386. H. K. Hess. Improvements in chemical electric generators. Complete Specification. Nov. 2.

25,388. L. Parvillée. Improved compositions for use in the construction of electrical heating apparatus and resistances and the processes for their manufacture. Complete Specification. Nov. 2.

25,400. E. Motz and H. F. Welch. Improved electrolytic process and apparatus for the separation of metals from their ores and solutions. Complete Specification. Nov. 2.

25,441. H. H. Lake.—From G. De Digoine, France. Improvements in electric accumulators. Nov. 2.

25,480. A. Graemiger. Improvements in electric accumulators. Nov. 3.

25,519. J. Hargreaves. Improvements in electrodes. Nov. 3.

25,674. S. T. White. Generation of electricity at gas works by utilising waste gases. Nov. 5.

25,703. W. W. Hanscom and A. Hough. Improvements in electrical batteries. Complete Specification. Nov. 5.

25,757. O. Imray.—From La Société Anonyme pour le Travail Electrique des Metaux, France. Improved manufacture of electrodes for electrical accumulator or secondary batteries. Nov. 5.

25,889. F. C. Geary and J. H. Rowbottom. An improved cell or box suitable for accumulator or other electric batteries. Nov. 8.

25,890. G. Bell and G. W. Bell. Improvements in or connected with electrolytic cells. Nov. 8.

26,067. F. E. Elmore.—Partly communicated by J. O. S. Elmore, India. Improvements in ectrolytical apparatus. Nov. 9.

26,097. R. J. Crowley, C. E. Monkhouse, and A. Smith. Improvements in or relating to galvanic batteries. Nov. 9.

26,441. C. A. Bennert. Improvements in the preparation of accumulator plates Complete Specification. Nov. 12.

26,543. F. E. Elmore.—Partly by J. O. S. Elmore, India. Improvements in apparatus for the generation and electrolytic application of electric currents. Nov. 13.

Complete Specifications Accepted.

1896.

25,701. J. H. May and F. King. Secondary batteries. Nov. 17.

26,059. C. C. Connor. Electrodes. Nov. 17.

27,167. C. E. O. Keenan. Electrical batteries. Nov. 10.

28,314. R. Kennedy. Electrical storage batteries and cells. Nov. 17.

28,511. L. Lucas. Manufacture of electric accumulators or secondary batteries. Oct. 27.

28,764. F. Hurter, and the United Alkali Co., Ltd. Apparatus for use in electrolysis, more especially intended for use in the electrolysis of zinc chloride. Nov. 3.

29,115. F. King. Electric storage or secondary batteries. Oct. 27.

1897.

425. F. King. Secondary batteries. Nov. 17.

10,439. C. E. Lee. Secondary batteries. Oct. 27.

18,430. F. Pescetto. Manufacture of electric accumulators. Nov. 10.

22,041. R. W. James.—From The Bell Electric Company, United States. Electric batteries, electrodes, and method of making same. Nov. 17.

22,045. R. W. James.—From The Bell Electric Company, United States. Electric batteries. Nov. 17.

XII.—FATS, OILS, AND SOAP.

Applications.

24,217. J. Peacock. Improved manufacture or production of soap. Oct. 20.

24,438. W. J. Engledue. Improvements in purifying and clarifying oils. Oct. 22.

24,773. E. Wilczynski. The manufacture of an improved soap. Oct. 26.

24,779. G. H. Evans. Combined Soap. Complete Specification. Oct. 26.

24,868. J. Stockhausen. Improvements in the manufacture of soap for use in dyeing and finishing textile fabrics and materials, and for other purposes. Complete Specification. Oct. 27.

25,505. H. Harris and H. Harris, Sons, and Co., Ltd. Improvements in the manufacture of soap tablets. Complete Specification. Nov. 3.

25,570. J. Sebaschl and H. Hinterberger. Apparatus for purifying oily fluids and from separating the water therefrom. Nov. 4.

26,137. W. E. Woodyer. Improvements in and relating to soap. Nov. 10.

26,191. C. Culmann. An improved process for deodorising oils and fatty substances. Complete Specification. Nov. 10.

Complete Specifications Accepted.

1896.

26,396. R. Gesell. Process for making soap. Nov. 17.

27,906. F. W. Wright and The United Alkali Company, Ltd. Manufacture and production of dry soap or soap powder. Oct. 27.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

23,957. F. J. Bergmann. Process for the production of graphite. Complete Specification. Oct. 18.

24,178. C. E. Lee, C. F. Lawton, A. L. Lawton, and A. W. Lawton. Improvements in the manufacture of paint. Oct. 19.

24,504. A. Stoop. An improved process for the manufacture of paints and pigments. Complete Specification Oct. 22.

26,065. A. J. Smith. Improvements in the manufacture of varnish Nov. 9.

26,107. H. Markus and G. B. Behrens, of the firm of D. Lee and Co. Improvements in and in the manufacture of india-rubber and gutta-percha. Complete Specification. Nov. 10.

26,268. W. N. Hird. Improved fire-resisting paint. Nov. 11.

Complete Specifications Accepted.

1897.

18,533. D. J. Ogilvy. Method of making printing inks. Oct. 27.

20,902. W. Walters and A. Webb. Patent leather harness enamel. Oct. 27.

XIV.—TANNING, LEATHER, GLUE, AND SIZE.

Applications.

24,080. A. J. Boult.—From G. Brigalant, France. Improvements in or relating to the treatment of leather waste and in apparatus employed therein. Complete Specification. Oct. 19.

24,083. A. J. Boult.—From G. Brigalant, France. Improvements in or relating to the manufacture of boards, plates, or sheets from leather pulp. Complete Specification. Oct. 19.

24,294. A. S. Bury and T. A. Brown. Improvements in machines for mixing and agitating size. Oct. 20.

25,008. H. Streich. Improvements in the preparation of leather. Complete Specification. Oct. 28.

25,324. P. Leyendecker. An improved machine for treating skins and hides. Nov. 1.

25,670. W. E. Walker and J. Longshaw. Improvements in and connected with tannin, and allied processes. Nov. 5.

25,871. H. H. Lake.—From C. Dreher, Germany. A process for waterproofing leather and rendering the same impervious to aqueous solutions. Nov. 6.

26,077. J. Birtwisle. Improvements in tanning. Nov. 9.

Complete Specifications Accepted.

1896.

23,742. J. T. McQuinn. Tanning hides, skins, and the like. Nov. 3.

1897.

20,125. J. Mossop, jun., and E. B. Garland. Tanning hides and skins. Oct. 27.

21,601. E. H. Dewson. Tanning of hides and skins and the like, and apparatus employed therein.

XVI.—SUGARS, STARCHES, GUMS, Etc.

Applications.

24,333. H. Machin, of the firm of Batger and Co.—From E. A. Dix, Russia. Improvements in or relating to the manufacture of sugar and in apparatus therefor. Oct. 21.

24,593. D. Loiseau. Improvements in the processes of purifying and clarifying sugary products in sugar-houses, sugar, and molasses refineries, refineries, or factories of glucose and the like. Oct. 23.

25,752. A. Classen. Process for obtaining dextrin from starch and from formalindextrin. Nov. 5.

25,753. A. Classen. Process for obtaining directly from starch a compound of formaldehyde with dextrin soluble in water. Nov. 5.

25,860. M. Sachs. Improvements in crystallising vessels for crystallising sugar. Nov. 6.

26,418. A. Classen. Manufacture of albuminous substance soluble in water and processes therefor. Nov. 12.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

23,938. J. Boulton, J. F. Jackson, and J. Ward. An improved process for the distillation of alcoholic spirits. Oct. 18.

25,020. J. Blauenfeldt and H. Tvede. A process for drying and preserving yeast. Oct. 28.

25,140. C. Ameye. A process and apparatus for sterilising and saturating with carbonic acid gas liquids contained in casks or like vessels. Complete Specification. Oct. 29.

25,996. B. J. Atterbury. Improvement in the production of alcohols. Nov. 9.

26,106. L. P. Ford. Improvements in apparatus for brewing. Nov. 10.

26,520. G. Delory. Improvements in the manufacture of beer. Complete Specification. Nov. 13.

26,532. J. F. H. Gronwald. Improvements in and relating to preserving beer. Nov. 13.

Complete Specifications Accepted.

1897.

15,200. A. Fraser and Maclay and Co., Ltd. Manufacture of pale ale. Oct. 27.

18,713. D. J. Etty and C. R. Long. Process of and apparatus for ageing whisky. Oct. 27.

23,249. J. D. Møller. Improving or oxidising alcoholic beverages. Nov. 17.

XVIII.—FOODS, SANITATION, Etc., AND DISINFECTANTS.

Applications.

A.—Foods.

24,172. The Aerating Citroid Syndicate, Ltd., and G. Bamberg. Improved process of preparing soluble food in solid form. Oct. 19.

25,153. A. T. Perkins. Improvements in process and apparatus for the conservation of fruit and other perishable products. Complete Specification. Oct. 29.

26,292. R. R. Bell and A. C. Butler. Improvements in and relating to tea essences. Nov. 11.

B.—Sanitation.

23,913. J. Rudman and H. J. G. Rudman. New or improved process for the effective removal of noxious and other vapours, and appliances for use in connection therewith. Oct. 18.

25,259. J. Grossman. Improved method of treating sewage. Nov. 1.

26,069. G. E. Gale. System of apparatus for treating garbage. Nov. 9.

C.—Disinfectants.

26,250. J. H. G. Winckler.—From G. A. Spranger, Germany. An improved proceeding for making antiseptic ozone preparations. Nov. 11.

Complete Specifications Accepted.

A.—Foods.

1897.

20,013. E. G. N. Salenius. Method and apparatus for sterilising milk and other liquids. Oct. 27.

B.—Sanitation.

1896.

24,833. B. Walker. The Walker process for treating sewage sludge. Nov. 3.

C.—Disinfectants.

1896.

29,288. W. B. Bottomley. Material for use in the treatment and purification of sewage and other impure waters. Nov. 3.

XIX.—PAPER, PASTEBOARD, Etc.

Application.

24,560. F. Cox, F. Kornacher, and M. Brumm. An improved writing paper, and ink therefor. Complete Specification. Oct. 23.

Complete Specification Accepted.

1896.

26,045. C. Marter. Artificial ivory. Oct. 27.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

Applications.

24,704. A. H. Goldberg, W. Siepermann, and H. Flemming. A process for the production of canarine. Oct. 25.

24,891. E. C. C. Stanford. An improved process for separating and obtaining the active constituents of the thyroid gland of the sheep and other animals. Oct. 27.

25,942. A. Classen. Manufacture of formaldehyde compounds of the proteïn bodies soluble in water. Nov. 8.

25,945. W. P. Thompson.—From J. Brissonnet, France. Improved creosote compound. Complete Specification. Nov. 8.

26,303. E. J. Boake. An improvement in the treatment of essences, liquid drugs, and the like for facilitating their transport. Nov. 11.

26,442. W. G. Whiffen. Improvements in the preparation of a crystalline compound of emetine. Nov. 12.

Complete Specifications Accepted.

1896.

25,778. H. Helbing. Preparation and application of menthol. Oct. 27.

29,446. O. Imray.—From H. Hoffmann, La Roche and Co. Production of certain organic therapeutic preparations. Nov. 3.

1897.

656. J. Y. Johnson.—From C. F. Boehringer and Soehne. Preparation of alkylated uric acids. Nov. 17.

17,693. L. Lederer. Preparation of hexymethylentetramin and its derivatives.

XXI.—PHOTOGRAPHY.

Applications.

23,917. R. W. Sharp. Appliance for photographing by acetylene light. Oct. 18.

24,021. A. G. C. Hunter. Improvements in and connected with photographic dry plates, films, and the like. Oct. 19.

24,763. W. H. Fellows. Improvements in or relating to photography. Oct. 26.

24,942. T. Raab. Method of, and means for obviating marginal cloudiness in photographic pictures on films. Oct. 27.

25,433. W. J. Holt. Improvements in the obtainment of coloured reproductions or prints from photographs. Nov. 2.

25,933. A. Schwarz. A new or improved process for the treatment of sensitised photographic paper. Nov. 8.

26,122. C. E. Benham. Benham's ink process of photography. Nov. 10.

XXII.—EXPLOSIVES, MATCHES, Etc.

Applications.

24,136. A. Luck and C. L. Nichols. Improvements in the manufacture and production of explosives. Oct. 19.

24,195. L. Braly. Compositions suitable for the manufacture of matches. Complete Specification. Date applied for, March 30, 1897, being date of application in France. Oct. 20.

24,385. G. Beneké. Improvements in the waterproofing of blasting cartridges and new waterproofing compounds suitable therefor. Oct. 21.

25,204. J. C. Chapman.—From C. W. Volney, United States. An improved manufacture of gunpowder. Complete Specification. Oct. 30.

25,413. J. C. Chapman.—From C. W. Volney, United States. An improved gunpowder. Complete Specification. Nov. 2.

25,806. O. Hohnrodt. An improved fog-signal for railways. Nov. 6.

26,174. C. E. Bichel. An improved process for the preparation of nitro-glycerin explosives. Nov. 10.

Complete Specifications Accepted.

1896.

25,704. A. E. Pike and W. H. Thew. Manufacture of explosives or explosive compounds. Nov. 17.

26,819. T. Jenkins. Charges of detonating railway fog-signals. Oct. 27.

XXIII.—ANALYTICAL CHEMISTRY.

Complete Specification Accepted.

1897.

23,321. F. Hugershoff. Centrifugal machines for examining and testing milk, sediments of urine, and other fluid substances. Nov. 17.

PATENTS UNCLASSIFIABLE.

Application.

24,085. G. Barker.—From L. Rissmuller, Germany. Process of and apparatus for the treatment of waste matters. Complete Specification. Oct. 19.

Complete Specifications Accepted.

1897.

8227. G. L. Schaefer. Improvements in chemical compounds. Nov. 17.

23,126. A. J. Boult.—From A. O. Richter, Germany. Compound for use in extinguishing fires. Nov. 17.

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NOTICES.

Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, half of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows:—

To Members (see Rules 25 and 27) and Past Members (see Rule 30); Libraries, Corporations, and Exchanges on the Society's List	Each copy	10s.
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Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. Eyre and Spottiswoode, the Society's printers and publishers, to whom all communications respecting them should be addressed. The circulation of the Journal is now more than 3,000 per month.

Members are reminded that the subscription of 25s. for 1898, payable on January 1st, 1898, should be sent in good time to the Treasurer, in order to ensure continuity in the receipt of the Society's Journal. Any changes of address to appear in the new List of Members now in course of preparation, should reach the General Secretary not later than January 15th next.

Telegrams.

The code address of Palace Chambers is "Palatable, London," and the Society's number is 59. Hence telegrams need only be addressed "59, Palatable, London," without words "Society of Chemical Industry."

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Wülfing, Dr. C. F., l/o North Woolwich; The Woodlands, Maryon Road, Old Charlton, Kent.

Deaths.

Bowrey, J. J., Kingston, Jamaica. Nov. 19th.
Field, S. E., Lewin's Mead Brewery, Bristol.
Hammersley, W. A. L., Leek, Staffordshire. Dec. 26th.
King, Jas. E., 2, Dover Road, Birkdale, Southport. Oct. 18th.
Rawcliffe, Harold, Gillibrand Hall, Chorley. Dec. 18th.

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John Heron, 110, Fenchurch Street, E.C.

SESSION 1897-98.

Monday, Jan. 3rd, 1898:—
Prof. H. R. Procter and Dr. J. Gordon Parker. "Standard Methods for Analysis of Tanning Materials."
Dr. J. Gordon Parker. "The Effect of Different Temperatures on the Extraction of Tanning Materials."
Messrs. J. H. Coste and E. J. Parry. "Neatsfoot Oil."
Monday, Feb. 7th, 1898:—
Messrs. A. R. Ling and J. Baker. "Clerget's Method of Estimating Sugar, and a New Modification of it, specially applicable to After-Products and Molasses."
Mr. Thos. Fletcher. "Note on an Improved Drip-proof Bunsen."
Mr. Eug. Ackermann. "Notes on Cement."

Meeting held on Monday, December 6th, 1897.

DR. R. MESSEL IN THE CHAIR.

THE SULMAN-TEED (BROMO-CYANIDE) PROCESS OF GOLD EXTRACTION.

BY H. LIVINGSTONE SULMAN AND FRANK L. TEED.

The reaction upon which our gold extraction process is based was described in a paper read by one of us before the Institution of Mining and Metallurgy in 1895. At that time we were, however, unable to give any results beyond those obtainable in large laboratory practice, the largest ore extractions then available being from some 2 cwt. up to 1 ton.

Faraday and also Prince Pierre Bagration (Jour. prakt. Chem. **31**, 367) observed the solubility of metallic gold in potassium cyanide, and both noted that the solution was assisted by air. Dr. L. Elsner (Jour. prakt. Chem. **37**, 441) proved that the presence of oxygen was necessary. It may come as a surprise to some to learn that the oft quoted Elsner equation is not contained in this paper. But since it embodies Elsner's views it may still retain his name: $4KCy + Au_2 + H_2O + O = 2KAuCy_2 + 2KHO$.

No mention of the necessity of the presence of oxygen is made in any one of the MacArthur-Forrest series of patents, and Mr. J. S. MacArthur denied this necessity as late as 1890 (this Journal, 1890, 270).

Elsner's original observations as to the necessity of the direct intervention of oxygen in the solution of gold by cyanide have since been confirmed by Skey in New Zealand, and more recently by J. S. Maclaurin (this Journal, 1893, 359), whose conclusions are:—"1. That oxygen is necessary for the dissolution of gold in potassium cyanide, and that it combines with the potassium of the potassium cyanide in the proportion required by Elsner's equation. 2. That the rate of dissolution of gold in potassium cyanide solutions passes through a maximum in passing from dilute to concentrated solutions, and that this remarkable variation is capable of explanation by the fact that the solubility of oxygen in cyanide solutions decreases with the concentration."

Prof. Christy confirms Elsner and Maclaurin.

Maclaurin's paper makes the vexed question of "selective action" perfectly clear, and it is now pretty obvious that a cyanide solution will dissolve base metal from an ore as a function of its strength, but will dissolve gold as a function of its strength and its oxygen contents. Since Maclaurin's proof that the oxygen dissolved is an inverse function of the cyanide strength, it became a pretty problem to determine the best practical strength, so as to give the maximum solution of gold with the minimum of base metal. The answer to this problem naturally varies with the ore and with the method of working, but we believe that the strongest liquors now employed seldom exceed 0·3 per cent. of potassium cyanide, and may go down to a tenth of that strength.

The solvent power of a cyanide solution in practice depends on the oxygen supplied from air (1) previously dissolved in the liquors, (2) entangled between the grains of ore or tailings, and (3) dissolved by the surface layers of liquid and slowly diffused throughout the mass during leaching.

These supplies are sufficient to effect the fairly rapid solution of the finer particles of gold at the commencement of the leach, but the varied applications of strong and weak solutions, the necessity in many cases of alternate drainings of the partially extracted ores, and, in general, the effecting of as much aëration as possible, all point to the comparatively rapid exhaustion of the oxygen supply, and consequently to the intermittent nature of the solvent action occurring with simple cyanide liquors. The coarser the gold particles and the richer the ore, the more striking does the necessity of oxygenation become. The difficulty in extracting rich pyritic concentrates by the ordinary cyanide method, and the great length of time required, is partly due to this among other factors; and it is not all to be laid to the more or less complete overlaying of gold laminæ by crystals of pyrites. Moreover, the large masses of pyrites and other oxidisable minerals immersed in alkaline solutions intercept the oxygen supply necessary for the dissolution of the gold, thus lengthening the latter process and enabling the cyanide to be destroyed partly by the oxidised products formed and partly by greatly prolonged contact of the cyanide with the naturally occurring compounds of the base metals.

Many attempts, embodied in numerous patents, have been made to improve the cyanide process by the employment of oxidising agents as a substitute for, or adjunct to, aerial oxygenation, but these "oxidation" improvements are already practically obsolete.

Any cyanide oxidised to cyanate is so much loss, Maclaurin having proved that cyanate has no influence on the extraction of gold.

If oxygen *must* be used, air is the safest and cheapest source. The inevitable defects, however, in all air-oxygenation—or chemical oxidation—cyanide processes are:—

(1) The slow oxidation of pyrites and other base sulphides, with consequent destruction of cyanide and loss of oxygen.

(2) The production of caustic potash as a necessary reaction product of the solution of the gold.

With free-milling or well-oxidised ores, containing none of the worse "cyanicides," the production of caustic alkali is not harmful, and may even be useful; but directly sulphides of iron, copper, arsenic, lead, zinc, antimony, &c., make their appearance in an ore, soluble metallic and sulphide compounds are produced from them (mainly by reason of the caustic alkali formed), which react directly on the cyanide and destroy it.

The oxygenation-cyanide process is, as a rule, inapplicable in the following cases:—(1) To ores containing coarse gold *only*. These may at once be dismissed as being fit subjects for the cheapest method of gold extraction, *viz.*, amalgamation. (2) Rich pyritic ores and concentrates. These, where not too heavy in gold contents and under specially

favourable conditions, may be occasionally treated by cyanide; but general Transvaal practice, even when the cyaniding can be done—as, for instance, with the Robinson concentrates—is to fall back on roasting and chlorination. (3) Ores containing arsenical or copper pyrites, even in moderate quantities.

After a long search for an accelerator which shall not be an oxidising agent, we find that when a haloid compound of cyanogen—in practice, bromide of cyanogen is found to work best—is present with cyanide of potassium, the rapidity of the solution of the gold is greatly increased. Bromide of cyanogen *per se* is no more a solvent of gold than potassium cyanide *per se*.

The following equation represents the main chemical reaction that takes place, although there may be, and in fact are, other minor changes taking place at the same time :—

$$CyBr + 3KCy + Au_2 = 2KAuCy_2 + KBr.$$

This is a pure cyanidation reaction, quite distinct from an oxidation one. No air is necessary, since oxygen takes no part in the reaction. No caustic potash results from the reaction, so that the evil results which it causes with certain ores are avoided.

Many attempts have been made to prove this reaction to be only another form of oxidation. All such theories, however, require the intervention of *hydrolysis*. That hydrolysis is extremely improbable, is evidenced by the stability of the simple aqueous solutions of these haloid compounds, especially when below 1 per cent. in strength.

It has been suggested that thermo-chemistry might explain the greater rapidity of our solvent. It, however, points the other way, more heat being produced by Elsner's equation than by ours.

Comparing the thermal equations of the two types of solvent, and eliminating from each the heat of formation of potassium aurocyanide, as common to both, we have :—

(1) Aëro-cyanide type—

$$H_2O + O + \begin{matrix} KCy \\ + \\ KCy \end{matrix}\ \left.\begin{matrix} Au \\ Au \end{matrix}\right\} = \&c.$$

		Calories.
Heat liberated by oxidation of 2K to 2KHO.	=	164·6
Heat absorbed by dissociation of 2 KCy	=	− 129·4
Balance		+ 35·2

(2) Halo-cyanide type—

Chlorine is here taken for the comparison, no data as to the heat of formation of bromide of cyanogen being available:—

$$\begin{matrix} KCy \\ + \\ ClCy \end{matrix}\ \left.\begin{matrix} Au \\ Au \end{matrix}\right\} = \&c.$$

		Calories.
Heat liberated by formation of KCl.........	=	100·8
Heat absorbed by dissociation of KCy.......	=	− 64·7
Heat absorbed by dissociation of ClCy	=	− 9·9
Balance...................	=	+ 26·2

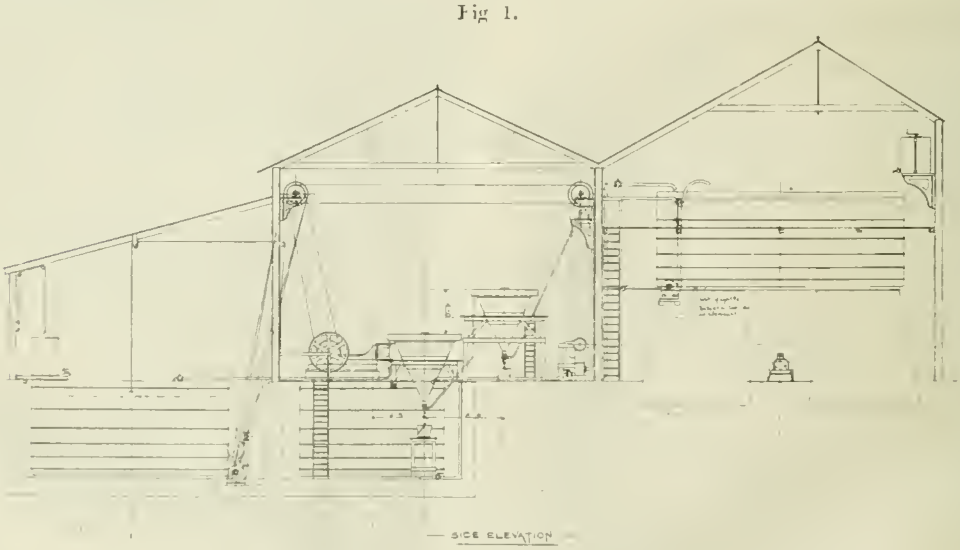
Both reactions are therefore exothermic, but on paper the balance of advantage is certainly in favour of the oxygen combination. The explanation of the total reversal of thermo-chemical indications in practice is that, in the case of the aëro-cyanide type of solvent, the thermo-chemical equation represents an ideal condition of affairs, which actual physical factors render impossible of realisation.

Assuming the solubility of oxygen in dilute potassium cyanide solution is the same as in water, and that oxygen constitutes one-fifth of the atmosphere, calculation (on the basis of Elsner's equation) shows that 18 cb. ins. of plain cyanide solvent would be required to dissolve one grain of gold. If every particle of solution were brought into contact with every particle of ore, applying a weight of solvent equal to one-third of the weight of the ore, the aërocyanide solvent could not possibly extract more than 2 oz. 2 dwt. 9 grs. of gold per ton of ore. In the above calculations it is assumed that no oxygen is wasted on oxidisable matter in the ore, but also that no atmospheric reoxygenation takes place during the progress of the extraction.

It would only require rather less than half a cb. in. of a solution of 0·1 per cent. of potassium cyanide and 0·025 per cent. of bromide of cyanogen to dissolve 1 grain of gold, or a solution weighing one-third the weight of the ore would extract as a maximum about 80 oz. of gold per ton of ore. The advantage of our solvent is its great superiority in "potential" cyanogen.

A gold extraction process by leaching methods is not confined to the application of a solvent, but may be divided into three parts: (1) The solution of the gold from the ore, (2) The precipitation of the gold from its solution, and (3) The gold clean-up, or separation of the gold from its accompanying precipitant.

Fig 1.



General Diagram of Plant.

In our method of precipitation we employ the finely divided by-product known as "zinc fume." Great difficulty was experienced at first, in large scale operations, in intimately mixing the small requisite quantity of zinc fume with a large mass of gold-bearing liquor. Ultimately, the simple form of apparatus now known as a "zinc cone"

was evolved. The diagram shows this to be a light metal cone standing on its apex; it is charged with a certain small amount of zinc fume in the state of an emulsion. The gold-bearing liquors rise through an opening at the apex, become thoroughly mixed with the cloud of zinc fume, and then, as the liquors rise further, their velocity becomes so small by reason of the increasing area, that the suspended zinc fume and gold-zinc is completely deposited before the precipitated liquors flow over the upper circular edge of the cone. The cone combines the following factors:—(1) Completeness of admixture; the extremely finely-divided zinc being presented continuously to every portion of the liquor in a great excess for a short time, whereby the liquid is brought into almost molecular contact with the precipitant. (2) The clarifying of the liquors by continuous deposition. (3) The production of rich gold-bearing zinc precipitate (which can be obtained containing as much as 30 per cent. of gold).

The enormous surface exposed by a given weight of zinc fume, when compared with that of zinc shavings, enables the former to precipitate the gold much more rapidly and therefore, during given equal times, more completely than is possible by the application of zinc shavings in practicable systems of zinc boxes.

This method of treating solutions by zinc to recover their contents of other more electro-negative metals is naturally not confined to the treatment of the liquors obtained by our particular solvent; it is applicable to ordinary cyanide liquors, and in fact to any liquor, acid, alkaline, or neutral, containing dissolved metal precipitable by zinc.

The third operation is the gold clean-up. Whether from the MacArthur zinc boxes or from our zinc cones, the gold-bearing precipitate has to be freed from the accompanying zinc for conversion into bullion. The somewhat elaborate process adopted for the ordinary zinc-box "gold slimes" would be equally applicable to our product. We prefer, however, to distil the air-dried gold-bearing zinc from a retort, leaving the gold behind, and again recovering the zinc in the very form in which it is required—zinc fume.

Before entering on the description of practical working we may add a word as to the testing of liquors. Cyanide liquors are tested in the same manner as cyanide liquors from the MacArthur-Forrest process. When the amount of bromide of cyanogen has to be determined, however, recourse must be had to a reaction which we think has a certain amount of interest apart from its technical application. Bromide of cyanogen mixed with excess of potassium iodide and acidified with hydrochloric acid, liberates iodine in the ratio expressed in the equation—

$$BrCy + 2HI = HBr + HCy + I_2.$$

The liberated iodine is determined in the ordinary manner by thiosulphate. Excess of cyanide does not interfere with the reaction, and consequently the text books that state that iodides cannot be detected in presence of cyanides by means of chlorine or bromine require amendment. Bromide of cyanogen is a most excellent reagent for detecting traces of iodides, as it has not the property, when used in excess, possessed by free chlorine and bromine, of converting the liberated iodine into colourless iodic acid. We have successfully detected iodine in sea-water, without previous concentration, by first shaking with zinc fume, and then, after filtration, adding bromide of cyanogen, hydrochloric acid, and starch water, viewing the water in a foot tube.

We have now to consider our process as applied in practice, and for this purpose we shall take two mines, as examples, where the ores are as dissimilar as possible:—

Day Dawn (West Australia), where the process is worked upon battery tailings from a free-milling ore; and Deloro (Canada), where we are extracting an ore *direct*, without any preliminary roasting or chemical treatment, consisting of massive arsenical pyrites often carrying over 40 per cent. of arsenic.

Crushing.—At Day Dawn the ore is battery-crushed from 25 to 30 mesh to the linear inch, and passed over plates, and the tailings only are treated. The gold is simply as it has escaped in the quartz sands from the battery plates, and is mostly fine and accessible. The tailings are taken straight from the battery pits or from the tailings heap, and when from the latter contain about 15 per cent. of air-dried and agglomerated slimes.

Fig. 2.

Gruson-Krupp Mill.

At Deloro, the gold is in a laminar condition between crystals of arsenical pyrites. The ore is there crushed by a Krupp Grusonwerk ball mill to 30—40 mesh. A certain amount of fines is separated by an air grader. This part of the process is in course of modification, as it is found that, despite many applications of the principle of air separation, a large proportion of still leachable though fine material is carried off with the unleachable slimes by means of applied air currents. In several cases investigation showed that the air-separated product contained 40 to 60 per cent. of material, which, though very fine was still granular enough to have been dealt with in the leaching tank. To retain this it has been found desirable to have recourse to wet separation, whereby not only is the mass of the fines greatly reduced, but the absolute slimes rejected by it are very much poorer than the average product separated by an air current. Slimes from Deloro ore by wet separation can be obtained as poor as 3 dwt. per ton, or even less, whereas the dry separated material often runs from 8 to 9 dwt.

At both mines the crushed ore or tailings is simply dumped direct into the leaching vat. Both plants are for working 50 tons a day.

Extraction.—No preliminary wash of acid, alkali, or water is used at either mine. At Deloro a preliminary alkali wash was found to considerably increase the cyanide consumption without any compensating advantage in the subsequent extraction. It was according discarded. We have found this result constantly reproduced in treating heavily arsenical-pyritic ores.

In addition to the ordinary cyanide plant, a bromide of cyanogen solution tank is provided, which is shown on the elevation of the plant and in some of the photographs. It is made of wood throughout, provided with a gauge glass graduated in gallons, is fitted with an air-tight cover, a spigot cock outlet, and a vent orifice, to admit the necessary air in running off the liquor. No metal is used in its construction. There is also a perforated wooden box placed centrally over the extraction vat, into which the delivery pipe of the cyanide liquor is taken, and into which also the bromide of cyanogen solution is conducted by a small rubber hose-pipe from its dissolving tank. The mixed liquor is thus spread on the charge of ore through the box perforations. The bromide of cyanogen solution

is prepared in its tank by direct solution of the solid product in water; at ordinary temperatures a 7 to 8 per cent. solution can be obtained. The perforated distributing box enables a fairly uniform mixture of the two liquors to be obtained before they are run upon the ore, and breaks up the heavy inlet current, preventing, by means of its rose-like discharge, "burrowing" into the surface of the ore charge.

In addition to the bromide tank there is the usual strong cyanide or dissolving tank (250 galls.) placed over or near the sump tank, fitted with a sieve and counterbalance. The sieve is provided with a cane bottom, over which is placed twill or calico; upon this rest the lumps of cyanide of potassium required for making up the strong liquor. A small pump circulates the liquor round and round on to the sieve until the solution of the cyanide is completed. The amount of strong potassium cyanide required to be added from time to time to the leaching liquors is calculated from the analysis of the sump liquor, and the necessary addition of strong liquor is run into the sump store. It is important to note that the cyanide and bromo-cyanide solutions are kept separate and are only mixed immediately before use.

The addition of bromide of cyanogen is proportioned to the work to be done, calculated from a previous trial. Any unnecessary excess should be avoided, as it does not survive (as bromide of cyanogen) its passage through the zinc cone. Speaking generally, it should not exceed, as a maximum, one quarter of the weight of the cyanide of potassium present, though in most cases a less proportion of bromide of cyanogen than the above is found to suffice.

In charging the ore with the mixed solutions, the vent peg of the bromide tank is loosened, and the spigot tap turned partially on, so that the calculated quantity of strong bromide of cyanogen liquor shall be roughly apportioned in its rate of flow to the time occupied running on the main charge of cyanide liquor.

A circulating pump is attached to the leaching tank having its inlet below the filter bottom, and its outlet, returning over the top of the tank. When the addition of the mixed bromo-cyanide leaching charge to the ore is completed (about one-third of the weight of the ore), the liquors may be circulated for two or three hours, until fairly uniform in composition. The circulation may be repeated at intervals if desired.

The time of extraction is 14 to 15 hours at Day Dawn and 30 to 40 hours at Deloro, the difference being entirely due to the very different distribution and occurrence of the gold particles in the two ores. At Day Dawn only one set of liquors is used, averaging 0·1 per cent. or less of potassium cyanide, with a proportionate amount of bromide of cyanogen. At Deloro, two sets are employed: the strong, 0·2 to 0·25 per cent., and the weak, 0·1 per cent., of potassium cyanide, each with its small requisite addition of bromide of cyanogen. The consumption of chemicals is at Day Dawn rather less than 4 oz. of potassium cyanide and 1¾ oz. of bromide of cyanogen per ton of ore. At Deloro it is 1 lb. of potassium cyanide and ⅓ lb. of bromide of cyanogen per ton of ore. The percentage of extraction averages about 90 per cent. at both mines. At Day Dawn the tailings average 3 to 4 dwt., and the residue, after extraction, 8 to 10 grains.

On more than one occasion these 14 hours extracted tailings were boiled with aqua regia, when no further diminution of the residual gold contents was effected, thus proving a complete extraction of all exposed gold by the short bromo-cyanide leach. It is of course obvious that no process that does not dissolve quartz can attack a particle of gold completely surrounded by quartz.

In endeavouring to ascertain the minimum amount of cyanogen bromide necessary, it was once reduced to ¾ oz. to the ton of ore; here the solution began to approximate more nearly to one of plain cyanide. After two days the extraction was still imperfect, but on the addition of the balance of bromide of cyanogen, it was completed in a few hours. This experiment very clearly demonstrates on a practical scale the immense advantages of bromide of cyanogen as an accelerator even in the case of tailings from a clean quartz ore.

We would here remark that it was the work done at Day Dawn by one of the authors and Mr. Hans Knutsen which primarily established the great superiority of bromo-cyanide over plain cyanide in dealing even with free milling tailings. The details of precipitation and the overcoming of some difficulties, both in this and the clean-up, not at first experienced there, were worked out by Mr. Hugh K. Picard at Deloro, in treating this highly refractory ore.

The Deloro ore averages about 17½ dwt. of gold to the ton, and "is an arsenical sulphuret of iron, in a gangue of quartz and calcspar, an analysis of which gives about 42 per cent. of arsenic, 20 of sulphur, and 38 of iron" (Lock, "Practical Gold Mining," p. 667). Mr. Picard's experience is that it always contains small though variable quantities of copper pyrites, also slate and mica. The crushed material treated, varies from 25 per cent. of mispickle up to a mass containing sometimes as much as 40 per cent. of arsenic. It is roughly hand-concentrated, as the mispickle carries practically all the gold. It was formerly treated by the Mears process of chlorination, after hand-picking, concentration by jigs, and roasting (*ibid.* p. 665). But this process proved a complete economic failure. Experiments were conducted to see whether plain cyanide treatment would not suffice for the ore, but they were quite unsuccessful. It may be taken as a generally granted fact, that ores containing a notable proportion of arsenical pyrites are debarred from the ordinary cyanide treatment. In this particular case we have therefore supplanted both chlorination and plain cyanide treatment.

In Deloro ore 50 per cent. of the gold can occasionally be extracted by bromo-cyanide in six hours. Such amount may be regarded as the immediately available or freely accessible gold, *i.e.*, where the gold film lying between the crystals of pyrites has been thoroughly exposed by the removal of one of the overlying surfaces. After these free films have been dissolved, those imprisoned between the pyritic surfaces have to be attacked edgewise—a much longer operation—so that the total time amounts to from 30 to 40 hours.

It was found here that, with the ore only crushed to from 20 to 25 mesh, extraction ruled low—often only 65 per cent.—whereas, upon crushings carried to from 35 to 40 mesh, the 90 per cent. removal was again obtained. These experiences indicate that it is not the presence of the arsenical pyrites from a chemical point of view that prevents the total extraction of the gold, but the fact that it actually mechanically encloses the gold. As is generally the case, finer crushing exposes more gold, and permits of a higher extraction.

Bromide of cyanogen being a solid is more readily taken by shippers than bromine. It is exported in hundred-weight and half-hundredweight cases, lined with 5 lb. lead, previously tested by hydraulic pressure to 30 lb. to the square inch. The case is not opened until required, and then the whole of the contents is emptied into the bromide of cyanogen tank.

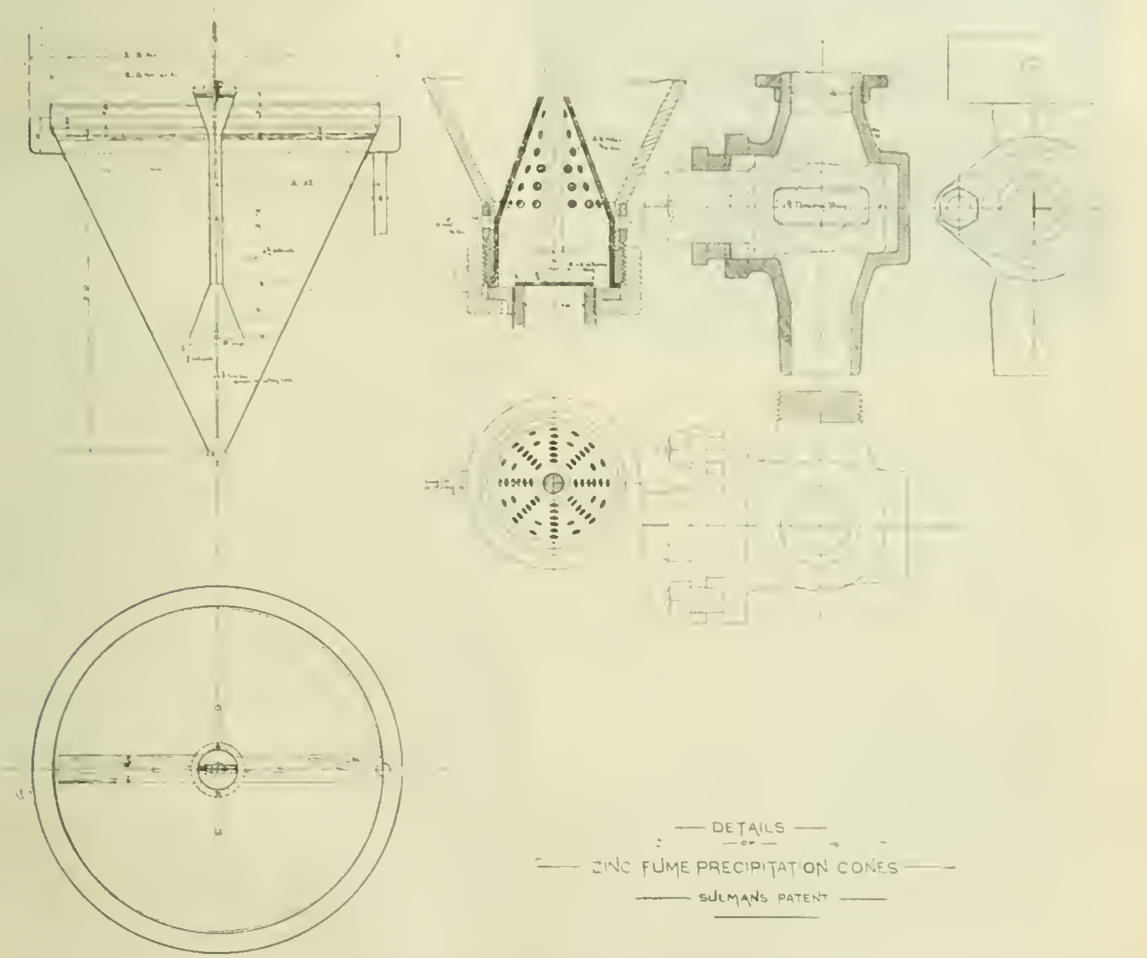
The total bromide of cyanogen admixture requisite to an ore charge is not necessarily made all at once, but may be in two or more additions, during circulation, from time to time. This is found advisable when, from the laminated and partially enclosed nature of gold films held between crystals of pyritous or similar minerals, the extraction is necessarily somewhat prolonged.

A point of much importance, from its being a divergence from ordinary cyanide practice, requires mention. The dissolved gold salt in even dilute bromo-cyanide liquors is so very stable that, in spite of all cyanide experience to the contrary, it is found not only convenient, but desirable, to make one charge of liquor extract several vats of ore in succession, without intermediate precipitation. Even with the extremely refractory Deloro ore, no tendency whatever to reprecipitation of the gold in the fresh ore charge is noticeable. One batch of liquor is usually made to extract four vat charges of ore, and even five vats have been successfully extracted, before passing the liquor through the zinc cone. The bearing of this method on economy in precipitation is evident.

Precipitation.—We prefer to at once discharge the whole of the gold-bearing liquor derivable from the extraction vat into an intermediate drainage sump; by so doing, two advantages accrue: (1) It enables the gold-bearing liquors to be run off from the exhausted charge much more rapidly than would be the case if the rate of withdrawal were made dependent upon the comparatively slower rate of flow necessitated through a precipitation plant; the leaching tanks are thus freed more quickly for the reception of a fresh charge of ore. (2) We also secure that uniform quality and strength of liquor and rate of flow through the plant which is so essential to the best results in any precipitation method.

We prefer to use two zinc cones in series, placing the one 2 or 3 feet above the other. They are constructed of light sheet iron, painted with zinc fume in linseed oil, or

Fig. 3.



may be made direct from galvanised sheet iron. They are about 5 ft. to 5 ft. 6 ins. top diameter, and of similar depth. Round the top of each is fixed a circular gutter for collecting the uniform overflow of clear liquor, which is conducted thence, as the case may be, either from the first cone to the second, or from the second cone to the storage sump for precipitated or gold-free liquor.

The unprecipitated liquor enters through the bottom of the first cone from a small tank, placed to give some 2 or 3 feet of head above the top level of the cone. This tank is fed directly from the intermediate sump by a small plunger pump. The stream, on entering the bottom of the cone, is distributed by means of a small perforated inverted cone-nozzle, inside which is fitted a valve arrangement to prevent any backward flow of zinc emulsion from taking place when the cones are stopped. The bottom is also provided with a three-way cock, which either permits the inflow of liquor for precipitation, or shuts this off to permit the discharge of the gold-impregnated zinc fume as desired.

Occasionally a small "flock" of material, consisting of a few zinc-gold couples, buoyed up with a bubble of hydrogen gas, may rise through the deposition zone to the surface of the liquor in the cone. To prevent such coarse flocks being carried into the overflow, a screen, 3 ins. deep, of coarse calico, is placed around the overflow rim, and is carried by a circular rod supported on small standards. In the photograph, the calico screen itself has been removed to show these. The addition of fresh zinc fume is made in the form of thick emulsion in water, by means of the central small funnel shown. It has been found advisable in practice to again expand the bottom of this funnel for the purpose of producing a sort of vortex chamber at the bottom of the zinc cone, which ensures a more thorough admixture of the zinc fume emulsion with the inlet liquors.

Assuming the zinc cones to be full of old precipitated liquor, and the gold-bearing liquor from 100 tons of exhausted tailings to require precipitation (this gold-bearing liquor being of course in the intermediate sump), the following is the routine:—

The gold-zinc fume from cone No. 1 is removed for clean-up: the partly used gold-bearing fume from cone No. 2 is transferred to cone No. 1, and a charge of about

12 lb. of fresh zinc fume in an emulsion with water is run into cone No. 2. The gold-bearing liquor is now run through the series at the rate of 400 to 600 galls. per hour, and occasionally even at a greater rate.

Fig. 4.

Cone and Circulating Pump—Deloro.

The zinc cone automatically grades the zinc particles, if of varying degrees of fineness, so that the heavier, coarser particles are in the richest gold-bearing stratum, and the liquor, in rising through the cone as it becomes poorer in gold contents, constantly meets finer and finer suspended particles of zinc. But inasmuch as these latter become weighed after a time with gold, there is a tendency for them to be withdrawn from the top zone of slow upward travel. For this reason it is found advisable to add about every one or two hours or so, some fresh zinc fume emulsion, containing about 1 lb. of dry material, through the central funnel.

The zinc consumption at Deloro over a period of four weeks (Oct. 1st to 28th) amounted to 0·54 lb. of fume per oz. of fine gold recovered.

Very little difficulty has been experienced from the overflow of even the most finely divided particles of zinc, but as a precautionary measure a small settling tank (containing slats, placed at an angle to the vertical, about half an inch apart, and extending across its whole width) is introduced between the second cone and the sump. This tank (omitted in the diagram) is interposed in case of a slight overflow of fine zinc fume on starting the precipitation, and effectually stops the passage to the sump of even the finest zinc particles; this small catchment tank only requires a very occasional clean-up. The consumption of zinc is very small, and the gold-bearing fume generally weighs only a pound or two less than the charge originally introduced. The precipitated liquor, as in the plain cyanide process, is used over again after being made up by an addition of strong cyanide liquor to its normal strength.

It has been found possible to reduce 1 oz. gold liquor (per ton) to 3 grains when passing through a single cone at the rate of 400 gallons per hour, and frequently the outlet liquors do not exceed 10 grains per ton. Such great closeness of precipitation is, however, in general, unnecessary, as the unextracted gold is not lost, but only represents a very small portion of locked-up capital. It never increases, as the liquors are used over and over again, and precipitated to this standard. In Canada it has not been found necessary to precipitate closer than 1 dwt. per ton, which is, however, a figure that is never exceeded.

After the passage of the whole liquor charge, the gold-zinc fume is removed, after a short settlement period, from the first cone by means of the three-way cock, in the course of a few minutes. It is then drained on a calico filter preparatory for the final distillation (clean-up). As mentioned above, the fume from the second cone is transferred to the first.

We have not had occasion to treat very rich liquors as yet. The Deloro liquors run between 2 and 3 oz. to the ton, while those at Day Dawn are very much poorer. At the Hannans Brownhill mine, where a very large installation of the bromo-cyanide process is now in course of erection, much richer liquors will be obtained, as the ore will probably average from 3 to 4 oz. per ton. Here three cones will be used in series, and from preliminary trials it is anticipated that the zinc-gold fume removed from No. 1 cone will uniformly exceed 30 per cent. in gold contents. It is proposed to add an automatic zinc-fume feed to the cone series in this case, in order to make the necessary small additions of zinc fume uniformly and continuously.

Clean-up.—The clean-up by the zinc-box method consists in sifting the zinc shaving fragments, covered by the light purplish-brown deposit of powdery gold, upon sieves submerged under water. The larger fragments of zinc, retained upon the sieve, are returned to the richer zinc-box compartments, whilst the very fine and valuable deposit which passes through, much of it in a state of extremely fine suspension, is, after settling, washed with care by decantation (or in a filter press) and finally dried.

The first step in the removal of the zinc is careful roasting upon a suitable iron plate or hearth freely exposed to the air; but as a large proportion of the zinc *débris* is in fragments of sensible size, a complete oxidation by mere atmospheric means entails much time and trouble. Nitre is therefore frequently added to complete the oxidation. This addition may vary from a few per cent. upon the dry "gold-slimes," as they are termed, to, in extreme cases, their own weight in nitre. The fully roasted charge is treated with a sufficiency of dilute hot sulphuric acid to dissolve the great bulk of the oxide of zinc as sulphate. The residue is now naturally very rich in gold contents, and is still highly divided; it is washed free from soluble zinc salts, collected, dried, and smelted with varying additions of borax, sodic carbonate, silica, &c., to obtain the bullion in ingots.

Mr. James has recently collated the sources of loss which arise from zinc clean-up, in a paper before the Institution of Mining and Metallurgy; and Mr. T. H. Leggett, in another paper before the same body, states: "In the roasting with nitre method in vogue on the Rand, the loss due to stirring and the escaping fumes is something the Rand metallurgists do not like to talk about." It is very difficult to form a just estimate of these losses, but we do not think they would be over-estimated if put at an average of 2 per cent. of the gold contained in the original gold-slimes. Moreover, the process is a lengthy one, entailing several days' work, and necessarily implying the stoppage of the zinc boxes for a while. The clean-up under the zinc-box system naturally, therefore, only takes place monthly or bi-monthly, when the zinc shavings have become sufficiently rich to warrant it.

The Siemens-Halske electro-deposition process also consumes a very considerable time in the removal of the old gold-lead cathodes and their replacement in the frames by fresh lead sheets, &c. Moreover, the Siemens-Halske clean-up necessitates the cupellation off of an excess of lead, which, roughly speaking, constitutes about 90 per cent. of the weight removed from the precipitation apparatus.

In our process, the removal of the zinc-gold fume from the precipitation apparatus is effected daily, or after the passage of one or more charges of gold liquor through our plant, by the mere process of turning a three-way cock; and occupies, with the substitution of fresh quantity of zinc fume, only a few minutes; and the precipitation plant being dissociated from the leaching plant, no hindrance is offered to the general work of extraction. The removed zinc-gold fume is drained upon an ordinary calico filter.

The general impression among metallurgists is that zinc and gold cannot be heated together to the temperature of volatilisation of the former without serious loss of the latter. It is true that if gold and zinc be melted together in a muffle furnace through which a current of air is passing, the loss of gold is serious. We contend that the losses there experienced are caused through the oxidation of the

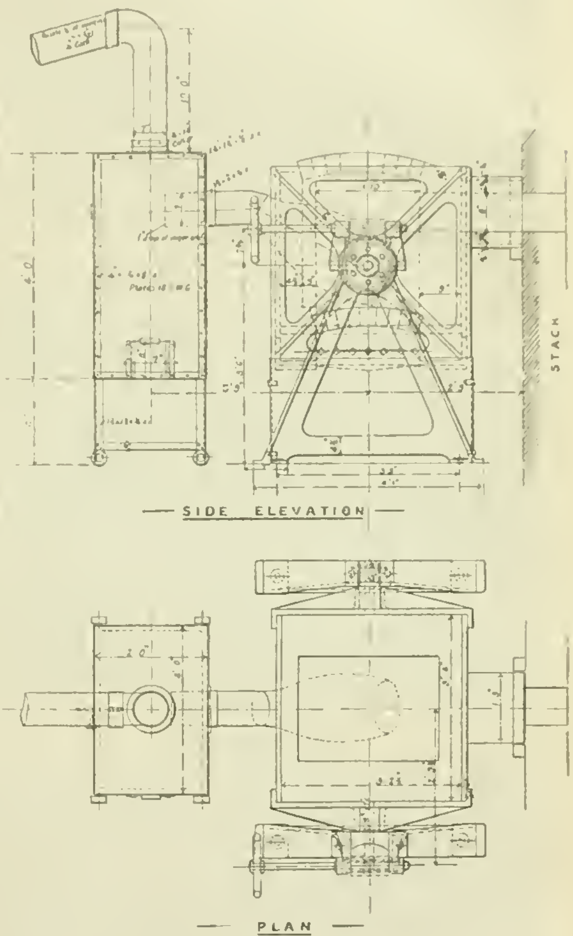
zinc, the gold being carried off mechanically entangled with the minute solid particles of zinc oxide. In the distillation of zinc-gold fume at Day Dawn, it was noticed that whenever oxide of zinc was allowed to issue from the mouth of the retort freely—that is, to be formed in the retort itself by a back-flow of air—the loss of gold during distillation of considerable charges became serious; and the interior of the flues which conducted away the fumes of oxide of zinc was soon found to be studded with minute beads of gold. Whereas in experiments in well-luted crucibles, when the zinc-gold charge contained a small proportion of carbon, and the temperature was sufficiently maintained to ensure a continuous current of metallic zinc vapour through a central escape orifice, so that air could not enter, the distillation of zinc from gold took place with almost complete absence of gold loss, the resulting bullion being found melted at the bottom of the crucible after completion of the operation over 800 in fineness. This led us to make a series of experiments as to the best means of employing the convenient form of zinc distillation plant known as the Balbach retort and furnace for our purposes. The difficulties attending the mechanical loss of gold did not cease entirely with the mere exclusion of oxygen, a further deficit being caused through the escape of permanent gases, and zinc vapour itself, in the form of small fountains or "geysers," through the hot, fine powdery retort charge. This difficulty has been brilliantly overcome by Mr. H. K. Picard, A.R.S.M. To the zinc gold fume, on removal from the final cone, he adds some soluble carbonaceous substance, such as gum, flour, flour-paste, starch, sugar, or molasses, together with a little borax. The resulting mass is dried down by any available gentle waste heat; the bone-dry cake is broken into lumps about the size of walnuts and charged into a capacious plumbago retort. A sheet-iron condenser is luted to the mouth of the retort and the furnace fired up. The organic admixture becomes carbonised and the zinc-gold contents are held bound together, though in an open spongy condition, by a fine carbonaceous skeleton. This serves to fix the finely divided contents temporarily while affording free passage to the escaping zinc vapours during distillation, and provides an excess of carbon to combine with any atmospheric oxygen that may enter. The loss of gold is thus reduced to a minimum. In the distillation of $\frac{3}{4}$-cwt. charges of zinc-gold fume, the condensed zinc and zinc oxide vapours, deposited in the sheet-iron condenser, show constantly upon assaying only slightly over 1 oz. of gold to the ton, or a total loss of less than 1 dwt. of gold on a charge of $\frac{3}{4}$ cwt. of zinc-gold precipitate carrying 20 per cent. of metallic gold. This is a loss in gold clean-up of considerably less than 0·1 per cent. of the gold present in the precipitate. The metallic and oxidised zinc products deposited in the condenser run curiously constant in gold contents whatever be the richness of the zinc-gold fume distilled, namely, about 1 oz. to the ton. But the relative loss is so small that hardly any appreciable difference is made whether the gold-zinc from rich or poor liquor precipitation be distilled.

In the diagram given of the Balbach design of plant it will be seen that the condenser can be removed from the mouth of the retort when the distillation of the zinc is sufficiently perfect, and that thereupon the whole furnace can be tilted by means of the cog and worm wheel adjustment fitted to it.

As an example of zinc fume distillation, conducted at at Deloro, we may instance the following:—A 55-lb. charge of zinc-gold fume containing 70 oz. of gold was balled with $12\frac{1}{2}$ lb. of sugar and 1 lb. of borax, the proportions adopted being roughly the use of 5 lb. of sugar and $\frac{1}{2}$ lb. of borax to each 20 lb. of precipitate. It will be noticed in the photo of the distillation plant that a small talc door is provided at the back of the condenser, through which the progress of the distillation can be watched, and any accumulation of zinc oxide round the mouth of the retort may be removed, if necessary, once or twice during distillation, by means of a small rake or slice. The coal required for complete distillation is about 3 cwt. and the time 10 hours.

The ingot, cast direct from the retort, contained 64 oz. of gold out of 70 oz. possible, the remainder being in the few pounds of slag left, which one smelt in a No. 35 crucible dealt with directly and recovered. Coal for remaining smelt, say 1 cwt.; so that the total coal used throughout is 4 cwt., = 1 dollar at Deloro. Add to this, $\frac{1}{4}$ share of a 7-dollar Dixon fireclay plumbago retort (assuming a retort to last four times only), say 2 dols., fluxes and crucible for subsequent smelt, say 1 dol. Total clean-up cost 4 dols., which Mr. Pickard considers an outside figure.

Fig. 5.



Zinc Retort and Furnace.

In Mr. Pickard's latest reports, slightly less proportion of flux and molasses have been used, with the result that only 3 lb. of residual slag remained in the retort to be smelted after the gold ingot was tipped. The clean-up of 70 oz. of gold, say of value of 280*l.* sterling, thus consumed only 16*s.* worth of fuel, chemicals, &c., being under 0·3 per cent. We would also point out that this operation of clean-up takes place twice a week, or more often as required, as one of the ordinary operations of the assay house. No hindrance or dislocation of general work is caused by such a clean-up. The operations are kept in constant touch with the process, and the capital is recovered practically contemporaneously with the extraction of the ore.

On the scale of work at present proceeding, Mr. Picard estimates the cost of clean-up at rather less than 6 cents for every ounce of fine gold recovered. Were this scale increased to that of a Transvaal clean-up, this small cost would undoubtedly be further and considerably reduced.

We may summarise the results achieved:—

(1) The bromo-cyanide process excels the older cyanide process on its own ground, *i.e.* on free-milling battery tailings, both in rapidity and economy of extraction.

(2) The use of cyanogen in gold extraction has been extended to a province of refractory ores hitherto entirely beyond the reach of any process.

(3) The introduction of a new improved process for precipitating gold from cyanide and other liquors.

And finally (4) The clean-up of zinc-gold fume with an efficiency and simplicity not yet approached by other means.

In the working out of this chain of processes we have been greatly helped by the efforts of many able chemists. We gratefully acknowledge the highly important work accomplished by Mr. Hugh K. Picard, A.R.S.M., at Deloro. It is in a very great degree to Mr. Picard's efforts—ably aided by Mr. R. H. Harland, F.I.C.,—that we are indebted for such a satisfactory demonstration of the capability of the bromo-cyanide process. To many other gentlemen we are indebted for practical help: to Messrs. L. G. Attenborough, A.R.S.M., F. B. Dick, and A. Carr, B.Sc. In Western Australia we received great assistance from Mr. Hans Knutsen—who first suggested the use of the Balbach form of furnace for distilling the zinc-gold charge—from Mr. Edwin Bull, and others.

With the advent of a direct leaching or percolation method, as typified by the older cyanide process, a fundamental reconsideration of the bases of gold extraction, and the dethronement of the battery as the universal gold-winning process, became only a question of time. The revolution has been slow in taking place, but it has been none the less sure. In perhaps the great majority of cases this will be to submit the whole of the suitably crushed ore to direct chemical extraction—thus removing fine and medium gold—and leaving the coarse gold particles in a cleaned condition far more amenable to amalgamation, which at this stage may suitably step in. The perfecting of the cyanide process, or, more widely, of leaching processes in general, must therefore have a far-reaching influence in the modern developments of gold extraction.

Whilst the old battery process, which, with the weapons at its command, was a perfectly rational one, took for its axiom, "Get the bulk of your gold as soon as you can"; the newer system, with the newer weapons at its disposal, rather takes as its motto, "Take care of the fine gold, and the coarse will take care of itself."

Discussion.

Mr. W. Crowder had followed the development of the process with great interest since its announcement at the Institute of Mining and Metallurgy two years ago. He was surprised, however, that no reference had been made to any possible application of the process to Transvaal gold ores, seeing that the Transvaal mines were now the source of the largest production. He regarded the use of zinc fume as a most important advance on the original process, since it effected its purpose admirably, without interfering with any other portions of the operations.

Mr. A. C. Claudet could not discover that the paper contained much more information than was given by the authors two years ago. It was not at work then on a large scale, but had been worked since in Canada and in Western Australia. No mention was made of the results of trials the speaker believed had been made in the Transvaal. It appeared to him that the portion of the paper relating to zinc precipitation had been elaborated to the loss of the paper as a whole. He would leave its discussion in the theoretical aspect to gentlemen more qualified in this respect than himself, but there were one or two practical points with which he would like to deal. In one experiment the authors stated they lowered the amount of bromo-cyanogen to $\frac{3}{4}$ oz., and the extraction was immediately reduced, but they did not state the original strength of the solution. If thus lowered, in order to fairly show the result, the authors should have added its equivalent in cyanide. He did not understand that the application of the process was limited to refractory ores, as suggested by the Chairman, but that it was in all respects, and claimed to be, a competitor of the old MacArthur-Forrest process. It was noticeable, however, that no records of comparative experiments with the two processes were given, and therefore they had simply to accept the authors' statements. That being the case, until such comparative results were forthcoming, he must reserve his judgment on the matter. He had himself made experiments with the Deloro ore, and he found that a very weak solution of ordinary cyanide would extract as much gold as the authors had claimed. The authors' retort to that would probably be that there was an economy in cyanide. On the other hand, it must be remembered that the whole of the bromo-cyanogen was absolutely lost; and in his opinion a true comparison of the two processes could only be got at after an extended series of trials side by side. From the precautions which the authors took to keep the air from the solution he concluded that the bromo-cyanogen was quickly decomposed, and he thought would probably disappear altogether in the course of three or four hours, after which time they would be working with ordinary cyanide only. If that were so, where was the gain? Neither was it stated that the process had paid anywhere. Much was said, however, of the advantages it would offer to the Brownhill Company; but they were not told whether the process was to be as described in the paper. As a matter of fact, this process, like all percolation methods, depended entirely on the nature of the ore dealt with. The ore there was a peculiar one, and he understood that the process used would be to treat the slimes or fine ore obtained by dry separation, equivalent to about 30 or 40 per cent. of the total ore crushed. He was, however, pleased to hear that the authors had experimented on the distillation of the zinc precipitate. He regarded that as an important advance. He had suggested that two years ago, because in the ordinary process the presence of zinc in the final bullion influenced very considerably the value obtained when sold. As the authors claimed that they refined their bullion, he would like to know what its fineness was at Day Dawn and at Deloro. He would also like to know whether the zinc contained lead, and if they preferred to use a zinc containing lead; in the latter case distillation would not remove the lead. He could not agree with the summary of the results claimed, because sufficient time had not yet elapsed to enable them rightly to estimate the value of the process.

Mr. R. H. Harland had had some practical experience of the process, having made tests, in conjunction with Mr. Claudet, on a large scale in London, which led to its adoption at Deloro. Difficulties were encountered in the early stages, more especially with regard to the clean-up of the zinc dust, losses of gold taking place during distillation from a Balback furnace. This had now been entirely overcome by the ingenious method, devised by Mr. Picard, of balling the zinc dust with sugar before charging into the retort. As to the purity of the zinc fume employed, one parcel used at Deloro contained—

	Per Cent.
Lead	1·74
Cadmium	0·69
Iron	0·11
Arsenic	Faint trace
Copper	Nil
Sulphur	"
Siliceous matter	0·19
Carbon	0·52

with a varying proportion of zinc oxide, 3 to 5 per cent. The same impurities in about similar quantity were found in all samples of commercial spelter, and no doubt their presence materially assisted in the rapid precipitation of the gold from the cyanide liquors. After precipitation through two cones the liquor contained from 1 to 3 dwt. per ton, and in some cases less than this. With properly regulated rate of flow and charge of zinc, the liquor from each cone was absolutely free from suspended particles of zinc dust.

At the mine mentioned about 1,300 tons of arsenical pyrites had been treated. The bullion produced without treatment by sulphuric acid was about 690 fine, and contained 25 to 28 per cent. of zinc; it was therefore not so pure as that obtained by the Siemens-Halske process, but the conditions of precipitation and clean-up were simple and entailed little trouble.

Mr. A. C. Claudet said it was quite true that he had watched the process, and the results obtained were favourable; but his remarks now were meant simply to indicate that

there was no apparent advantage after all in using the Sulman-Teed process instead of the old cyanide process.

Mr. D. A. Louis agreed with Mr. Crowder and Mr. Claudet that, with the exception of the new cone and the results of practical working which were now published, the authors had added very little to the information given in their former paper. On the new points not nearly enough had been said to enable one who, like himself, had no practical experience of the process, to form a judgment on their value. Mr. Claudet's statement that with a sufficiently dilute cyanide solution he could get as good an extraction as was claimed for this process, was very important, but he had omitted to say what time would be occupied, and how much cyanide would be used. These were essential points; and one would like to know also what else, if anything, was extracted with the gold. He had himself treated many ores —some very foul ores, and he ultimately got out the gold, but with it other metals of little value, and at an expense for cyanide greater than the value of the gold recovered. Under such circumstances, if no information were demanded as to the time occupied and cost of the cyanide used, one could correctly state that the gold was extracted by cyanide, but not that the extraction was economical or efficient; hence the value of supplying the missing information in such cases. He would like to hear more as to the equation $CyBr + 3KCy + Au_2 = 2KAuCy_2 + KBr$, which suggested that the whole of the cyanogen was utilised solely in dissolving the gold; also as to the words "we get the same thing, but there is no hydrolysis": and again as to the presumed cause of the great increase in the rapidity of the extraction. It was well known that in many cases weak cyanide solution would do all that was claimed by the authors; but the special advantages claimed for the present process were a decrease in the cyanogen used, and, what was very often still more important, a considerable saving in time under adverse circumstances.

Mr. H. de Mosenthal thought that the best assurance that the authors could give of the success of their process was that some among the large number of mines at present using the cyanide process had consented to adopt the addition of bromide of cyanogen to their liquors, and had found their results so improved as to warrant them in continuing to do so. Even if the improvement effected were only that of saving time, it would be sufficiently important, provided that saving was not effected at too great a cost in additional chemicals and royalty.

Mr. A. James had hoped, with other speakers, to find some information about the bromo-cyanide process proper, and was disappointed to find that the authors had confined themselves so much to the zinc fume process, which was after all a secondary matter. With regard to the acceleration of the rate of solution, that was a point which could not be dismissed in a few words. There were times when the rate of speed of the solution was all important, and at such times the solution proposed by the authors would be of great efficiency, for it would undoubtedly, under certain conditions, dissolve the gold much more rapidly than would the ordinary cyanide solution. He found it difficult to agree with the authors when they said that the aero-cyanide solvent could not possibly dissolve more than 2 oz. 12 dwt. of gold per ton, as with exceedingly rich ores it was occasionally found that with a $\frac{1}{4}$ per cent. solution nearly the full theoretical proportion of gold was extracted, or considerably over 100 oz. per ton. One point upon which the authors had given no information was the effect of the decomposition products. He assumed that they had overcome the difficulty which used to arise by the continued formation and accumulation of those products, which occurred to such an extent, even with a weak solution, as to make it impossible to attempt to re-use the solution. One point had a special interest for him. On page 961 the authors referred to the aerocyanide process as being inapplicable to certain ores, particularly mentioning Transvaal concentrates. Mr. Butters, the great authority on chlorination, had, however, himself stated that cyanide was slowly but surely displacing chlorination for all purposes on the Rand, even for the treatment of concentrates. He himself felt convinced that bromo-cyanide had its special function, but he thought the authors were mistaken in claiming for it any superiority for the treatment of simple ores. Had they restricted their claims to special classes one might have agreed with them, but for simple ores general experience was against them. If one took two ordinary ores and ran them side by side, one with bromo-cyanide and cyanogen, and the other with plain cyanide, the result would be a greater decomposition of cyanide where the bromo-cyanide was present. On the other hand, if one took a special ore—one that contained marcasite, for example—then the decomposition might be greater with the plain cyanide, owing to the longer time required for treatment by the latter method. The reason for this was that the addition of bromo-cyanide had the effect of increasing the solution rate of the gold, and thus lessening the period of contact during which decomposition took place. They were told that with a particular ore—that of Day Dawn—the consumption of cyanide was 4 oz., and the consumption of bromo-cyanide was an additional $1\frac{3}{4}$ oz. But he knew more than one instance where the consumption for such tailings was 4 oz. of cyanide alone, without using any bromo-cyanide at all.

Mr. Sulman: What would the time be in that case?

Mr. James: Four to five days.

Mr. Sulman: What we wish to make clear is that by using the $1\frac{3}{4}$ oz. additional, you save four out of those five days, and surely that is worth while there.

Mr. James, continuing, said this had been tried, and had not been considered worth that. The extra $1\frac{3}{4}$ oz. of bromo-cyanide doubled the cyanide cost. He might almost say that extractions by plain cyanide from those same Deloro ores had been obtained as good as that the authors had stated in their paper. It would be observed that the consumption at Deloro was very heavy, without taking the bromo-cyanide into account at all; and he would like to ask the authors whether they had ever calculated how much of the BrCy was left at the end of 30 to 40 hours. He thought that the general experience was that it had all disappeared as bromo-cyanide long before that time. With regard to the zinc process, he considered the cone described in the paper a very ingenious way of getting out of their difficulties. One weak point, however, was that whereas zinc shavings would act as a filter and prevent the gold going off, Mr. Sulman had to resort to some mechanical arrangement, hung on poles around the vat, to prevent the loss of the hydrogen-buoyed bullion particles. Then the authors had made a point about the simplicity of their process in the clean-up. He himself thought it a disadvantage to the process to have to clean up once a day, or to add anything to it every two hours. The advantage of being able to run a box for a month without cleaning up was very great. As to running 400 gallons through the apparatus in an hour, he might point out that it was not at all unusual to run 6,000 gallons through a set of zinc shavings boxes in that time. With regard to the fineness in gold, the use of zinc fume was not altogether new; they had tried it before the shavings, but not perhaps so scientifically as Mr. Sulman, and they got so much zinc with the bullion that they discarded it in favour of shavings. Mr. Sulman said that the fume contained 30 per cent. of gold, but at Deloro it contained only 10 per cent., and apparently much other metal. In spite of all the difficulties of the old zinc process, they had no difficulty, in more than one place, in turning out bullion giving 960 fine, without acid treatment.

Prof. H. E. Armstrong, F.R.S., regretted that the authors had not dealt more at length with the theoretical side of their process. He considered that the Society was one peculiarly fitted for discussing the higher aspects of industrial processes, and recommended the researches of Maclaurin, already referred to, as model investigations.

Mr. H. L. Sulman, in reply to Prof. Armstrong's observations, said that the paper they had originally presented to the Society had undergone considerable compression, in order to bring it within the limits of the time at their disposal that evening. Why they had given more prominence to the practical aspects of the process was that when their first paper was read at the Institute of Mining and Metallurgy they were met with the objection that it was then mainly theoretical, and were advised to show what

they could do on a practical scale. They had endeavoured to follow that advice. They had since devoted their efforts mainly towards the practical details of the process, and had that evening brought the results achieved before this Society, assuming that those who had heard their former paper were practically agreed as to the validity of its theory.

As examples of practice they had given results obtained upon two entirely dissimilar types of ore, one of which had resisted every other process, and its goldfield, in spite of trials for 30 years, had remained unworked. The chlorination mill formerly put up was now a ruin. On the other hand, the bromo-cyanogen process had now been at work there for eight months past, and was economically extracting the gold: and the plant for that process was now to be enlarged. He thought that was a fair proof that they had accomplished what they had originally claimed.

Mr. Crowder, Mr. Claudet, and others had asked why they were not at work in South Africa. They were not, for this reason. The great Rand mines with their very large capital responsibilities were naturally very difficult places upon which to get experiments tried. Before they would consent to disorganise their works even to the extent necessary for practically investigating a new process, they would require previous practical demonstration of its success on a similar scale elsewhere. Therefore they had gone further afield, and as a result they now had proofs of successful working in Canada, Australia, and Hungary, and were thus in the position to answer such requirements. Mr. Louis had inquired respecting the time factor and the consumption of cyanide. Mr. James had given a partial reply to this in pointing out that cyanide would extract the gold from certain refractory ores after prolonged action, but with such a consumption of cyanide that process was not profitable; on the other hand, the bromo-cyanogen process was extracting such at a profit; shortly, cyanide was inapplicable to heavy arsenical pyritous ores, whereas bromo-cyanide was successful. On the question of the fineness of the bullion, that which they obtained at Deloro averaged from 750 to 820 fine, which he thought would be considered distinctly good from an ore of that character. He would be glad of Mr. James's opinion on that point.

Mr. James: Yes, 820 fine bullion would be very good work.

Mr. Sulman, continuing, said that they had been asked whether they had made parallel comparative tests upon vat charges running under both processes. The same argument applied to them in that case as to the Transvaal mines. They were under a contract to extract certain ores economically, and they had applied their energy to this and not to demonstrating the superiority of bromo-cyanide by lengthy comparative experiments. They had, however, already quoted the results of a large vat experiment, in which they had shown that the small amount of $1\frac{3}{4}$ oz. of bromide of cyanogen per ton of 0·1 per cent. cyanide liquor yielded complete extraction of a tailings in 14 hours, whereas when by greatly decreasing this addition the solution approximated to plain cyanide, more than two days was necessary. At a present cost of about 2*s.* 6*d.* per pound for cyanogen bromide, that meant a charge of about 2*d.* per ton for this reagent, and was the sum representing the saving of the four days referred to by Mr. James.

By the use of the $1\frac{3}{4}$ oz. of cyanogen bromide the time necessitated was reduced to 14 hours; and as the wages of a common labourer on the Murchison fields at the time was 4*l.* per week, he thought it would be agreed that it was well worth while to incur the cost of between 2*d.* and 3*d.* per ton for the additional bromide than to run up a far greater time bill. Mr. Claudet had said that in order to get a fair comparison in the experiment referred to, they ought to have added a weight of cyanide equal to the bromide of cyanogen added, but as that would mean an addition of only 0·005 per cent. more of potassium cyanide, this was a quantity which he thought they might very safely neglect. Mr. Claudet's other remarks has been adequately dealt with by Mr. Harland.

Mr. James and others had referred to the possibility of loss from the zinc cones, but he thought rather too much stress had been laid upon the occasional "buoyed-up" flocks of material. When precipitation took place, two or three particles of gold-zinc couples would sometimes hang together, entangling a bubble of hydrogen gas, and would rise to the surface, discharge the bubble, and the metallic particles would thereupon at once fall back to the cone bottom. In order to prevent any possible loss from the floating over of these, they hung round the cone a narrow strip of calico, which acted as a coarse strainer; but as Mr. Picard had recently found that no such loss occurred in practice, the screen had latterly been dispensed with. He would like to ask Mr. James whether, when he talked of the rate of flow being 6,000 galls. in the ordinary cyanide process, he meant those figures to apply to one series of boxes only.

Mr. James: I made use of the word "series" so that there should be no misunderstanding; I meant a series of four to six boxes.

Mr. Sulman, continuing, said that under those circumstances the rate achieved at Deloro with one cone would compare well with the rate of liquor flow through a zinc-box. As to the equation they had put forward, he could only say it had been adopted by many experts well known in connection with this subject as the most probable explanation of what actually occurred. Amongst others, Prof. Christie had practically adopted it in his well-known paper, and he believed he might quote Mr. James as also accepting it.

In reply to Mr. de Mosenthal, he was glad to say that bromo-cyanide had already superseded plain cyanide solutions on two mines, and on a third cyanide had been altogether inapplicable. As to the question of royalty, it must be a foregone conclusion that only such a royalty could be charged which would still leave a good margin of advantage in favour of the adoption of the process over and above that effected by any other process (and its royalty amount) with which it competed.

Mr. Sulman regretted that he had omitted to reply to Mr. James's question as to the interference of certain inferred decomposition products. He was pleased to say that such had not been found to occur nor to hinder them in the least, even in dealing with the highly arsenical ores at Deloro. In proof of this, they had used the same liquors there for four months straight away, and these were the same liquors that carried them through all their first more or less experimental trials. The total decomposition of the solution was, of course, accurately measured by the additional amounts of bromo-cyanide and cyanide it was necessary to use in each case in order to again produce liquor of the normal strength. As already pointed out, it was not advisable or necessary in cases of refractory ores to make the whole addition of bromide of cyanogen at once to the liquor charge pumped on the crushed material, but to apply it in two or three smaller quantities.

Dr. F. L. Teed, in further reply to the discussion, said that, with regard to the question of precipitation, it was obvious that since they could run one cone at a certain rate they could run cones in parallel series, and so without difficulty treat any quantity of material.

All alternate theories of the action of the solvent hitherto proposed involved hydrolysis and the formation of a cyanate. The fact that cyanate did not assist the solution of gold, as proved by Maclaurin, was a sufficient condemnation of such theories. The equation proposed in the paper has been accepted by Prof. Christy and Mr. Alfred James, among others.

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SESSION 1897-98.

Wednesday, Jan. 26th, 1898.—Dr. J. T. Conroy. "Some further Experiments on the Manufacture of Cyanides."

Meeting held on Wednesday, November 24th, 1897.

DR. C. A. KOHN IN THE CHAIR.

THE MAGNETIC CONCENTRATION OF SO-CALLED NON-MAGNETIC MATERIAL.

BY J. H. DARBY.

As far as the writer is aware, no account of the Wetherill concentrating machine and the material suitable for treatment has been given in this country, although in February, 1896, Messrs. H. A. J. Wilkens and H. B. C. Nitze contributed a paper to the American Institute of Mining Engineers on the subject. The writer is indebted to Mr. Wilkens for much of his information.

The title of this contribution may seem paradoxical, but it is usually understood that a substance is magnetic if it is attracted or repelled by a permanent magnet. The magnetic machine invented by Mr. Wetherill goes a step further, and is so powerful that not only can magnetic materials as usually understood be separated, but substances which apparently have no effect on the magnetic needle are readily concentrated. This opens up the economic treatment and concentration of materials that have hitherto been partially effected by washing or smelting.

Faraday and others have shown that magnetism is a property common to all substances, solid, liquid, and gaseous; but in by far the majority of cases the manifestation of this force is very feeble. Para-magnetic substances are attracted, dia-magnetic repelled. The latter represent the larger class. Dia-magnetic substances show but slight differences in magnetic capacity, but fortunately, on the other hand, para-magnetic substances vary within a wide range. Faraday's list published in 1846 gives the following metals as para-magnetic: iron, nickel, cobalt, manganese, chromium, cerium, titanium, palladium, platinum, and osmium.

Attempts have been made to determine the relative specific magnetic permeability of various substances, with more or less success; and to give some idea of how widely this varies, I may state that, according to Delesse, the magnetic permeability of—

Soft steel is	100,000
Magnetite	65,000
Siderite	12?
Hematite	93 to 13
Limonite	72 to 43

Of the substances named, steel and magnetite alone have hitherto been called magnetic. No practical use, as far as the writer knows, has been made of the magnetic property of substances other than those which were so marked as to respond to the hand magnet, such as iron, nickel, cobalt, magnetite. It was only after Mr. Wetherill took up the matter, perfected and patented his machines, designing them after many experiments, so as to present various minerals to electro-magnets having practically a saturated field, and built in a peculiar way, that he was able to separate and concentrate many minerals that it had been previously impossible to treat magnetically.

Many attempts, more or less successful, have been made to concentrate magnetite, and amongst others I notice the elaborate plant designed and erected by Mr. Edison, at Edison, New Jersey, where he crushes down the run of a mine containing about 20 per cent. of magnetite, and by a series of machines and crushings concentrates the magnetite so that the final samples contain as much as 68 per cent. of iron, and at the same time the percentage of phosphorus is sensibly diminished, while the waste of iron in the tails is very small.

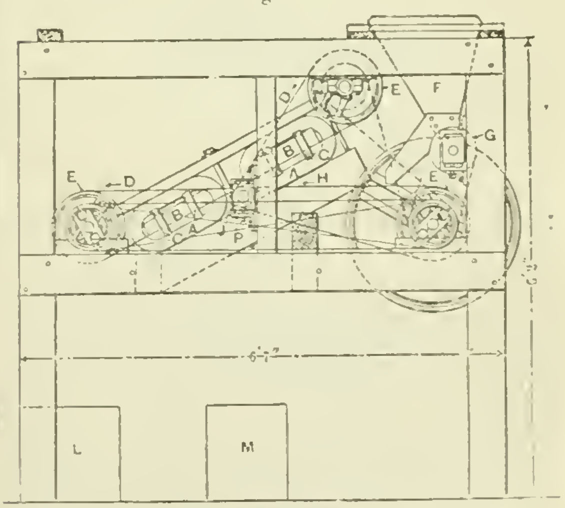
In order to render iron ores susceptible to a magnet, recourse has been had to preliminary roasting, but this is not altogether satisfactory, as at times part of the gangue, which may contain mineral, becomes more or less magnetic, and is found in the concentrates instead of the tails. As examples of the above may be mentioned the treatment of limonite ore, suggested by Clemens Jones; the treatment of the red phosphoric ores of Alabama, by Dr. W. B. Phillips.

I cannot do better than give you in Mr. Wilkens' own words the following description: "Mr. J. P. Wetherill, manager of the Lehigh Zinc and Iron Co., South Bethlehem, Penns., found that not only franklinite, but also garnet and similar iron- and manganese-bearing minerals, could be treated by direct magnetic concentration.

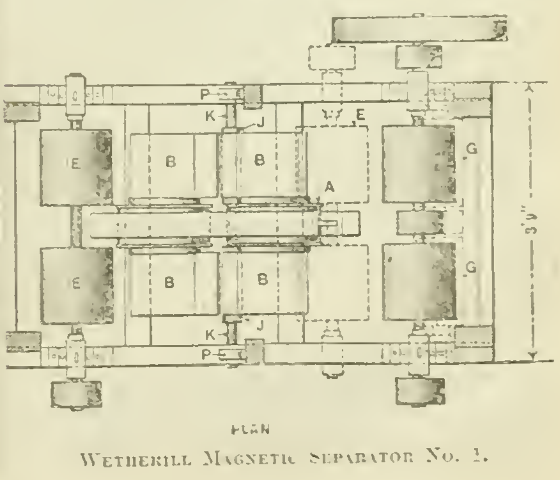
"These surprising and important results were attained by utilising a peculiarly high magnetic power, applied in machines especially adapted for the purpose, and sufficient to act upon these minerals of almost infinitesimal magnetic permeability, instead of attempting (as had heretofore been done) to raise this low permeability to a point known in practice as 'magnetic,' that is, capable of attraction by a common hand magnet, or the electro-magnet heretofore employed.

"Further investigations showed that a large number of ferruginous and manganiferous minerals, such as hematite, limonite, siderite, garnet, pyrolusite, &c., besides many

Fig. 1.



ELEVATION



PLAN

WETHERILL MAGNETIC SEPARATOR No. 1.

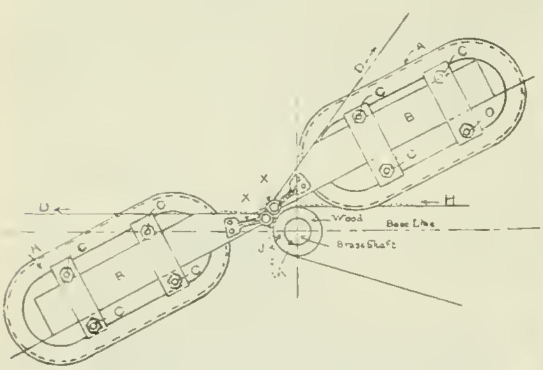
chemical salts of iron, manganese, and chromium, were affected by this extraordinary magnetic power, and were thus adaptable to this method of concentration."

Letters patent were granted to Mr. Wetherill for his method of direct magnetic separation of para-magnetic substances of low permeability from mixtures containing the same, as well as for the machines employed to obtain this result, and these patents are now owned by the Wetherill Concentrating Company.

Method and Apparatus.—The machines for making these separations are of several types. Figs. 1 and 2 show a machine more especially adapted for the treatment of fine ores and for concentrations in which rich magnetite heads are more particularly sought. It has been operated very successfully on the Clinton fossil ores of the Birmingham district, Alabama.

In this type the magnetic particles are lifted away from the original mixture. The machine consists of two magnetic cores and bobbins A, and four pole-pieces B, which are pointed in the manner shown, and are adjustable by the bolts C, so that they may be moved nearer or farther apart, as desired.

Fig. 2.



Enlarged Section from Fig. 1, showing Arrangement of Pole-pieces and Feed-belt.

For substances of very low magnetic permeability two of the pole-pieces are dispensed with, and a solid yoke is used instead. For substances of higher magnetic permeability, such as garnet, franklinite, siderite, &c., the yoke may be removed and two pole-pieces substituted for it in the manner shown. About each pole-piece revolves a belt D (driven by a pulley E) in the direction shown by the arrows.

The ore is fed from a hopper F by means of a feed roller G, upon a belt H, which carries it in a thin layer, say ⅛ to $\frac{3}{16}$ in. thick, to and about the pulley J, which is of small diameter, and is upon a brass axle K, which may be raised or lowered by the adjustable bearing P. The feed can be accurately adjusted by means of a sliding shutter at the discharge of the hopper F.

The ore is thus delivered in close proximity to the space between the poles, and the magnetic particles are withdrawn and lifted up into the highly intensified field existing at this point. They are removed by the horizontal belt D, and carried into a receptacle L.

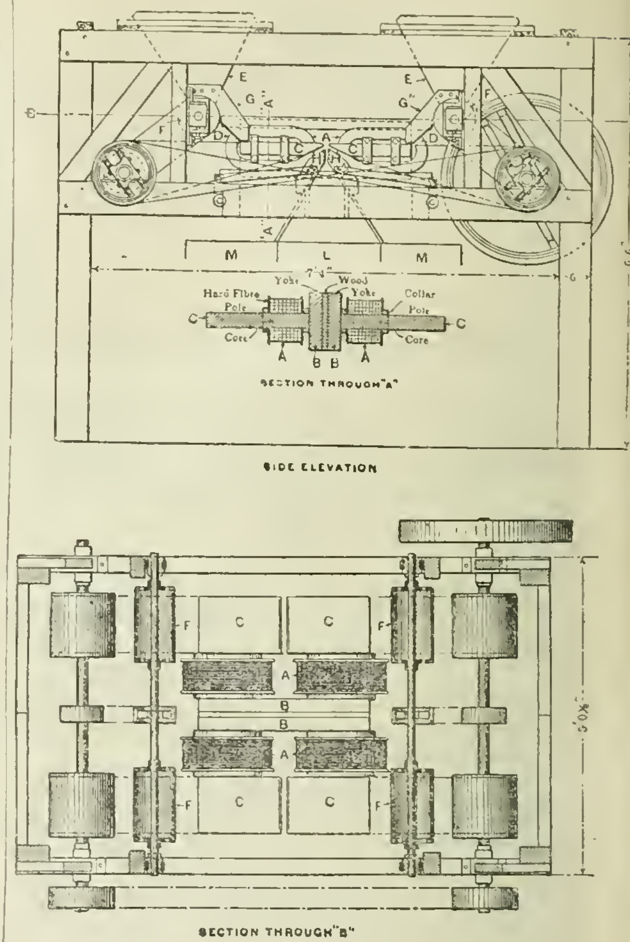
The non-magnetic tails fall from the belt H into the receptacle M.

The intensity of the magnetic attraction can be accurately adjusted by changing the distance of the feed-belt from the pole-points, or by changing the distance between the pole-pieces, or finally, by changing the strength of the current.

Fig. 3 shows a machine adapted more especially for coarser sizes, material as large as ½ in. in diameter has been handled with it. Excellent results have been obtained by its use on franklinite ores, where clean non-magnetic tails, consisting of willemite and zincite, are sought.

In this type of machine the magnetic particles adhere to the feed-belt as it rounds the pole-points, while the non-magnetic material drops away from it. The apparatus

Fig. 3.



Wetherill Magnetic Separator No. 2.

consists of magnets with cores and bobbins A, yokes B, pointed pole-pieces C, belts D, revolving about the pole-pieces in the manner shown, hoppers E, feed rollers F, and shutes G, by means of which the ore is fed in a thin layer upon the belts D. These belts travel in the direction shown by the pointed arrows, and the ore is delivered directly into the opening between the two pointed pole-pieces; means are provided by which the pole-pieces may be set further apart or closer together, as desired. Two shutters H, one beneath the point of each pole-piece, are adjustable, so that the magnetic particles which adhere slightly to the pole-pieces are carried to one side by the moving belts D, into the receptacles M and N on either side of the shutter, while the non-magnetic fall into the central space between the shutters and thence into the receptacle L.

In the third type of machine the material to be treated is fed on to a belt which passes in the horizontal plane through the gap formed between the ends of soft iron poles, which above and below the gap are encircled by powerful solenoids. Above the material so brought within the influence of the magnet, but still between the pole-pieces, another belt runs at right angles, and as the magnetic portions are lifted from the lower belt against the upper, they are carried forward by the cross belt and deposited in a hopper arranged to receive them.

"The degree of concentration desired can be regulated by changing the position of the shutters and the speed of the feed-belts, or by changing the distance between the pole-points, or by changing the amount of current through the coils."

So far, machines have been used with a capacity of from , to 3 tons per hour, depending upon the number of pole-pieces used, the speed of the belts, and the nature and size of the material operated on. By widening the pole-pieces and the feed-belts, the capacity can be raised; but this may ultimately reach a point where it will be more advantageous to multiply the number of machines instead of further increasing their size.

The amount of current necessary for producing the extremely intense field is comparatively small. This is due to the large area of magnetic circuit, correct proportion of the pole-pieces employed, and the almost perfect condensation at the pole-points of the vast number of magnetic lines produced in them.

For the separation of the Franklin minerals, where the magnetic heads produced consist of franklinite, tephroite, rhodonite, garnet, &c., from 3 to 8 ampères, with from 16 to 30 volts, are necessary. In the concentration of the Clinton red fossil ore from 4 to 8 ampères were used. For limonite and pyrolusite a somewhat larger current is employed, from 10 to 15 ampères giving a good separation.

"To illustrate the extreme condensation of magnetic lines at the pole-points, it may be mentioned that the current of one Daniell cell (such as is used in telegraphy) is too powerful to permit a satisfactory separation of magnetite. For this purpose its trength must be reduced by means of resistance coils, in order to prevent tangling, and allow the belts to draw the ore away from the pole-points.

"The ability to adjust delicately the magnetic intensity of these machines permits in many cases the practical isolation from one ore of several products showing a slight difference of magnetic permeability."

There is also another machine arranged with a series of magnets, where the space between the material and the magnet differs, and where the intensity of the field also differs. The strongly magnetic portion is picked out by the first magnet, less strongly magnetic portion is picked out by the second magnet, and so on. The final tails are by this method thoroughly cleaned from all magnetic heads, and the magnetic heads are graded according to their varying degrees of permeability, all being conducted in one machine.

"If two such unlike properties as magnetic permeability and specific gravity can be compared, it might be said that a much smaller difference in the former is necessary to permit a separation by magnetic means than would be required in the latter for making a water or air separation."

It would be difficult, however, to tabulate the minerals in distinct groups capable of being magnetically separated from each other, for the reason that the magnetic permeability of each mineral is, *per se*, subject to wide variations.

"Aside from the separation of several minerals from one ore, the discriminating power can be utilised for the separation of a desired quantity or quality of heads where the magnetic mineral varies in itself in permeability. For example, in the concentration of the Clinton fossil ores, the magnetic permeability of the hematite particles increase apparently with their percentage of iron, and either a small amount of high-grade heads can be produced with a low current, or a large amount of low-grade heads with a higher one. From a commercial standpoint, the exact quantity or quality which it would be advisible to produce would depend upon the increase in value of the concentrates per unit.

"The machines above described and illustrated are adapted to dry concentration. Their arrangement for wet concentration would necessitate but a slight change in some of the mechanical features."

As far as the writer knows, all iron-bearing minerals, with the exception of the sulphides, are capable of separation by the machines just described, and can be successfully concentrated. In the manufacture of iron and steel it is an acknowledged fact that, with one or two exceptions, the high-grade ores formerly used in this country are gradually becoming leaner, and are associated with a larger percentage of inert material. This has to be passed through the furnace and slagged off, consuming additional flux, coke, and labour. The economic assembling of iron-making materials—that is, ironstone, limestone, and coke—is of great importance, and it is obvious that if an ore can be concentrated at the mine, before it has to pay railway carriage or freight, it will command a higher value, and if this is sufficient to cover the cost of treatment and still leave a margin of profit, the operation will be carried out with advantage. On the other hand, it must be borne in mind that it is often necessary to reduce iron ores to a small state of division, in order to free them from the accompanying gangue, and to render them suitable for magnetic treatment. Iron ores in a small state of division have not been looked upon with any great favour by furnace managers. In America modern plants have been designed to treat the rich ores from the Mesabi district, near Lake Superior, containing 40 to 50 per cent. of very fine material, and it is an acknowledged fact that the magnificent plants in Pittsburg and Chicago treat them with astonishing results, and without the troubles which have usually been associated with finely divided ores in this country. In the writer's experience finely divided ores may be economically treated in the blast furnace under suitable conditions, and if there is sufficient pressure of blast available to force the air through the high column of materials, which in the finely divided condition offer more or less resistance to the reducing gases. The writer feels sure that with proper apparatus there will be no difficulty experienced in the manipulation of a considerable proportion of finely divided rich concentrates.

For the purpose of ascertaining how far results similar to those mentioned might be obtained by treating ores in the United Kingdom, arrangements were made with the Wetherill Concentrating Co., and subsequently an experimental machine was erected by the writer for the Brymbo Steel Co. There seems to be a possibility of concentrating the aluminous ores of Antrim, and tests have been made in this direction with some prospect of success. Should these be borne out, the heads and tails will each have a value of their own—the heads from an iron-maker's point of view, and the tails from their high percentage of alumina.

There is a mineral in Cornwall containing tin and wolframite, both of which are valuable, but the difficulty in separation has hitherto greatly reduced the market value. The mineral can be readily treated by the magnetic separator, and thus the value of the separated minerals is greatly enhanced.

The writer has recently experimented with a mineral mined in Mexico, but treated in this country. As imported it contains a large proportion of garnet associated with copper ore. It has to be brought down from the mine on the backs of mules, and between the mine and the smelting works in England has to pay a very high carriage. By passing it over the No. 1 separator, with current, pole-gap, speed, &c., suitably adjusted, as much as 40 per cent. of the imported bulk can be picked out as valueless garnet, at a small cost, and the same quantity of mineral can be conveyed by the costly method described by carrying 60 per cent. of the original weight. Samples of the original material referred to, garnet separated, and the concentrated copper ore as obtained by the writer, are exhibited.

To treat this mineral efficiently in the magnetic machine it was found necessary to grade it. Thorough mechanical separation of the garnet from the mineral was not effected until the whole passed through a sixteenth-inch mesh. It was then found advisable to divide what passed through the sixteenth mesh into two portions; that is to say, through a sixteen, and on thirty, and through thirty. These portions were then treated in the No. 3 machine, and the magnetic heads, or garnet, separated.

The assay shows that the mineral concentrates have been increased, giving—

——	Cu.	Ag.	Au.
	Per Cent.	Ozs. Per Ton.	Ozs. Per Ton.
60 per cent. of mineral concentrates.	37·7	56·7	3·02
40 per cent. of garnet heads separated.	1·5	2·6	0·02

The loss in copper and other valuable minerals is therefore not as much as it would be in smelting, and the saving in carriage is very large. It must not be understood that it is necessary in all cases to "break down" a mineral as fine as the above example, but when dealing with the rare metals a very small loss in mineral has to be guarded against. The mineral in question had to be broken down for concentration by washing, and it only meant that the larger portions had to go back to the rolls before passing the magnetic machine.

The applications of this machine no doubt can be extended beyond what is known at the present time, and a hope is expressed that this incomplete communication will be of interest to the members of the Society.

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Manchester Section.

Rooms of the Chemical Club, Victoria Hotel.

Chairman: George E. Davis.

Vice-Chairman: D. Watson.

Committee:

B. W. Gerland.	J. Lewkowitsch.
J. Grossmann.	A. Liebmann.
J. M. Irving.	A. Ree.
E. Knecht.	E. Schunck.
M. J. Langdon.	C. Truby.
I. Levinstein.	W. Thomson.

Hon. Local Secretary:
J. Carter Bell,
Bank House, The Cliff, Higher Broughton, Manchester.

SESSION 1897–98.

Friday, Jan. 7th, 1898.—Mr. J. Carter Bell. "The Oxygen Test for Sewage and other Effluents, as carried out in the Laboratory of the Mersey and Irwell Joint Committee."

Meeting held on Friday, December 3rd, 1897.

MR. GEORGE E. DAVIS IN THE CHAIR.

INDIGO TESTING BY PERMANGANATE.

BY J. GROSSMANN, PH.D.

In a paper which I read in Bradford in May (this Journal, 1897, 701) I gave a *résumé* of the methods proposed for the testing of indigos. I there drew attention to a passage in Mr. C. Rawson's paper on the testing of indigos by permanganate (*ibid.* 1885, 489–491) in which he recommends those who are not well versed in chemical manipulation, and desire to determine the value of a sample of indigo to use the permanganate process, *viz.*, ordinary sulphonation and direct titration with permanganate. He says further on, "The results obtained by this method are somewhat too high, yet they give one a fairly approximate idea of the relative values of different samples under examination."

Referring to this passage I said :—

"No doubt with indigotine and the highest class of Bengal and Java the results obtained are fairly accurate for comparative purposes, but with lower qualities the results are quite unreliable and may vary 20 per cent. or more." In making these remarks I did not overstate the case. In the discussion which followed, Mr. Rawson passed over my criticism on the direct titration with permanganate and went on to say that *after* purification there was in his opinion no better method of estimating indigotine in indigo than by means of potassium permanganate, thus shifting his ground by introducing another test which was neither the one which I criticised nor another proposed by Mr. Rawson and known as the salting-out test.

Again, in summing up the merits of the different tests which he has investigated, Mr. Rawson says :—

2. The permanganate method affords a quick and ready means for the approximate valuation of indigos; but as substances soluble in dilute acids are, at the same time, more or less acted upon, the results obtained are somewhat too high.

3. If the solution of indigo be saturated with sodium chloride the colouring matter is thereby precipitated. When the precipitate is washed, dissolved in dilute sulphuric acid and extracted with potassium permanganate, results are obtained which for all practical purposes are trustworthy and reliable. Indigo red and indigotine are simultaneously estimated by this modified process.

Now I quite agree with Mr. Rawson's opinion about the modified permanganate process, under 3; if used in conjunction with my dyeing test it gives results by which no buyer of indigo can possibly go wrong. But as regards the direct method and his remarks on it I entirely dissent on two grounds. In my opinion persons not well versed in chemical manipulation have no business with chemical analysis. In gravimetric analysis it is usual for a competent analyst to examine whatever precipitate he has weighed to make sure that it is pure and the actual substance he wanted. In the case of volumetric tests, where such subsequent verification cannot be made, chemists who employ them should be, if anything, still more competent. An incompetent person using the direct permanganate test might describe a mixture of sawdust and iron filings coloured blue with some aniline colour as a high class Bengal indigo. My second ground of objection is that there are only two kinds of methods which should be used in analysis, *viz.*, such methods which are correct within almost scientific limits and such methods which though not being absolutely correct have a constant error. Thus a method which in each case would only give, say, 95 per cent. of the actual quantity present would be quite admissible for commercial work, as by dividing the result by 95 the test would be quite accurate. But a test which showed at various times from 80 to 95 per cent. of the actual contents would be absolutely worthless, as whatever result were obtained would convey no information. Sometimes it might be right other times 5, 10, 20, or more per cent. wrong. The direct permanganate test is of this type. Take Mr. Rawson's own figures and compare them with each other; the following table will show you that they differ from the modified test by from

Per Cent. of Indigotine by Direct Permanganate Test.	Per Cent. of Indigotine after Precipitation with Salt.	Percentage Error by Direct Permanganate Test.
76·32	73·82	3·40
76·05	73·27	3·80
66·71	63·50	5·05
62·85	57·74	8·85
62·32	57·30	8·76
62·66	57·50	8·97
50·04	44·90	11·50
47·15	43·30	8·90
47·15	42·90	9·90
39·50	37·40	5·61

3·4 per cent. to 11·5 per cent. In other words, by using the direct permanganate method you may be able to judge within 6*d.* per lb. what an indigo is worth. It does not require a chemist to do that; any judge of indigo could come nearer the truth by the appearance of a sample.

My own experiments, however, show that by this test you cannot even tell to a shilling per lb. what indigo is worth. The following table gives the results of a number of samples of high class indigos tested by the direct and the modified permanganate process:—

Per Cent. of Indigotine by Direct Permanganate Test.	Per Cent. of Indigotine after Precipitation with Salt.	Percentage Error by Direct Permanganate Test.
69·1	67·8	1·9
53·4	49·8	7·2
62·9	59·4	5·9
62·1	59·0	5·2
64·6	62·0	4·2
53·4	48·9	9·2
59·0	55·0	7·2
62·9	58·6	7·3
68·2	62·0	10·0
66·4	61·8	7·1
59·7	55·3	7·9

The difference in the tests of these varies from 1·9 per cent. to 10 per cent.

In the lower qualities, the difference is considerably more marked, as the following table will show:—

Per Cent. of Indigotine by Direct Permanganate Test.	Per Cent. of Indigotine after Precipitation with Salt.	Percentage Error by Direct Permanganate Test.
43·2	39·7	8·8
42·0	35·8	17·3
41·9	36·2	15·7
47·2	45·0	4·9
46·3	43·7	5·9
44·2	43·3	2·0
34·5	33·6	2·7
45·9	44·1	4·1
43·3	40·1	8·0
43·7	42·4	3·1
43·3	39·3	10·2
45·4	44·5	2·0
37·6	36·2	3·9
37·2	27·6	34·8
46·3	43·2	7·2
39·7	35·8	10·9
38·0	34·1	11·4
41·9	39·2	6·9
43·1	38·8	11·1
34·5	33·6	2·7
45·8	44·1	3·9
38·8	37·0	4·9
42·4	38·0	11·6
44·2	41·9	5·4
50·1	44·4	12·8
42·1	40·8	3·2
48·1	40·5	18·7
47·7	45·0	6·0
30·2	25·6	18·0
48·1	45·5	5·7

This table represents the tests of Kurpahs, Oudes, and similar classes of goods, and I have given a large number of samples because these qualities are largely used in Lancashire for dyeing cotton yarns and piece goods. The tests are from 2·4 per cent. to 34·8 per cent. too high, and invariably against the buyer. They show conclusively that the direct permanganate test is worthless. The tests which I have given are not selected samples, but have been copied at random from my laboratory notes.

There is one more variety of test with permanganate which I may discuss so as to complete the subject; it consists in extracting the sample of indigo in succession with weak acid and caustic soda or ammonia, then drying and dissolving in sulphuric acid and titrating direct with permanganate. The results are disappointing and such as do not warrant me to recommend this method.

Per Cent. of Indigotine by Direct Permanganate Test.	Per Cent. of Indigotine after Extraction with Acid and Caustic Soda.	Percentage Error by Direct Permanganate Test.
48·3	48·3	0·0
48·6	47·1	3·2
46·8	43·1	8·6
48·4	44·9	7·8
49·8	48·0	3·8
45·6	41·9	8·8
49·6	46·8	6·0
43·7	42·1	3·8
57·0	53·3	6·9
69·5	66·8	4·0
70·4	70·4	0·0
74·4	71·6	3·9
66·5	62·5	6·4

From the table which I have given it will be seen that although in some cases the results are exact, in others they are nearly 9 per cent. wrong, and against the buyer.

The conclusion which I draw from these experiments is that the only permanganate test which should be used for commercial testing of indigos is the one proposed by Mr. Rawson in which the sulphindigotate is previously thrown down with salt. This method, save in exceptional cases, gives reliable results; in conjunction with my dye test it is perfectly reliable. I may also add that the instructions given by Mr. Rawson for dissolving the indigo in sulphuric acid should be scrupulously adhered to, otherwise the results will be unsatisfactory.

Discussion.

Dr. Gerland said, of the chemical substances which were formed they had no idea. The quantity of oxygen consumed certainly did not agree with the formation of isatin or any other known compound; but, on the whole, he agreed with Dr. Grossmann as to the testing of indigo. He had often tried salting out. If salt were added to saturation, and the solution allowed to stand overnight, all the indigo had gone out of it, and it was left perfectly clear. If, on the other hand, they followed Rawson's advice, and precipitated with a certain amount of salt, avoiding saturation, then came the difficulty of making allowance for the indigo afterwards left in solution, which was unsatisfactory. The salted-out indigo sulphonate did not show the disappearance of the blue colour sharply enough to use it for the hydrosulphite titration.

Dr. Green referred to W. Brylinski's paper on methods of testing indigo given in Monit. Scient. (this Journal, Nov. 1897, 938), in which preference was given to Engler's method.

Dr. Grossmann, in reply, said that in using Rawson's salt test it was necessary to use the exact quantities recommended by Mr. Rawson. If Dr. Gerland used more salt than Mr. Rawson recommended, he would get wrong results; but as Mr. Rawson had examined the subject thoroughly, and worked out the right proportions, there was no reason why anyone should deviate from the quantities which he gave. The salting-out method was a very reliable one. In a few exceptional cases it might not give accurate results, but when used in conjunction with the dye test it was absolutely reliable. The paper referred to by Dr. Green had escaped his notice, but, so far as he could learn from Dr. Green's remarks, there was only this difference, that Mr. Engel proposed a vanadium volumetric test in conjunction with a dye test, and he advocated Rawson's salting-out permanganate test in conjunction with his dye test.

STEAM, GAS, AND ELECTRICITY, AS SOURCES OF ENERGY IN CHEMICAL WORKS.

BY F. H. BOWMAN, D.SC., F.R.S.E., ASSOC. INSTS. C.E. AND M.E., AND M.I.E.E., ETC. ETC.

Power in chemical works does not occupy so important a position as it does in textile industries, where the whole of the transformation is effected by mechanical power. In chemical works it is principally confined to subsidiary

operations, such as grinding and pulverising raw materials, working pumps for the purpose of lifting the various liquids, compressing air or other gases, mechanical furnaces, grinding, centrifugal drying machines, making boxes or barrels, actuating cranes, or in the form of locomotives for haulage in the yards.

In the carrying out of any works with which I have had to do I have always endeavoured to avoid mechanical operations; and when these were necessary, the machine was made as simple as possible. Chemical works abound in dust, dirt, gases, or liquids which injure machinery, wherein lies the difference between them and a textile factory. It is thus advisable to use the very simplest forms of engine which can stand the rough wear and tear to which they have to be subjected.

Chemical works usually cover a large area of ground, and it is not possible to centralise the power, because operations are rarely simultaneous, and often far from one another, and small independent engines have to be relied on.

Even under the best conditions the steam engine is a wasteful machine. The source of loss arises in many parts. First of all there is the inefficiency of the boiler itself. The boiler ought to be as simple as possible, because it is usually tended by unskilled labour, has to work night and day, and is more liable to strain by continuous working than if it could be stopped and attended to oftener.

In my opinion there is no boiler equal to the two-flued Lancashire. It is not as efficient as the locomotive type, or even the externally-fired tubular type; but it is the simplest, requires little attention, is easy to clean and repair, and will burn—if the draught is properly regulated and the size of the grate area properly designed—with every kind of fuel. For an internally-fired boiler, in charge of a skilful fireman, hand-firing will meet all the requirements, but in large works it is advisable to have mechanical stoking, because there is the same efficiency at all hours, and this more than compensates for the extra expense of the mechanical stokers, which, moreover, tend to the prevention of smoke, and an increased economy. Mechanical stokers are of two kinds—coking stokers and sprinkling stokers. The coking stokers are fed continuously at the front, and the evolved gases pass over the hot fuel at the back of the grate, and this I have found to be the best where the work is continuous.

Mactear furnaces in the ammonia-soda process, or the salt pots and furnaces in the Le Blanc process, which require a continuous flame, cannot be worked efficiently if it is necessary to consume all the fuel, and hence where the works are large enough it is advisable to mix the unburnt fuel from these furnaces with a small portion of good slack, and then use it for steam raising with a forced draught either by use of the fan, or, what is simpler still, the steam jet properly arranged, which makes the furnace practically into a blacksmith's hearth.

A pound of ordinary Lancashire slack or Burgy coal will evaporate from 8 lb. to 9 lb. of water when the boilers are free from scale and the water sent in at near boiling point.

A Lancashire boiler fitted with an economiser almost equals a tubular one, but the latter does more work in the same space, and though it costs more to keep up it saves expenditure in plant.

With an efficient boiler and first-class engines the cost of delivering power at the fly-wheel shaft amounts to 10*l.* per indicated h.p., or, say, 5,000*l.* for a 500 h.p. engine. This includes a moderate price for the area of land upon which the plant stands. An average pound of hydrogenous coal will give about 14,500 heat units, and about 10,800 out of the 14,500 are delivered as steam to the cylinder. If we allow about 8 per cent. for ash we may say that 1 lb. of coal will practically yield about 10,000 heat units. This represents about 7,720,000 ft. pounds of energy, and if this could all be utilised in a steam-engine, this 1 lb. of coal would represent roughly 4 indicated h.p., working for one hour, and this would be about a $\frac{1}{4}$ lb. of coal per indicated h.p. per hour. Unfortunately no steam-engine will accomplish this. The very best marine engines consume about 2 lbs., while ordinary condensing engines use from 3 to $3\frac{1}{2}$ lbs. of coal, taking 1 lb. of coal to represent about 9 lb. of water evaporated. The best triple expansion engines measured by the quantity of steam which they use, consume from 12 lb. to 14 lb of steam per indicated h.p. per hour. Compound condensing engines use from 15 to 18 lb. of water; single cylinder condensing engines from 20 to 25 lb., and high pressure engines, varying with the steam pressure and the size of the engine, from 20 to 50 lbs. Some of the numerous engines that are found in chemical works doing the pumping under the ordinary conditions of open air and other disadvantages, and so frequently of such a small size, would be found to use even more than this.

If we raise steam with 1 lb. of coal with the very best appliances our results would be something as follows:—

	Per Cent.
Loss by imperfect combustion, radiation from the furnace, and heat carried up the chimney	20
Heat lost by radiation from the boilers, pipes, cylinders, &c.	2
Carried away into the condenser or sent out as waste in the exhaust steam, if a high pressure	63
Converted into indicated h.p. in the cylinder	15

Now, as we have no engine that will work with 1 lb. of coal per indicated h.p. per hour, it means that we do not and cannot at the present moment utilise 85 per cent. of the energy in the coal.

In large chemical works, power has to be distributed over miles of pipes exposed to the condensing action of the air, and hence a very large percentage of inefficiency must be added to that which has already been mentioned. I doubt in many of our small engines whether we actually use 5 per cent. of the power which was originally in the fuel.

Now comes the question of cost per indicated h.p. per annum. From a series of experiments with a compound condensing engine of 500 h.p., the cost of which was, roughly, 5,000*l.*, the cost came out as follows:—

	£	*s.*	*d.*
Interest on plant, 10 per cent.	500	0	0
Wages	160	0	0
Cleaning and repairing boilers and brickwork	90	0	0
Oil, grease, boiler composition, &c.	80	0	0
Repairs, and keeping up of engine, including packing	100	0	0
Rates, taxes, and insurance	100	0	0
Sundries	50	0	0
	1,080	0	0

The boilers were Lancashire two-flued boilers working at 100 lb. pressure, which is as high as I should recommend for chemical works. The boilers were 8 ft. diameter by 30 ft. long, with five tubes in each flue and a set of two Green's economisers.

This gave for power, without water or fuel, about 2*l.* 3*s.* 3*d.* per indicated h.p. per annum. The coal was good slack, 6*s.* 8*d.* per ton, delivered in the firing floor; and the amount of coal consumed, including banking and other purposes connected with the power, but not including the heating of the works, which was supplied from a separate boiler, gave 4·5 lb. of coal per indicated h.p. per hour; the ashes and banking represented about $\frac{1}{2}$ lb., so that the efficiency of the engine was about 4 lb. per horse. The coal cost about 2*l.* per indicated horse, per annum, making a total cost per h.p. per annum, 4*l.* 3*s.* 3*d.* This is about $\frac{1}{3}$ of a penny per indicated horse per hour on a working week of $56\frac{1}{2}$ hours per week, and 50 weeks per annum. The engine under test conditions gave under 2·83 lb. of coal per indicated h.p. per hour, measured by the actual steam used, as calculated from the indicator diagram; but the tests were taken of the cost based on five years working. This was working the ordinary factory hours; and in chemical works where 24 hours are worked, there is not the banking to take into account; but I very much doubt whether an equivalent of steam delivered to the engines of any chemical works costs less than this.

Steam is not a good vehicle for obtaining power, because it can never be a perfect gas unless absolutely dry; and the moment it begins to expand—which it must do if worked economically—it ceases to be a dry gas.

I thought at one time of using producer gas. I put down producers, but found that to keep up a constant supply of gas it required a series of producers rather than storage space in a gas-holder as this enabled me to equalise the flow of the gas, and utilise the heat of the gas at the time of its generation in the producer. I also found that it was useless in two-flued boilers, as a large portion of the efficiency of the boiler arises from radiation from the fuel, whereas the gas flame was non-luminous, and its efficiency as a steam raiser was greatly reduced. If complete combustion were not attained and the flame thereby rendered luminous, it deposited on the surface of the boiler enormous quantities of soot, which seriously interfered with the efficiency of the heating surfaces. If the flame was made to impinge on refractory material, such as filling the flues partially with bricks stacked so as to leave interstices between them, the efficiency was increased. But my experiments led me to the conclusion that for the use of gaseous fuel, tubular boilers were absolutely essential, so that the whole of the flame might be brought into intimate contact with as large and as thin an absorbing surface as possible.

For ordinary steam-raising I believe the present method of using solid fuel is far the best and the most economical. Where gas is a by-product it practically costs nothing. If gas is to be used as a source of power, we must look for its utilisation to the gas-engine; and already in this direction results have been reached which exceed in economy the use of steam.

In first-class gas-engines an indicated h.p. has been obtained on a large scale with rather less than 1 lb. of coal per hour, which is about 50 per cent. more economical than the best steam-engine. Gas-engines, however, are complicated and intricate, and this renders them unsuitable for chemical works, although if kept in a separate building and then worked with a mixture of water gas and producer gas, I believe that they might come in at far less than the cost of steam. On the other hand, in nearly every part of a chemical works, steam is required for purposes other than power, and the pipes that convey the steam can readily be drawn on for power. For power alone the gas-engine is a most efficient machine, especially for the production of an electric current. I myself have obtained power in this way at less than half the cost of steam, and with a 200 or 300 h.p. plant it is possible to produce an indicated h.p. at a cost of not more than 30s. per annum. The cost of a gas plant and gas engines complete may be taken at about 12*l*. per indicated horse, so that the original cost is about 20 per cent. dearer than a steam plant, but it is 50 per cent. more efficient, so that the first cost need not be considered. Thus a 500 h.p. plant for steam being 5,000*l*., a similar gas plant would be 6,000*l*., but a saving of only 2*l*. per h.p. per annum would repay the extra outlay in 12 months and represent a saving of 1,000*l*. per annum ever afterwards. This is quite as low as any figures I have been able to obtain with regard to water power, either here or on the Continent. We have so little water power in this country that it is hardly necessary to discuss its merits, but it becomes an item to be taken into calculation when electrical processes are to be used and power becomes a considerable item.

I understand that in Germany an indicated h.p. can be produced by water at 50 marks per annum, and in some other parts of the Continent and in America at 2*l*.; but water power is often distant from the centre of distribution, and also from places where coal can be obtained, and thus the cheapness of the power is more than balanced by increased cost of fuel and carriage.

In chemical works, electric distribution is almost impossible. Dust and gases have great effect on conducting wires and electric motors. I have used lead-covered wires with a coating outside, and yet they were eaten through in a short time; and, except where motors can be enclosed in special rooms or boxes and conducting wires kept out of reach of corrosion, electric distribution of power is not suitable for such works. With electric chemical processes the conditions are different, but I shall not deal with them to-night, as possibly they may form the subject of a future paper.

Electrical distribution can best be applied in chemical works to lighting. You can obtain at least twice the quantity of light by the arc lamp than by burning gas or even Wells' lights. To compete with steam as a motive power, electricity must be supplied at not more than ½*d*. per Board of Trade unit, or half this cost to compete with gas.

Nottingham Section.

University College, Nottingham.

Chairman: J. O'Sullivan.

Vice-Chairman: J. M. C. Paton.

Committee:

L. Archbutt.	W. G. Johnston.
S. F. Burford.	J. F. Kempson.
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Treasurer: S. J. Pentecost.

Hon. Local Secretary:

J. T. Wood, 29, Musters Road, West Bridgeford, Nottingham.

Meeting held on Wednesday, November 24th, 1897.

CHAIRMAN'S ADDRESS.

THE UNORGANISED FERMENTS, OR ENZYMES CONCERNED IN THE BREWING AND DISTILLING INDUSTRIES.

BY JAMES O'SULLIVAN, F.I.C., F.C.S.

When food is consumed by animals it is not, as is well known, always in a condition suitable for absorption, and before this can take place it must be changed by the complex process of digestion in which each organ of digestion performs its allotted functions. The digestion in highly organised animals, of which we are able to speak with certainty, is entirely external or in other words, it takes place outside, for the alimentary canal is simply a continuation of the external surface. Digestion also takes place in the vegetable kingdom, and, although there are instances of external digestion, interior or interstitial is the most prominent. When we come to the germination of seeds and the growth of bulbs and tubers we have exclusively only to deal with interstitial digestion.

The varied food consumed by man requires for its digestion seven or eight soluble ferments, or enzymes.

In the brewing and distilling industries the enzymes which bring about the solution or digestion, of the substance of a seed, play a most important rôle and I propose in this address to consider what, from a practical point of view, is known of their action.

A grain of barley, the seed almost exclusively used in the industries mentioned, consists of embryo and endosperm, which are, broadly speaking, composed of starch, sugars, cellular matter, albuminoids, cellulose, fat, and mineral matter. These reserve materials, which are mainly situated in the endosperm of the grain, are almost all insoluble. Now the young plant in order to avail itself of this insoluble material must either be provided with, or it must provide for itself, a means of rendering it soluble for use during its growth.

It appears that it was known in the early years of the present century that wheat contained a soluble ferment possessing the power of dissolving starch. Kjeldahl (1879) and Lintner and Eckhardt (1889) studied the properties of diastase found in barley, and compared its action on soluble starch with that of malt-diastase. In 1890 (Trans. Lab. Club) I found this ferment in the endosperm of barley, maize, rye, wheat, and oats. Lintner and Eckhardt found

that in its hydrolytic power barley-diastase was more active at low temperatures than malt-diastase, which showed a difference between them. This information about the hydrolysis of "soluble" starch did not, however, explain how the starch granules (comprising about 50 per cent. of the total weight of the grain) were made soluble during the germination of the barley. Brown and Morris ("The Germination of some of the Gramineæ," J. Chem. Soc. 1890) named the less active diastase of barley "diastase of translocation." It does not liquefy starch paste, nor does it erode starch granules.

The endosperm of barley is made up of a network of cellular matter which encloses the starch granules. It follows from this that as the hydrolyst in the ungerminated grain does not dissolve the starch granule that some active agent must come into existence before the starch of the endosperm can be dissolved. The cellular structure in which the starch granules are enclosed must itself be dissolved. That this does take place has been shown by Brown and Morris who have demonstrated that the cellular network enveloping the starch granules is dissolved by diastase, prepared from air-dried malt after the manner in which diastase from kiln-dried malt was prepared by C. O'Sullivan (J. Chem. Soc. 1884). The body thus prepared has been proved to possess cyto-hydrolytic as well as amylo-hydrolytic properties. It is well known that the starch granules of barley are eroded and dissolved during germination.

As the diastase "translocation diastase" already present in the barley, does not possess the power of eroding starch granules the agent which does this must be produced during germination. I proved (Trans. Lab. Club 1890) that on the third day of germination there is present in the endosperm of germinating barley sufficient diastase to completely dissolve the starch when digested at a temperature of 65° and this was observed prior to any visible growth of the embryo.

Baranetzkey in 1878 found that the starches of grain were eroded by malt diastase. It is interesting to mention that malt diastase has not this action on potato starch (C. O'Sullivan, J. Chem. Soc. 1876) which indicates that the diastase of the germinating potato differs from malt-diastase.

Brown and Morris established that the diastase produced during germination is a secretion of the absorbtive epithelium of the embryo and have named it "diastase of secretion."

Here then are the two enzymes which play the most important part in rendering soluble and changing the material of the grain, to the required extent, in the preparation of malt. One a cyto-hydrolyst possessing the power of dissolving cellular matter (cellulose) the other an amylo-hydrolyst possessing the power of dissolving starch.

When barley is germinated the proteids which are insoluble are made soluble. However, at the present time, we are not in a position to speak more definitely about a proteid-hydrolyst than to infer that a somewhat similar process of solution of the proteids takes place to that of the carbohydrates.

I have proved (Trans. Lab. Club, 1890) the presence of invertase in the acrospire and rootlets of the embryo barley plant, which is unquestionably concerned in the assimilation of the metamorphosed constituents by the plant. This observation is, I believe, highly important, supported as it is by evidence which I have at hand of the presence of invertase in all portions of the gramineæ from seed to plant.

The enzymes we have up to this considered are only treated from the point of view of the production of malt, which is the first and not the least important of the processes involved in the brewing and distilling industries.

The diastase of secretion plays another and a most essential part in the solution of the starch of malt in what is called the "mashing process."

C. O'Sullivan in a series of classical papers (J. Chem. Soc. 1876—79) published the results of his work, "On the Action of Malt Extract on Starch," and showed that when starch is dissolved by malt extract—diastase—there are produced maltose and dextrines. Since 1879, although he has been active in other spheres of research, he has ceased to contribute to this subject, which is very much to be regretted. However, the method of following up the transformation of starch was established by him, and has led to numerous and valuable contributions on the subject.

Hertzfeld in 1879 proved the presence in starch transformations of a body to which he gave the name of maltodextrin, a combination of a maltose group and two dextrin groups. In the same year Brown and Heron (J. Chem. Soc. 1879) published on this subject and corroborated in the main the results of O'Sullivan.

Brown and Morris in 1890, from a vast amount of work, supported by practical observations, put forward the following hypothesis of the solution of starch by diastase:—That when starch is dissolved by diastase there are produced maltose, free dextrin, and varying types of malto-dextrin or amyloins.

Maltose is the final product of the action of diastase on starch, and the free dextrin is only hydrolysed very slowly (C. O'Sullivan, J. Chem. Soc. 1876, 127), while, according to Brown and Morris the amyloins, which are produced with the free dextrin, are rapidly hydrolysed to maltose.

I have not drawn a distinction between the brewing and distilling industries as to the use made of the enzymes. There is, however, a difference. In brewing, the aim is to restrict the action of the diastase to suit the requirements, whereas in distilling the object is to obtain its highest action, so as to produce the maximum amount of fermentable sugar.

I give in the following table a scheme of the enzymes concerned in the brewing and distilling industries up to this stage of the process—the production of a "fermentable wort."

Table of enzymes which we know are concerned in the production of brewers' and distillers' wort:—

Origin.	Enzyme.	Properties.
Barley	Diastase of translocation.	Does not erode starch granules or liquefy starch paste, but dissolves "soluble starch."
Germinated barley (malt).	Cytase Cyto-hydrolyst.	Dissolves cellullar matter and is rendered inactive on heating. Therefore is not present to any extent in kiln-dried malt.
" "	Diastase of secretion (Amylo-hydrolyst).	Liquefies and hydrolyses starch paste at all temperatures up to 80°.

The distiller does not dry his malt at as high a temperature as the brewer, nor does he boil the wort prior to fermentation, in fact his object is to obtain the highest possible diastatic action. The diastase of the wort is present during the alcoholic fermentation in the distillery, and continues to hydrolyse the non-fermentable starch products of the wort to fermentable maltose. In consequence of the presence in distillers' wort of the free dextrin, which is only slowly hydrolysed, there is almost invariably to be found in distillers' "wash" from 18 to 20 per cent. of the original malt solids unfermented, of which 5 to 6 per cent. is dextrin, and apparently maltose.

Although brewers' and distillers' yeast contains the well known enzyme-invertase, which inverts cane sugar, we are not able to say what function this fulfils in the fermentation of malt wort, other than the inversion, prior to fermentation of the small amount of cane sugar usually present.

In the fermentation of cane sugar it is first hydrolysed to dextrose and levulose, and these sugars are always present in solutions of cane sugar undergoing fermentation. Maltose, which chiefly composes the fermentable sugar of wort, is fermented without its hydrolysed product being noticed; it is in fact directly fermented. This led many to look upon the fermentation of maltose as being analogous to the fermentation without previous hydrolysis of cane sugar by monilia candida. Fisher (Ber. 1894, 271) has, however, shown that from air-dried brewers' yeast a maltose-hydrolysing enzyme can be extracted, to which he gave the

name "beer yeast-glucase." This work of Fisher in establishing the presence within the yeast cell of a maltose-hydrolysing enzyme, together with what we already know of the absence of its hydrolysed product, dextrose, during fermentation, and of the hydrolytic functions of yeast, as shown by me (J. Chem. Soc. 1892, 593) adds considerable weight to our belief that hydrolysis and fermentation takes place within the yeast cell.

The following are the enzymes which we know are concerned in the fermentation of a brewer's wort :—Invertase and beer yeast-glucase, and to these in the case of the distillers' wort we have to add diastase.

We must not leave the question of translocation diastase without referring to another and important part it plays in the brewing industry. In 1878 Baranetzky drew attention to the fact, which he observed, that diastase was present in all portions of plants in which he looked for it. He found it in seeds, tubers, stems, and in the leaves. Brown and Morris (Trans. Inst. Brewing, 1893) showed that dry hops which are added by brewers to finished beers contain diastase, which has the power of hydrolysing to maltose the amyloins, and the free dextrin in beer thus supplying a fermentable sugar for continuing the after or cask fermentation.

I have not given analyses of these enzymes, although there are many published, nor have I considered their properties, only so far as these relate to the technical processes in which they are concerned.

All analytical evidence proves that they contain nitrogen, and are not albuminoids, while it also shows the difficulty which attends their preparation in a pure state. It is needless to say that until an amorphous substance is proved to be homogeneous, its analysis is not of much use, and to add to this we have in the case of enzymes the greatest difficulty of all, *viz.*, "that of proving their purity without altering the properties upon which their designation depends." We are forced to the conclusion that we do not know with certainty the composition of these most important products of life.

Discussion.

Dr. J. J. Sudborough said that two diastases had been mentioned in the paper, namely, that of translocation and that of secretion. It was a matter of regret that these two evidently distinct substances should have received the same name. If one could be altered it would be a desirable thing to do in order to prevent confusion. In a recent article in the Berichte (1897, **30**, 2289) Wroblewski has shown that the ordinary diastase of malt as obtained by Lintners' method can be separated into two distinct compounds, namely, a solid carbohydrate, which on hydrolysis yields arabinose, and a proteid-like substance, which alone has any diastatic action. Wroblewski has suggested that the enzymes are closely related to albuminoids, and are probably to be regarded as a sub-group of the proteids.

Did the Chairman know whether any other chemists besides Stavenhagen had repeated Buchner's work on the isolation of zymase from yeast (Ber. 1897, **30**. 117, 1110, 2422; this Journal, 1897, 548 and 1030); and if so, with what results? If Buchner's work were confirmed, it would certainly be one of the most important discoveries in connection with fermentations that had been made for many years, as it seemed to indicate that fermentations induced by organisms were really fermentations by means of enzymes. Was not the enzyme, described by Mr. O'Sullivan as beer yeast-glucase, termed maltase by Fischer, who first isolated it?

Dr. F. Clowes (the President) had recently spoken with a high authority on the work of Buchner, who seemed to look upon his results as not proved. It was remarkable that no confirmation of the work had appeared from other investigators, and that the changes producible by other organised ferments besides yeast had not been shown by Buchner or others to be producible by an unliving enzyme derived from the ferment itself. He was not aware that Buchner's work had been either confirmed or extended in the way referred to.

The Chairman in reply said that, as the diastases prepared from barley and malt had been observed to possess different properties, Brown and Morris gave them names which showed at once their origin. It would be advisable in order to avoid confusion, when speaking of a preparation of diastase, to state its source, as for example, barley-diastase, malt diastase. Maize-malt diastase, a body prepared from maize-malt, in a similar way to barley or malt-diastase, possessed the power of hydrolysing maltose to dextrose (Geduld's glucase). Fischer had pointed out the importance of indicating the origin of enzymes, and when he discovered that air-dried beer yeast yielded a maltose hydrolysing enzyme he called it beer yeast-glucase.

It had been known for sometime that malt diastase, as usually prepared, contained a nitrogenous body and a carbohydrate, but it had been found most difficult to obtain these in a pure state. Wroblewski's work was certainly an addition to our knowledge, but it must be considered doubtful whether he had been dealing with a pure diastase; his numbers for nitrogen in his preparations appeared to show that he was not; his analysis in fact were as varied as any yet published. He was not aware of any English chemist having repeated Buchner's work. However, Stavenhagen and Pritzkow did not confirm Buchner's sensational announcement, and it was highly improbable that his work would ever be corroborated.

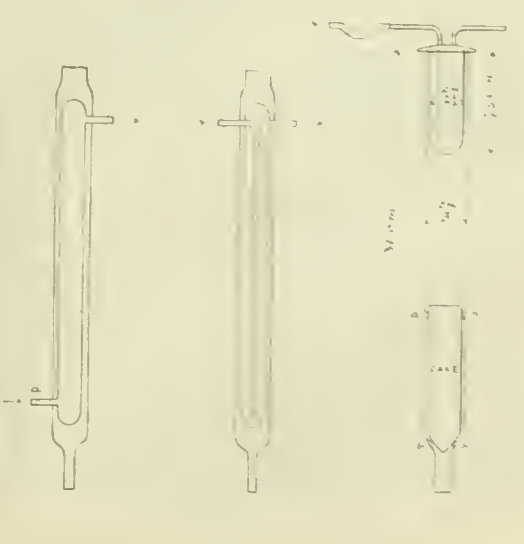
REFLUX CONDENSERS.

BY J. J. SUDBOROUGH, PH.D., D.SC., AND M. E. FEILMANN, D.SC.

Mr. Feilmann explained that in some of their work the authors required to boil ethereal and alcoholic solutions for several hours on the water-bath, using a reflux condenser. With the ordinary Liebig and bulb condensers they always found that the large amount of moisture deposited on the outer surface trickled down over the cork and was liable to permeate into the flask. In order to avoid this condensation on the outer surface the authors suggested the apparatus (Fig. 1), which had been constructed for them by Messrs. George, of Birmingham and London. In this apparatus the cold water was made to pass through the inner tube and the ether or alcohol vapours were condensed in the annular space between the inner and outer tubes. By this means the outer surface of the condenser was kept warm and the deposition of moisture avoided. For use with ether they had found the apparatus a great improvement on the usual form of condenser, as in addition to the cold water surface it also utilised the cooling action of the air, and condensation was therefore very complete.

For high-boiling liquids the authors suggested the following form of apparatus (Fig. 2), as in the apparatus first described there would be a great strain on the inlet tube (*a*) through extremes of temperature, causing a liability to crack.

FIG. 1. FIG. 2. FIG. 3.



The authors, of course, claimed no originality in principle for the apparatus proposed by them, as an inner cold-water condensing surface was already in use in the well-known metallic Soxhlet condenser.

DISCUSSION.

Mr. JOHN GOLDING said that being in Copenhagen last summer, he was shown by Dr. Storch the apparatus which he used, and which, although different, yet embodied the same principle as that described by the authors. There were 50 of these appliances going in the laboratory in two sets of 25. They were all working with ether, and although he smelt for it, he could detect none escaping. An ordinary combustion tube some 31 cm. long and 3 cm. internal diameter was drawn out to fit the cork of the boiling flask. The condenser, which was made of thin white metal, was $2\frac{1}{2}$ cm. in diam. and about $7\frac{1}{2}$ cm. long, and rested on the top of the tube by the projecting rim of the cover as shown in Fig. 3. This left an annular space of $\frac{1}{4}$ cm. between the condenser and the tube. This must be very cheap to make. He might mention that the oil cakes were extracted, after drying in a current of hydrogen or coal gas to prevent the oxidation of the oil which would otherwise take place. The cake, in the form of a cylinder, was kept away from the sides of the tube by projections in the interior of the latter, made by depressing the glass from the outside, as shown at *a* and *b* in the figure. This left a space which allowed the ether vapour to freely circulate round the cake.

Mr. R. M. CAVEN said that there was considerable loss with the ordinary Liebig condenser when working with ether, and if the apparatus described prevented this it would be a decided advantage.

The CHAIRMAN remarked that the great difficulty with all these condensers was to get the ether to permeate the whole of the substance under treatment. The drop only went through the middle of the mass leaving portions at the sides insufficiently acted upon.

Mr. J. GOLDING: Dr. Storch found no difficulty of the kind with his arrangement. They were left to act for four hours and could be left for six or eight.

The CHAIRMAN said that was a long time, but he had found an hour not long enough.

Mr. G. J. WARD suggested that to prevent the drop always falling in the same place the bottom of the condenser might be made flat.

Dr. SUDBOROUGH, or it might have a number of glass balls attached to the bottom.

Mr. TROTMAN said it was certainly necessary to have an apparatus that could safely be left to look after itself. For instance, he had known a case where after returning from lunch the ether flask was found dry.

Prof. CLOWES said that in Dr. Thorpe's London laboratories they had refrigerating arrangements so that they could supply iced water to the condensers. Such a plan necessarily reduced the losses very considerably. Closing the top of the condenser he also thought would prevent loss as there must be diffusion by contact with the atmosphere.

Mr. FEHMANN replied that they had boiled ether vigorously under it and found very little smell at the top.

The CHAIRMAN suggested that if the inner tube (Fig. 1) instead of being plain had a number of constrictions the apparatus would probably be found to be still more efficient.

Meeting held at the Midland Hotel, Derby, on Wednesday, December 15th, 1897.

MR. G. J. WARD IN THE CHAIR.

THE RECOVERY OF SULPHUR FROM HYDROGEN SULPHIDE.

BY I. J. R. CARULLA, F.C.S.

I.—IN the manufacture of sulphate of ammonia from gas-liquor and in other chemical operations, sulphuretted hydrogen is evolved. It is not permissible to send it into the atmosphere through high chimneys, except after burning and the products of combustion being considerably diluted, the Alkali Works Regulation Act fixing the limits of acidity for such gases from chemical works at 4 grains per cb. ft., which must on no account be exceeded.

Various methods have been adopted from time to time to get rid of this noxious gas, and, at the same time, obtain the sulphur in some useful form, either combined or in the free state. One of the earliest was to burn it in air, sending the products of combustion into sulphuric acid chambers.

It is at once evident that 33 per cent. of the air employed is used for the conversion of the hydrogen into steam, and consequently the corresponding amount of inert, useless nitrogen, which only takes up space and diminishes the working capacity of the chambers, has to be admitted into them. This, together with the carbon dioxide present, so reduces the yield of vitriol from a set of chambers as to render the process one that is somewhat expensive. Apart from this, however, it is not at every works where hydrogen sulphide is produced that vitriol chambers are available, and doubtless these considerations led Mr. Claus to devise his ingenious kiln for the recovery of sulphur in the free state from this gas (Eng. Pat. 3608, July 29, 1882).

No one who has seen the splendid results obtained with the Claus kiln, especially by the United Alkali Co. at Widnes, can have any doubt about its success. It is, therefore, not without some hesitation that the author brings before you a method which, although it cannot supersede the Claus kiln, may yet, under special conditions, be considered to have certain advantages over the older plan.

My process is to burn the hydrogen sulphide in an atmosphere highly impregnated with sulphur dioxide, whereby 50 per cent. more sulphur can be obtained with the same amount of hydrogen sulphide than is possible by the Claus method, according to the following equation:—

$$2H_2S + SO_2 = 2H_2O + 3S.$$

The practical result of the operations in the Claus kiln, leaving out intermediate reactions and only keeping in view what goes in and what comes out of it, may be represented by this equation:—$2H_2S + 2O = 2H_2O + 2S$, clearly showing how the new process gives so much more sulphur with the same quantity of hydrogen sulphide.

The reaction can be made to take place in an ordinary pyrites kiln, into which the hydrogen sulphide is properly admitted, with due regulation of the supply of air to the kiln. Or a combustion chamber may be provided, the two gases being led into it by separate passages.

The sulphur, which sublimes, can be collected in brick chambers in the same manner as is done in the Claus process. These, however, will require less provision for the drainage of water in proportion to the amount of sulphur made.

II.—The above portion of this paper was written in the autumn of 1893, after the author had obtained provisional protection for improvements in the manufacture of sulphur (Brit. Pat 15,424 of Aug. 14, 1893).

It may be interesting to explain how the idea of the process was arrived at.

At the works with which the author is connected, the H_2S from the sulphate of ammonia saturators used to be burnt in a pyrites kiln of the old pattern, having a long hearth. The conversion of the H_2S into SO_2, which went forward into the vitriol chambers with the gas from the pyrites, was effected without any cause for complaint. It was decided to replace the old kiln by new pyrites burners on the modern pattern. The nozzle for burning the H_2S was introduced through the front wall of the new burners, some half way between the top of the pyrites bed and the roof of the kiln. The presence of sulphur at every available opening proved that some detail had been misunderstood. The admission of air to any reasonable extent seemed insufficient to abate the trouble, which, however, was eventually overcome, although it took some considerable time to exactly find the actual requirements in this respect.

It was natural that one should try to understand the cause of the extraordinarily copious production of sulphur referred to. The author became convinced that, in the

absence of sufficient air, the reaction that has been explained between the H_2S and the SO_2 was the true cause. Seeing, further, the excellent character of the sulphur produced by this accidental process, it seemed quite possible to advance a step on the Claus process, as has been already mentioned. The specimens of sulphur before you were deposited in the tunnel leading the gases from the pyrites burners into the chambers. They will convince you of what could be done by the use of proper appliances when such an excellent material was the result of mere accident.

It will naturally be asked, Why was so promising a process not gone on with? To rely on the reaction as a protection for the patent was of course out of the question. The reaction coupled with particular sources of the two gases seemed more promising; still, it appeared to be better to depend on some special design of the plant.

The writer therefore waited for the time when he might be in a position to make the experiments necessary to take out such a patent.

Probably this matter would not have yet come before you had it not been that Messrs. Brock and Hurter have obtained protection for what is practically the invention that has been described to you (Brit. Pat. 13,844, June 23, 1896), an abstract of which appears in the Journal of our Society, Vol. XVI., p. 536. The object of this paper is in no way to institute any prior claim, but rather to show the great value that may attach to the invention, and to bear witness to the excellence of the product and likely efficiency of the process.

One must suppose that Messrs. Brock and Hurter's claim is founded on the plant and apparatus, although a superficial glance at the abstract in our Journal might lead one to infer that they relied for its protection on the reaction between the two gases concerned.

This forms a telling illustration of what can happen with our present patent laws. Formerly, assuming that the invention of Messrs. Brock and Hurter had been the same as that of the author, it could not have stood, as the latter would have been published, even though only provisional protection was accorded to it. At the present time, however, inventions that are only provisionally protected remain secret, and the consequence is the possibility of an inventor who has patented a process, but who has allowed his privilege to lapse after provisional protection, finding that he cannot use his own process without the permission of a subsequent patentee. When the provisional specifications were published after the abandonment of a patent this could not happen, but of course the whole world was also at liberty to use the invention.

There is, however, another and very different point of view. A patentee who finds that he has made a serious blunder in a provisional specification, can now drop it and take out another patent without disclosing anything to the public or to his rivals that might lead to his being anticipated. Nor could anything that he may himself have said in his abandoned specification do him any injury when taking out a new patent. This might well happen, and in fact, to the author's knowledge, did happen on one occasion when the specifications of patents that had only been granted provisional protection were published.

Discussion.

Mr. R. M. Caven said that the reaction between H_2S and SO_2, by which sulphur was liberated, was well known as a lecture experiment. He supposed, however, that the object of the paper was to show its application to a practical purpose. He did not see that the proposed process made made any more sulphur from the H_2S than the Claus process did. The extra amount was obtained by using something that added to the total of the sulphur-containing gases.

Mr. L. Archbutt asked what part the oxide of iron took in the experiment described. Was the Claus kiln mentioned in the abstract of Brock and Hurter's patent an essential?

Mr. Adams said it seemed unnecessary to use any pyrites. The SO_2 might be obtained by burning a portion of the H_2S in air and mixing it with the other portion of the H_2S.

The Chairman remarked that, although only remotely related to the subject, he might mention that they had been recently accused of emitting H_2S from the top of some blast furnaces. Although H_2S would no doubt be generated within the furnace, he failed to see how it could remain as such in the flame at the top.

Mr. Carulla, replying, said it was quite true that material containing sulphur had to be supplied in order to get, in process described, the extra yield over the Claus process. As, however, such material could be obtained very cheaply, the objection had little force. At the Sharon Chemical Works, for example, they had hundreds of tons of pyrites smalls from coal brasses, which, although containing some 30 per cent. of sulphur, were merely cumbering the ground for want of some such process. He would add that the difficulty of carrying on experiments with these gases was very great, owing to the stringency of the Alkali Act. He felt quite confident that plant could readily be put up to render the recovery of the sulphur contained in the smalls referred to commercially successful, but there was always the danger that it might not be possible to do it without a greater escape than the Act allowed, *viz.*, 4 grains. Indeed, he might say that this was only a legal limit, for, as a matter of fact, the average escape from their vitriol chambers would not be more than half that figure. So low was it at times that the last test taken by Her Majesty's Inspector was only 0·3. To hope to approach such figures with experimental plant was out of the question, and the fear of normally exceeding the Act limit, after spending a considerable sum, and being unable to come within it without such further expense as to render the process a losing one, was decidedly a deterrent. In reply to Mr. Archbutt, he did not think that the oxide of iron took any part in the reaction between the H_2S and the SO_2 in the pyrites kiln. The burning pyrites emitted SO_2, which met the H_2S being fed into the kiln some distance above the bed of pyrites. Certainly in the abstract of the patent to which reference had been made (this Journal, Vol. XVI., p. 536), it was stated that Brock and Hurter passed the mixture into a Claus kiln, so that possibly in that case the oxide of iron did take some part in the reaction. The process mentioned by Mr. Adams had been before suggested for obtaining sulphur from H_2S, but in that case the assistance that the process described in the paper gave to get sulphur from an otherwise worthless pyrites was sacrificed.

THE ESTIMATION OF CUPROUS OXIDE BY MEANS OF STANDARD POTASSIUM PERMANGANATE SOLUTION.

BY R. M. CAVEN, B.SC., F.I.C., AND ALFRED HILL,

University College, Nottingham.

One of the authors was recently engaged in the investigation of the composition of a mixture of cuprous oxide with metallic copper and an insoluble basic cupric salt, and it was found desirable to estimate the proportion in which the cuprous compound occurred, by means of a volumetric method depending upon its oxidation to the cupric state. The use of chlorine, bromine, or iodine in any form for this purpose is inadmissible, because, when a solution of a cupric salt is brought into contact with potassium iodide and starch, such as would be used as an indicator in connection with the above oxidising agents, a blue colour is at once produced, owing to the liberation of free iodine, consequent upon the formation of cuprous iodide.

Some preliminary qualitative experiments showed that an acidified solution of potassium permanganate readily oxidises a cuprous compound to the cupric state; and we have succeeded in making use of this reaction for the estimation of cuprous compounds, and more especially of cuprous oxide.

A solution of cuprous chloride in hydrochloric acid is completely oxidised by a solution of potassium permanganate, but considerable difficulties have to be overcome in the adaptation of this process for quantitative analysis. The reason for these difficulties is twofold.

Firstly, the solution of cuprous chloride very readily undergoes oxidation on exposure to air, and the results would therefore be low from this cause.

Secondly, the use of hydrochloric acid in connection with potassium permanganate solution is liable to be attended with oxidation of the acid itself and the evolution of chlorine. This result would introduce a positive error into the determination; and indeed, unless the oxidation of the hydrochloric acid is prevented, the end of the reaction is rendered very uncertain by the gradual fading of the permanganate solution, after a supposed fixed point has been reached.

The first of these difficulties has been overcome by excluding air from contact with the solution during the titration, by means of a current of carbon dioxide gas. The second difficulty was met by employing a solution of magnesium sulphate in conjunction with the hydrochloric acid, and always carrying out the titration under similar conditions.

The most favourable conditions for success were ascertained by a series of preliminary experiments, with different proportions of acid, magnesium sulphate solution, and water. Air-free water was used throughout the experiments, both for dilution and for the preparation of the solutions of hydrochloric acid and magnesium sulphate.

The following is the manner in which we have carried out the volumetric estimation of cuprous oxide according to the above principle.

The cuprous oxide, from whatever source obtained, having been thoroughly washed with hot water, to remove any soluble impurities adherent to it, is received in a stoppered tube of 100 c.c. capacity, and a little water is added to it. The air is then expelled from the tube by means of a brisk current of carbon dioxide gas, and during the passage of the gas, dilute hydrochloric acid (1 part strong acid to 3 parts water) is added from a pipette in sufficient quantity just to dissolve the cuprous oxide. The mouth of the tube is closed and the contents gently agitated to effect complete solution. The liquid thus obtained should be quite colourless.

Meanwhile about 300 c.c. of cold, air-free water are placed in a beaker of 450 c.c. capacity, together with a quantity of magnesium sulphate solution, containing 300 grms. of the crystallised salt per litre. The volume of this solution used must not be less than double that of the dilute hydrochloric acid employed to dissolve the cuprous oxide.

In most of our experiments from 20 to 25 c.c. of dilute acid and 50 c.c. of the magnesium sulphate solution were used.

A stream of carbon dioxide gas is directed on to the surface of the liquid in the beaker, the solution of cuprous chloride quickly added from the tube, and the titration immediately proceeded with. A white precipitate of cuprous chloride will be formed on pouring the solution into the water in the beaker, unless an undue excess of acid has been employed. The formation of this precipitate is advantageous, since in this case atmospheric oxidation is much less likely than when the cuprous chloride remains in solution.

The solution of potassium permanganate used should contain about 5 grms. of the salt per litre.

If the experiment is carried out under the above conditions, no difficulty is experienced in judging the end of the reaction, since a pure pink colour remains for a long time without fading. This colour is slightly bluer than that due to potassium permanganate alone, owing to the cupric salt present. If the proportion of magnesium sulphate to hydrochloric acid is diminished, the pink tint of the permanganate fades after the oxidation of the cuprous oxide is complete, owing to the action of the oxidising agent upon the hydrochloric acid, and if the proportion of water is diminished a similar effect is observed. We have also found that by using a larger proportion of water than that stated above, irregularities are introduced which interfere with the accuracy of the process.

The following results were obtained in the estimation of the cuprous oxide precipitated from Fehling's solution by a known volume of a solution of invert sugar.

The amount of cuprous oxide which was obtained from an equal volume of the invert sugar solution, and which was weighed as such on a tared filter, is placed side by side with the amounts calculated from the results of the titration:—

—	Cu_2O titrated.		Cu_2O weighed.
	C.c. $KMnO_4$ Solution	Grm.	
I.	17·2	0·1937	0·1970
II.	17·3	0·1948	
III.	17·4	0·1959	

The weight of Cu_2O corresponding to the potassium permanganate solution used is calculated by multiplying the oxygen value of the number of c.c. employed by the factor $8 \cdot 91 \left(= \frac{Cu_2O}{O}\right)$.

The above results are a little low, owing to the fact that, however great the precautions taken, slight oxidation of the cuprous solution does take place previous to its titration. It will appear that much care is necessary for the carrying out of the above process, and that it may easily fail if any of the precautions above mentioned are neglected. With practice, however, fairly accurate results may be obtained, and the process is quickly carried out.

We have now to describe a modification of the above process, in which the use of hydrochloric acid, and also of carbon dioxide gas, is done away with, and which can therefore be performed with greater ease and expediency. Cuprous oxide cannot be dissolved by oxy acids, such as sulphuric and phosphoric acid, because the corresponding cuprous salts do not appear capable of existence under ordinary conditions. In such cases the cuprous oxide is resolved into metallic copper, and the cupric salt of the acid used. Sulphuric acid, therefore, cannot be substituted for hydrochloric acid in the above process. Various other solvents for cuprous oxide were tried by us in order to dispense with the use of hydrochloric acid, and so of magnesium sulphate, but without success. Potassium permanganate solution acts slowly upon the metallic copper, separated from cuprous oxide by means of sulphuric acid, but such oxidation cannot be employed as the basis of a volumetric process.

Dr. Frank Clowes suggested to us that possibly by mixing together sulphuric acid and potassium permanganate solution in suitable proportions, and adding this mixture to the cuprous oxide, the separation of metallic copper might be prevented. This we have found to be the case. Cuprous oxide dissolves completely in a solution of potassium permanganate acidified with sulphuric acid, forming cupric sulphate.

We have therefore employed this reaction as the basis of a modified process, by means of which cuprous oxide can be estimated volumetrically. Excess of the acidified potassium permanganate solution is added to the cuprous oxide, and the resulting solution is titrated, under suitable conditions, with a solution of oxalic acid until the purple colour of the permanganate is discharged. The following are the experimental details of the process:—

Dilute sulphuric acid (1 part of acid to 3 parts of water) is added to the standard potassium permanganate solution in the proportion of four to one. 25 c.c. of the permanganate solution suffice if the quantity of cuprous oxide does not exceed 0·2 grm., and a mixture of this with 100 c.c. of the dilute acid is poured upon the clean cuprous oxide contained in a beaker of suitable size. The liquid is stirred with a glass rod to promote solution, and when the cuprous oxide is dissolved, boiling water is added to the solution in sufficient quantity to raise the whole to a temperature of 45°—50° C., care being taken not to exceed the latter temperature. Standard oxalic acid is then added until the colour of the permanganate solution is discharged. A neutral tint is produced just before the end of the reaction, this being due to the mixture of the pink colour with the bluish tint of the cupric salt. Finally, after becoming nearly colourless, the solution assumes a clear bluish-green colour, and the indication of the completion of the reaction thus observed is very delicate.

It is convenient to employ oxalic acid solution of such a strength that 1 c.c. is equivalent to 1 c.c. of the potassium permanganate solution. The volume of the latter solution

employed for the oxidation of the cuprous oxide is then obtained by subtracting the number of cubic centimetres of oxalic solution used from 25 c.c. The oxalic acid solution should be standardised by the use of the acidified permanganate solution in the same manner as in the actual estimation.

The vessel containing the acidified permanganate solution must not be heated by means of a flame previous to use, as local overheating tends to cause the separation of a brown precipitate of a hydrated oxide of manganese on the subsequent addition of oxalic acid; and in any case the results are variable if the temperature of the solution is too high.

We obtained the two following pairs of results by titration and weighing of the amounts of cuprous oxide, which were precipitated from Fehling's solution in each case, by equal volumes of solutions of invert sugar:—

—	Cu_2O by Titration.	Cu_2O by Weighing.
I.	0·1943	0·1960
II.	0·1953	0·1947

The most important application of this process is its use in the analysis of sugars by the determination of their cupric reducing power. For this purpose the hot solution of sugar is introduced into excess of Fehling's solution contained in a beaker immersed in the water bath, and the reduction allowed to proceed for 14 minutes, according to the method recommended by C. O'Sullivan (Watts' Dict., art. Sugar).

After the removal of the beaker from the water bath, the clear blue supernatant liquid is decanted through a suitable filter, and the precipitate washed several times by decantation until it is free from alkali and organic matter. Care should be taken to transfer as little as possible of the cuprous oxide to the filter during the process of washing.

When the cuprous oxide, both in the beaker and on the filter, is quite clean, a small quantity of the previously prepared acidified permanganate solution is poured upon the filter so as to cover the red stain of cuprous oxide, and it is allowed to remain in contact with the filter for a few seconds only. A pointed glass rod is next pushed through the bottom of the filter paper, and the paper is then thoroughly washed with hot water, the solution and washings being allowed to run into the beaker containing the cuprous oxide to which the remainder of the acidified permanganate solution has been previously added. The action of the permanganate upon the filter paper itself during the time during which the solution is in contact with it is so slight as to be negligible. We could not detect any diminution in the oxidising power of the permanganate owing to such contact. A slight stain of metallic copper is sometimes left upon the filter paper, but this too, when it occurs, is so small as not to affect the accuracy of the titration. Any possible error which might arise from incomplete oxidation of the cuprous oxide on the filter, is best avoided by allowing the precipitate to settle thoroughly after each washing, so as to transfer very little precipitate to the filter. We find that the whole process can generally be completed within about half an hour of the removal of the beaker from the water bath.

The following results, expressed in percentages, have been obtained by us in the analysis of two samples of sugar by this process. The factor 0·5045 has been used for the conversion of weight of cuprous oxide into weight of dextrose, levulose, or invert sugar. Analyses carried out by direct weighing of cuprous oxide are placed side by side with the results obtained by the volumetric method:—

Analysis of "Brown Sugar."

—	Volumetric Method.	Gravimetric Method.
Glucose	11·7	12·1
Sucrose	82·0	82·4
Moisture	4·7	4·7
Ash	0·8	0·8
	99·2	100·0

Analysis of "Preserving Sugar."

—	Volumetric Method.	Gravimetric Method.
Glucose	1·2	1·1
Sucrose	92·6	92·2
Moisture	5·7	5·7
Ash	2·4	2·4
	101·9	101·4

The authors are of opinion that this process will be found distinctly quicker than methods depending on the weighing of cuprous or cupric oxide, even leaving out of consideration the time necessary for the drying of the precipitate when it is weighed in any form. The permanganate solution when once standardised will keep unchanged for a long time, and the solution of oxalic acid will remain at a constant strength for several weeks.

It will be evident that the process can only be applied for the estimation of saccharine substances which will precipitate cuprous oxide in such a state that it can be readily washed free from organic matter. Whenever, in fact, it is permissible to weigh cuprous oxide as such, this method may be substituted for the gravimetric process with considerable saving of time.

Discussion.

Mr. Archbutt said he thought it would be a great advantage to be able to estimate the cuprous oxide by titration rather than to weigh it. In order to do away with any possible action of the filter-paper, why not filter through asbestos? It might also be found advantageous to use hydrogen peroxide instead of oxalic acid to estimate the excess of permanganate. It had the advantage that it could be used in the cold, and he might say could be prepared at any time by dissolving sodium peroxide in dilute sulphuric acid.

Mr. Adams described a method of preparing asbestos filters. Long-fibre asbestos was cut up into bits about an eighth of an inch long, washed with hydrochloric acid, suspended in water, and a small quantity poured into a Gooch crucible held air-tight in a funnel by means of a piece of india-rubber tubing, and connected up to a filter-pump. The thin mat of fibres thus formed made an excellent filter.

Scottish Section.

Chairman: G. T. Beilby.

Vice-Chairman: G. G. Henderson.

Committee:

A. C. J. Charlier.	A. Macdonald.
John Clark.	R. C. Menzies.
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R. A. Inglis.	E. C. C. Stanford.
Robt. Irvine.	R. T. Thomson.

Hon. Secretary and Treasurer:

Thomas Gray, Technical College, 204, George Street, Glasgow.

SESSION 1897–98.

Tuesday, Jan. 25th:—
Mr. L. Pitblado. "Cyanide Practice in the Marikuppam Field."
Mr. Charles J. Ellis. "Testing of Gold Ores, &c. in relation to their Treatment by the Cyanide Process."

Meeting held at Broxburn on Monday, Nov. 29th, 1897.

MR. G. T. BEILBY IN THE CHAIR.

THE AUTOMATIC MECHANICAL DRAWING OF SHALE RETORTS.

The Chairman said that as the first item on the agenda was the reading of certain papers and discussion on the mechanical drawing of shale retorts, he thought it desirable to pass in review on the screen the historical collection of retorts which were shown at the October meeting in Glasgow.

The retorts were then shown on the screen.

THE HISTORY OF SHALE RETORTS AT BROXBURN.

BY NORMAN M. HENDERSON.

It is perhaps quite appropriate that the Scottish Section of the Society of Chemical Industry should meet at Broxburn to discuss the retorting of shale, for Broxburn district has been associated with the manufacture of oil from shale for over 36 years, and it may not be out of place for me to give a summary of the history of the trade.

It was in 1859 that Mr. Robert Bell leased the minerals in this district, for the purpose of working coal and ironstone. He shortly afterwards found large supplies of rich shale on the ground, capable of yielding over 30 gallons of crude oil per ton, and he made arrangements in 1861 with Mr. Faulds, of Glasgow, to supply him with a daily quantity of shale. Mr. Faulds, in company with some others, erected a small work in 1862, just north of the canal, and set up 36 horizontal retorts, but they abandoned the works shortly afterwards, because the price of crude oil fell below 1*s.* per gallon. Mr. Fernie then took them up, in 1864, added 32 vertical retorts, and abandoned them in 1866; but they were again re-started shortly after by a firm known as the "Glasgow Oil Company."

In 1862, Mr. M'Lintock also erected 12 horizontal retorts at Roman camp; these became the property of Mr. Wm. Fraser, of Broxburn, in 1865, who increased the number of retorts to 75, and also erected a small refinery. In 1862 Mr John Poynter and Messrs. Miller and Steele erected small works at Broxburn; while in 1863 Mr. Robert Bell erected 100 horizontal retorts at Stewartfield, and in 1865 100 horizontal retorts at Greendykes. Then, in 1869, Mr. James Liddell erected a crude oil work of 40 horizontal oval retorts, and was the last to stop in 1877.

A small refinery was put up in 1863–64, and worked for several years by Mr. Thomas Hutcheson.

Most of these little works, however, being unable to continue at a profit, had fallen into the hands of Mr. Bell, when, in 1877, the Broxburn Oil Company, Ltd., was formed, took over Mr. Bell's lease, and started operations on a large scale.

I do not think it is too much to say that the advent of the Broxburn Oil Company, Ltd., opened up a new and brighter epoch in the history of the oil industry in this district. Before they commenced operations, the prices obtained for products were such as would now be regarded as enormous, but the cost of manufacture was so great with the old plant that, despite these high prices, the trade was quite unprofitable.

The old retort plant on the ground, which the Broxburn Oil Company took over, was of various kinds, but mostly horizontals, with a few old verteals. The "horizontals" at Greendykes were mostly cylindrical in shape, and about 9 ft. long; the "horizontals" at the Albyn Works were of various forms—one kind was rectangular in shape, about 10 ft. long, with charging hoppers on the top, and the other kinds were of the oval description, from 2 ft. 3 in. to 2 ft. 6 in. wide by about 1 ft. 3 in. high, and varied from 9 ft. to 10 ft. long. Some of these had charging hoppers on the top, and others were charged from the end, like a gas retort. The buildings and general construction of these retorts were of the most fantastic description, and, if they could be seen now as I saw them, would form an interesting study to the modern oilworks manager.

After four months' trial of these old retorts, while the erection of their refinery was proceeding at Broxburn, the Broxburn Oil Company found that it would never pay to continue using them, and the question of retorts, therefore, became one of paramount importance to the company.

At that time there were several retorts, by various inventors, in the market, the more prominent two competing retorts being Messrs. Young, Scott, and Stephen's, 1872, known as the "Gas Lute" retort, and my own 1873 retort, Patent No. 1327, both designed for the utilisation of the spent shale as fuel.

The merits of all the various retorts were taken into careful consideration by the Broxburn Oil Company, and they finally adopted the "Henderson" retort, 1873 patent.

The erection of these "Henderson" retorts at Broxburn in 1878 proved a great and immediate success. The cost of the manufacture of the crude oil was reduced by 50 per cent., as compared with the cost from the old retorts, and it also effected a very great saving in the costs as compared with the retorts generally in use elsewhere at that time. For example, the cost of the crude oil from the old retorts at Broburn was 4·28*d.*, while the cost of the crude oil from the "Henderson" was only 2·31*d.* per gallon. These prices do not take credit for the sulphate of ammonia.

It is also to be noted that the "Henderson" retort gave a yield of 16 lb. to 17 lb. of sulphate of ammonia per ton from Broxburn shale, as compared with 5 lb. to 7 lb. from the old retorts. This larger yield of sulphate of ammonia would further considerably lessen the nett cost of the crude oil, if credited, the nett valve of the sulphate of ammonia, after deducting the cost of production, &c., being 14*l.*

The best retort generally in use elsewhere at this time was what was known as the oval "vertical." The cost of the crude oil from the best constructed of this retort averaged 3·27*d.*, or practically one penny more that the cost per gallon from the "Henderson" retort.

Besides this, the "Henderson" retort with the Broxburn shale gave a yield of 33 galls. of crude oil per ton, and as the retort was worked at a low temperature, the crude oil was of excellent quality, easily refined, and contained a high yield of paraffin scale.

The retort was an intermittent one, and utilised the spent shale as fuel for the next charge by dropping it hot into the furnace below. It was heated from without by the combustion of its own permanent gas and that of the spent shale, supplemented as required with about one quarter cwt. of coal to the ton of shale.

It was heated within by the introduction of superheated steam. The charge was left in the retort for 16 hours at least. The longer the shale was left in the retort, acted on by the superheated steam, the greater was the yield of ammonia; but at this time it was not considered profitable enough to leave it in beyond a certain time, and the time that paid best had to be determined by experience. This suggested that a longer distillation under proper conditions would give increased ammonia.

It is not necessary for me to give any further and more minute description of this retort, because that is already given in a paper by Mr. D. R. Steuart, which appeared in this Journal, 1889, 100; and in the Gas. Inst. Trans. 1887, 21.

Meanwhile the Young and Beilby retort came out, giving its large increase of ammonia, associated with a great increase of temperature inside the retort during the second or ammonia part of the distillation.

This retort has also met with much favour in the trade, and was largely adopted by the oil companies. It seems to be well adapted for the poorer shales containing small yields of crude oil and large yields of sulphate of ammonia; but I have always held that this retort required improvement, and it would be interesting to have the opinion of those who have had experience in its practical working.

In 1889, I took out a patent for "Improvements in the Distillation of Shale," Patent No. 6726, as an improvement on the Young and Beilby method described in this Journal, 1894, 1038.

In this new retort I aimed at getting an increased yield of ammonia by adopting a retort of greatly increased length as the cheapest method of getting the shale a longer time in the retort, and exposed to a larger amount of heating surface, enabling the retort to be worked at a lower temperature, and thereby insuring decrease in the wear and tear and leakage, and a longer life to the retort.

At this stage I would call your attention to the drawings, with description of working.

Fig. 1 shows front elevation and part section.

Fig. 2 shows cross section.

The retorts are vertical, 28 ft. long.

In the upper cast-iron portion of the retort A, the oil is first distilled off the shale at a temperature of about 900° F. The spent shale is then passed downwards into the brick portion of the retort B, which is kept at a constant temperature of about 1,300° F.

The shale in passing down through the retort is kept in constant motion by a toothed roller C at the bottom, which delivers it into a hopper D below at the required rate.

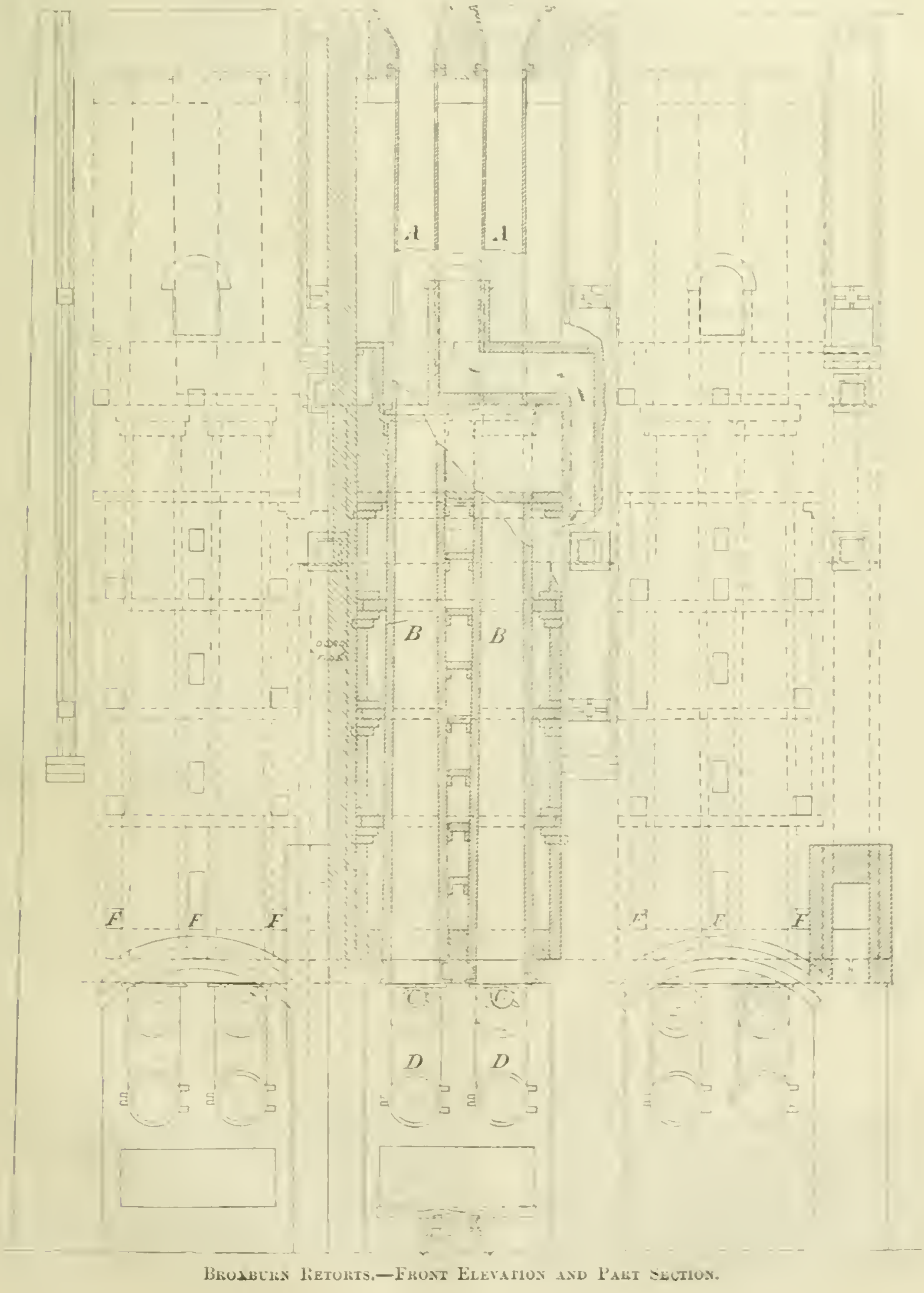
All the products of distillation are drawn off at the top of the retorts through the branch pipe E.

The gas from the producers for heating is introduced at the ports F.

Levers rotate the roller C with ratchet wheels.

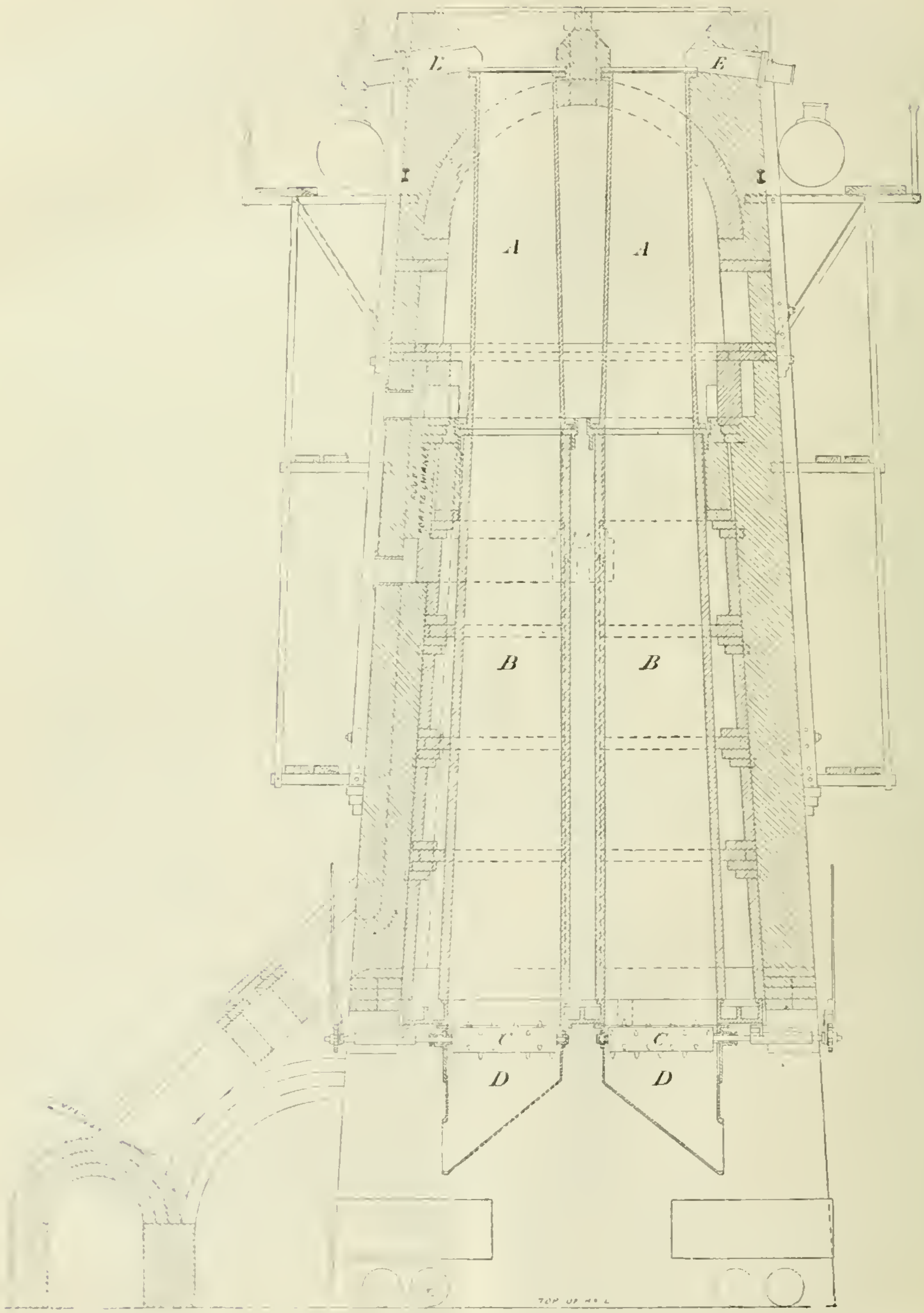
The greatly increased length of the highly heated brick chamber below, allowed for the better graduation of the heat from the bottom towards the top, where it joins the iron retort, and it also secured a better jointing of the iron with the brick retort. The retorts were carefully built with "V" jointed bricks, so as to give them greater strength and

Fig. 1.



Broxburn Retorts.—Front Elevation and Part Section.

Fig. 2.



Broxburn Retorts.—Cross Section.

secure absolute tightness; and care was taken at the same time in the construction that free expansion was obtained in the heating chambers.

This lengthening of the retort might have introduced new difficulties, from the danger of local overheating causing fusing and clinkering of the spent shale. To overcome this, I put a toothed roller into the bottom of the retort to cause the whole column of shale to keep constantly moving downwards, and thus prevent any fusion or clinkering. This continuous downward motion of the shale, by constantly exposing new surfaces of the material to the heat and to the action of the steam, largely increased the yields of oil and ammonia.

In the experimental sets of retorts, I introduced a toothed roller at the bottom of the iron retort to break up the spent shale coming from the oil retort, the shale at this stage being friable and easily broken. I did this in the expectation that the shale, being broken up into smaller fragments,

would be more easily acted on by the steam, and give more readily an increase of ammonia; but it did not have all the effect expected, and caused extra trouble, and the retort was simplified by using only the one roller at the bottom.

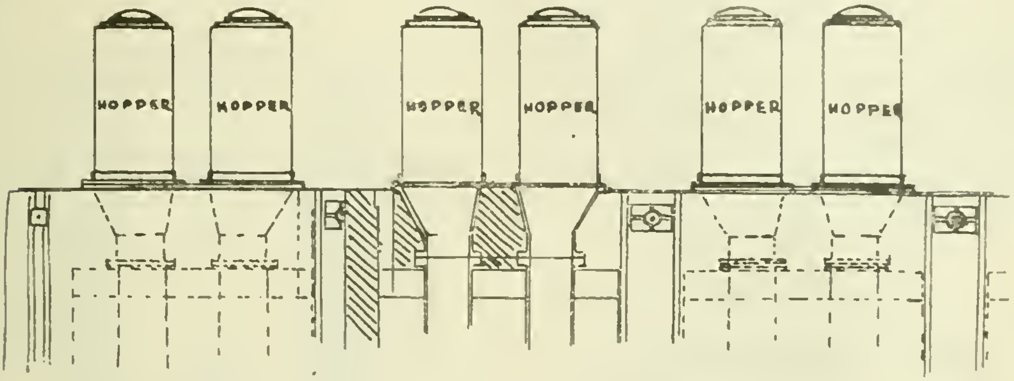
Another feature in this retort is that it is heated by gas made from dross coal in ordinary producers apart from the retorts. This was found to be very advantageous in manipulating and regulating the heats, while the flues were kept clean and free from dust, and a saving of fuel effected.

A further feature is the large hopper on the top, which gives a continuous supply of shale and holds sufficient for 18 hours; the advantage of this hopper being that it allows of all the charging to be done during the daytime, and the drawing and tipping to be stopped during the day on Sunday, which is better for both workmen and employers.

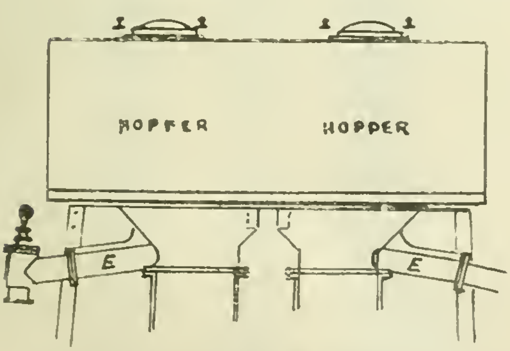
Labour with this retort has been very much reduced, and is of a more pleasant and easy nature for the men employed. A bench of 88 retorts, putting through about 160 tons of shale every 24 hours, is charged by two men during the day, and the spent shale is drawn from the hoppers below into hutches by one man each shift; the hutches are then taken away by the other men.

My aim, then, was to obtain an increase of ammonia without the violent heats of the old "vertical," or of the Young and Beilby retorts. The heightening of the retort, with its moderate heat, gave a greater yield of crude oil by 3 galls. per ton of shale, and an increased yield of sulphate of ammonia from 16 or 17 lb. to 13 lb. per ton from the Broxburn shale, over the old "Henderson" patent retort of 1873.

I may here remark that the first bench of these new retorts was erected at our Broxburn works six and a half



BROXBURN HOPPERS.—FRONT ELEVATION.



BROXBURN HOPPERS.—CROSS SECTION.

years ago, and that it has been constantly working ever since without the slightest repair. One set of these was examined only three weeks ago to ascertain its condition, and it was found that both the brick and the iron work were as good and tight, to all appearance, as when they were first started.

These retorts have been exclusively adopted by the Broxburn Oil Company, and I may say that, on the whole, the practical results of these retorts have exceeded my expectations.

The distinguishing features of the retort may be summarised as follows:—

1. The increased surface and capacity of the retort obtained from its increased length, and which gives the shale a longer exposure to the distillation temperature.

2. The moderate heat required in working the retort.

3. The lesser heat at the junction of the brick and iron retorts.

4. The tightness and durability of the retort, secured by the use of the specially jointed bricks.

5. The large hopper on the top, doing away with night charging.

6. The outlet branches being placed on the bottom of the hopper.

7. The constant downward movement of the shale in the retort, caused by the toothed roller in the bottom, and the regulation of the speed of the discharge into the hopper below.

8. The heating of the retort by gas made from dross coal in ordinary producers.

Appended to this paper, I have given a statement showing the comparative costs of the different retorts worked at Broxburn.

Comparative Results from the Actual Working of the Various Retorts worked on the Broxburn Shale at Broxburn, from 1877 till 1897, as described in this paper; the Shale and Sulphate of Ammonia being charged at the same Values in each instance, that is, the values ruling in 1877–78.

Description of Retorts.	Total Cost of Retorting, per Gallon, including Shale, Labour, Stores, Maintenance, &c.	Nett Cost of Retorting, per Gallon, after crediting the Sulphate of Ammonia at 14*l*. per Ton, the Nett Value in 1877–78.
	d.	*d.*
I. The old horizontal retorts in use at Broxburn in 1877.	4·28	3·91
II. The old vertical retort, the best constructed type of vertical retort in use in 1877.	3·27	2·89
III. The "Henderson" retort (Patent No. 1327 of 1873): first year's working at Broxburn, 1879.	2·31	1·62
IV. The "Henderson" new improved retort (Patent No. 6726 of 1889); present year's working at Broxburn, 1897.	1·89	0·16

It may be interesting to show here the increase of the population of this parish coincident with the progress of the

oil industry, which is the staple industry of the district :—In 1861 the population was 1,500; in 1881, 4,812; in 1897, 10,188.

Discussion.

The Chairman put the following questions to Mr. Henderson. First, "How long is the shale exposed to heat in the Henderson retort as compared with other retorts?" Second, "How much permanent gas does this retort give off per ton of shale, as compared with other retorts?"

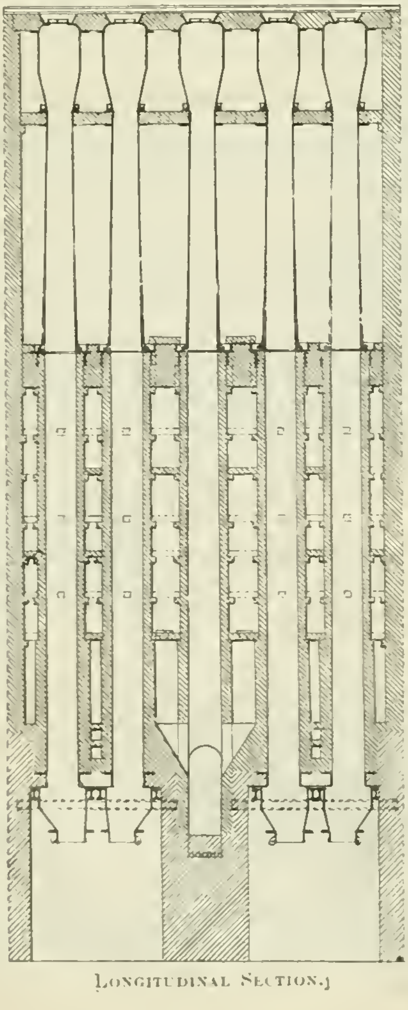
Mr. N. M. Henderson, in reply, said that the cubic content of the retort was 106 cb. ft., and as 33 cb. ft. of the broken Broxburn shale was generally taken to weigh 1 ton, the shale was $42\frac{1}{2}$ hours in the retort, drawing taking place at the rate of $1\frac{1}{2}$ cwt. per hour. In the old vertical retort, the cubic content of which was 26 or 27 cb. ft., and on the same calculation of the weight of shale, and drawing at the rate of $1\frac{1}{4}$ cwt. per hour, which was equal to $1\frac{1}{2}$ tons per 24 hours, the shale was in the retort $21\frac{1}{2}$ hours. In reply to the next question about the gas, he said it was very difficult to determine the exact quantity of gas coming off from these retorts. The only way they had of measuring it was by the exhauster. The measurements taken from one bench, with a new 30 cb. ft. exhauster going at 42 revolutions per minute with a $\frac{1}{4}$-inch water vacuum on the one side and from 4 to 5 ins. of water pressure on the other, gave practically 10,000 cb. ft. of gas per ton of shale, and this gas yielded about 41 per cent. of combustible matter.

THE PHILIPSTOUN RETORT.

BY A. H. CRICHTON.

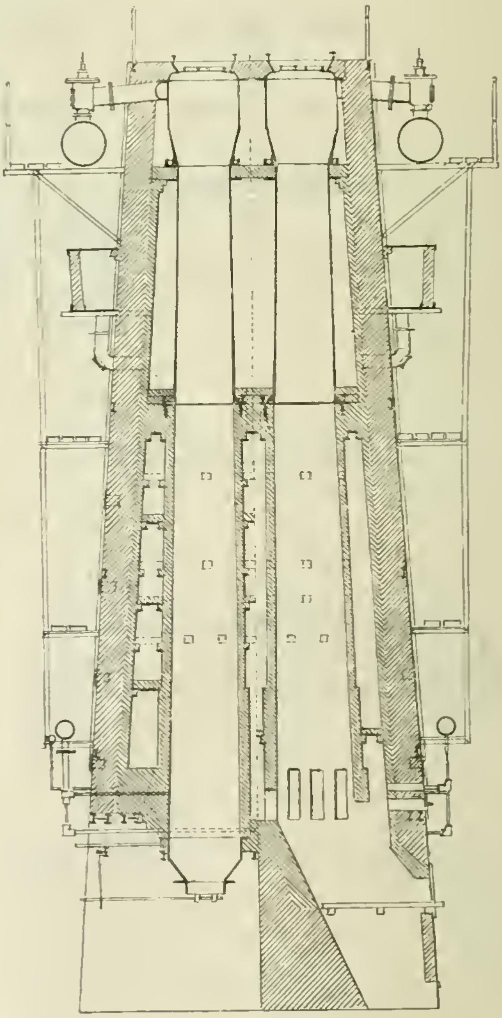
(Fig. 1.) This is a longitudinal section of the Philipstoun retort. It is built on the principle of the Young and Beilby retort, but is a combination of the best of all we knew at the time of erection. There is a bit of Mr. Henderson in it, there is the principle of the Young and Beilby retort, and a good deal of our own—let it be right or wrong. The retort bench is 40 ft. high from the ground to the top rail level. The retort is 28 ft. 9 ins. long including top hopper, $18\frac{1}{4}$ ft. of brickwork, 10 ft. of iron retort, and hopper $4\frac{1}{2}$ ft. The section shows an ordinary Young and Beilby gas producer or coal retort in the centre, with two shale retorts on each side, there being two coal and eight shale retorts in the setting. The iron portions of the 10 retorts are all within one large chamber; underneath, the brick portion is divided into four chambers. The gas from the coal retort distils

Fig. 1.

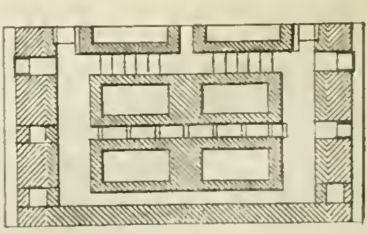


Longitudinal Section.]

Fig. 2.



— Cross Section —



— Plan —

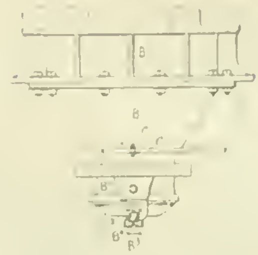
downwards and passes out of the retort to right and left into the bottom chamber. At the centre of the bottom chamber on each side the coal gas is joined by the permanent shale gas, whence the whole heating gases ascend by a zig-zag course to the large upper chamber. From this chamber the waste gases are drawn off by two external side flues to the chimney. The connection between the upper chamber and the side flues is by means of a 7-inch iron pipe or flue—each flue being provided with a damper—at each corner of the chamber a little above the floor. We thought the external flues, with downcoming shaft at the end of the bench, preferable to forming descending flues in the walls of the building of the retorts themselves for returning the gases from the top oven down to the ground and thence to the chimney. At the bottom of the retort the shale rests upon and is drawn by means of a mechanical appliance which we have designed and patented for the purpose. The bottom hopper is empty when the shale is at rest in the retort, and, the hopper being placed vertically below the retort, we can look right up to the grippers and see the shale resting upon them by simply opening the bottom door and shutting off the steam for the moment. If anything should go wrong in the inside of any retort, such as "dandering" of the shale, we can empty the retort, and, looking up or down the inside, observe the obstruction and have it removed, either from top or bottom as may be convenient. This can be done with any single retort in a set without interfering in any way with the working of the other retorts. We do not know any other shale retort which can be examined and treated in this way.

(Fig. 2.) This is a cross section of the retort taken on two different lines—one through the shale retort and another through the coal retort, the coal retort being purely and simply the form of coal retort applied to the Young and Beilby. The plan is also shown. We are taking steps, however, to do away with these coal retorts, and have in some benches done away with them, within the last few weeks, not because we find them to be bad gas producers, but because of their structural defects. There is a good deal of tear and wear on the coal retorts, and repair is necessitated on them when none is required on the shale retorts. We have had shale retorts at work now for more than four years which have never been entered, while the coal retorts on the same benches have had to be repeatedly repaired, especially at the furnaces, thereby interfering very much with the working of the shale retorts. We have erected some gas-producing plant alongside the benches, introducing the coal gas from the outside.

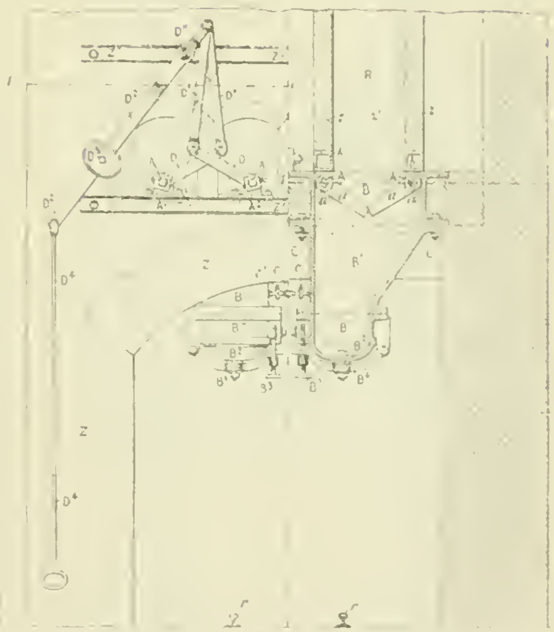
Fig. 3 is a drawing of the mechanical appliance, showing horizontal section through two retorts—one at bottom of brick part of retort, and the other immediately above the apparatus: also elevation showing external parts and vertical section. The apparatus is exceedingly simple. Through the bottom iron hopper we have two transverse shafts, passing through bottom hopper from the back to the outside of outer wall of bench. These shafts work into a plain boss at the back of the hopper and pass through an easy-working stuffing-box at the front to the outside of the bench. The outer ends of the shafts are carried on an iron bearer with cap and cover; on the end of each shaft there is an arm connected by links to a hand-lever, the two arms crossing each other as shown. Each shaft is fitted with a set of cast-iron arms or grippers. When closed and supporting the shale at rest in the retort, these arms are at an angle of 45°, and when full open are vertical. The grippers are opened and the shale relieved by an upward movement of the hand-lever, and closed by pulling the lever downwards. When drawing the retorts, a hutch is placed under two retorts, back or front, and half a hutchful taken from each retort. The system followed at present is to draw the retorts once in six hours, the throughput of shale being about the same as with the Young and Beilby and the present Henderson retorts. There is perfect control over the movement of the shale within a retort. You can take out a bonnetful or a hutchful, just as you please. There is no difficulty in that respect. The bottom door-piece and door have turned edges: the door is hinged, and kept tight when closed by means of a concentric link, which is hammered on to the bow of the bottom door of the retort.

Fig. 3.

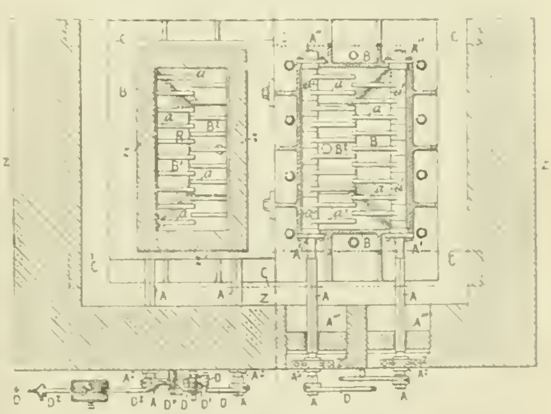
Mechanical Appliance for Removing Spent Shale.



Elevation.



Vertical Section.



Horizontal Section.

There is no screw there, although a screw is shown on the plan. We found, like many others, that the screws got very soon unworkable in the moist atmosphere which pervades the bottom of the retorts, and we have now simply the concentric link, which was designed by our late foreman, Mr. Muir, and forms a neat useful contrivance.

PUMPHERSTON PATENT RETORT.

BY JAMES BRYSON.

The description of this retort naturally follows that of the Broxburn and Philipstoun designs, being of a later date than either; and, as will be seen, it differs essentially from any of the forms which have been referred to in the previous papers.

Prior to the introduction of the new retort, the Pumpherston Company had distilled about 3,000,000 tons of shale by the Young and Beilby retort.

While always sensible of the advantages gained by the use of the Young and Beilby retort over the older forms of retorts, our experience convinced us that the subject had not been exhausted, and that finality had not yet been reached. Like many others in the Scottish oil trade, many modifications on the Young and Beilby retorts had been tried by the Pumpherston Company, with no practical advantages in any case. At the Seafield works of the Company two benches of 80 retorts each were built with an increased height of five feet, part of the increase being on the brick and part on the iron portion of the retort. This modification proved to us that any step in the direction we had taken only led to an aggravation of the difficulties connected with the working of the original Young and Beilby retort, *viz.*, sticking and dandering, with all the consequent evils connected therewith, and that such modifications did not lend themselves to greater economy in working.

It was after the foregoing experience that we determined to make some efforts towards the solving of the problem of retorting shale. In facing this question we had before us the fallacy in principle which, as it appeared to us, existed in all previous forms of retorts, *viz.*, that it was essential to the proper heating of the shale in the centre of the retort that the retort itself should be of very limited dimensions in its breadth, so that the external heat on the retort should be brought as near the centre of the shale within the retort as possible.

We were of opinion that such limitation of the size of the retort was fallacious, and on certain lines of construction might be ignored.

In view of the foregoing we were of opinion that a retort might with great advantages be constructed on the following lines:—

1st. Of large dimensions and capable of a greatly increased throughput compared to anything hitherto known to the trade.

2nd. That the whole structure must be designed and constructed upon lines of a much more substantial nature, and freed from all the structural weaknesses which had belonged to retorts hitherto in use at high heats.

3rd. That to effect economy in working, which was a leading condition aimed at, a passage should be constructed underneath the retorts, running parallel therewith from end to end of each bench.

4th. That the discharge from the retort should be continuous, or nearly so, and free from danger of intermittent movement.

The first point practically dealt with was the discharge or bottom part, this having an important bearing on the foundation building, and in the working out of this the shape of the retort itself was developed. As will be seen from the illustration, there is, at a distance from the lower end of the retort, a cast iron table or disc fixed to the inside of a box casting in such a manner that a space is left all round between the edge of the table and the sides of the box. This box is carried to a sufficient depth so as to form a hopper for the discharged shale. When shale is filled into the retort it rests on the table, while the space leaves room for some passing over the edge. Through the centre of the table a steel spindle or shaft projects, on the upper end of which is a curved arm, and this when rotated wipes the shale off, causing it to fall over the edge of the table into the hopper below.

The cast-iron hopper is formed so as to enclose the mechanism mentioned, and is large enough to take in two retorts, so that one outlet serves for the two. This outlet is closed by a conical lid, which is worked by steam pressure acting on a small piston and cylinder, and proves a quick and reliable means of emptying the hopper of its contents. The shaft carrying the curved arm passes through the bottom of the hopper and has a ratchet and lever fitted to the lower end. This is actuated by a bar or rod of tee iron, which is made to travel horizontally, being driven by gearing from a small steam engine. The bar in travelling moves the ratchet lever, which in turn moves the vertical shaft carrying the curved arm. The motion is comparatively slow, as the arm has only to make one revolution in 90 minutes or thereby. The action is most satisfactory, and the throughput of shale can be regulated at will.

Having such a bottom part, it is apparent that to get the hutch immediately under the opening was most desirable, and this decided the form of the foundation piers, which, as will be seen, are built so as to allow for a passage right under the middle of the bench, and running from end to end thereof. It will be readily understood that when the table and curved arm were found to work so satisfactorily, the desire to have the retort naturally arose, and the advantages and disadvantages of such a form had to be considered. Hitherto the idea had prevailed that it was essential to have the retort made rectangular or narrow in some direction, so that the distance from the centre of the shale to the heating surface might be kept as little as possible. This idea was deliberately set aside, we being convinced that, given a retort large enough to allow a slow and regular passage of the shale, there will be a fairly equal heating throughout, and if that was so, then the circular retort had advantages over the rectangular or square section, more especially in that when filled with shale there was less likelihood of free spaces being formed, as might be the case in the corners of rectangular retorts, and also in the fact of there being no such corners, shale would not have the same chance of hanging up, but would pass more evenly down through the retort. Another very important advantage we considered the circular form had over the rectangular was that a more efficient heating flue surface could be made by having no right-angle turns presenting surface for the impinging of the flame in its passage, but, being circular or nearly so, it had the effect of making the gas cling around the walls of the retort, thereby adding to the efficiency of the heating power.

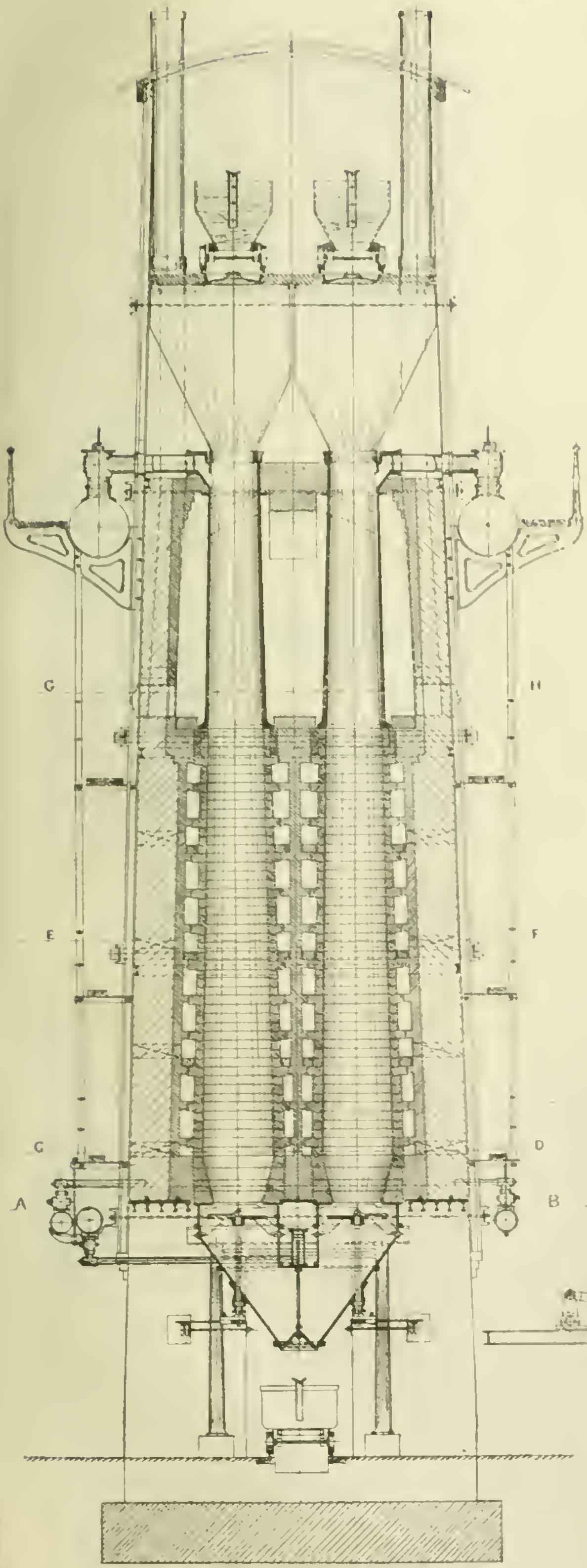
In deciding the dimensions of the retort there were various points which had to be considered, the chief of which of course was how far it would be safe to go in the direction of increased capacity, and, second to that, the difficulties that would accompany such increase in the matter of strength and stability, and also how far the increased capacity would tend to economy in working. After considering all these points we finally decided that a retort 3 ft. at largest diameter and 30 ft. long would probably best fulfil all the conditions.

With these dimensions we got a retort about 150 cb. ft. capacity, excluding top and bottom hoppers. The top hopper, made of mild steel plate, was of sufficient size to contain such shale as would serve for 24 hours without refilling or leaving the gas outlet uncovered with shale within that time. With such a retort we find that a throughput of 5 tons of shale per day can be efficiently worked, with almost a minimum cost.

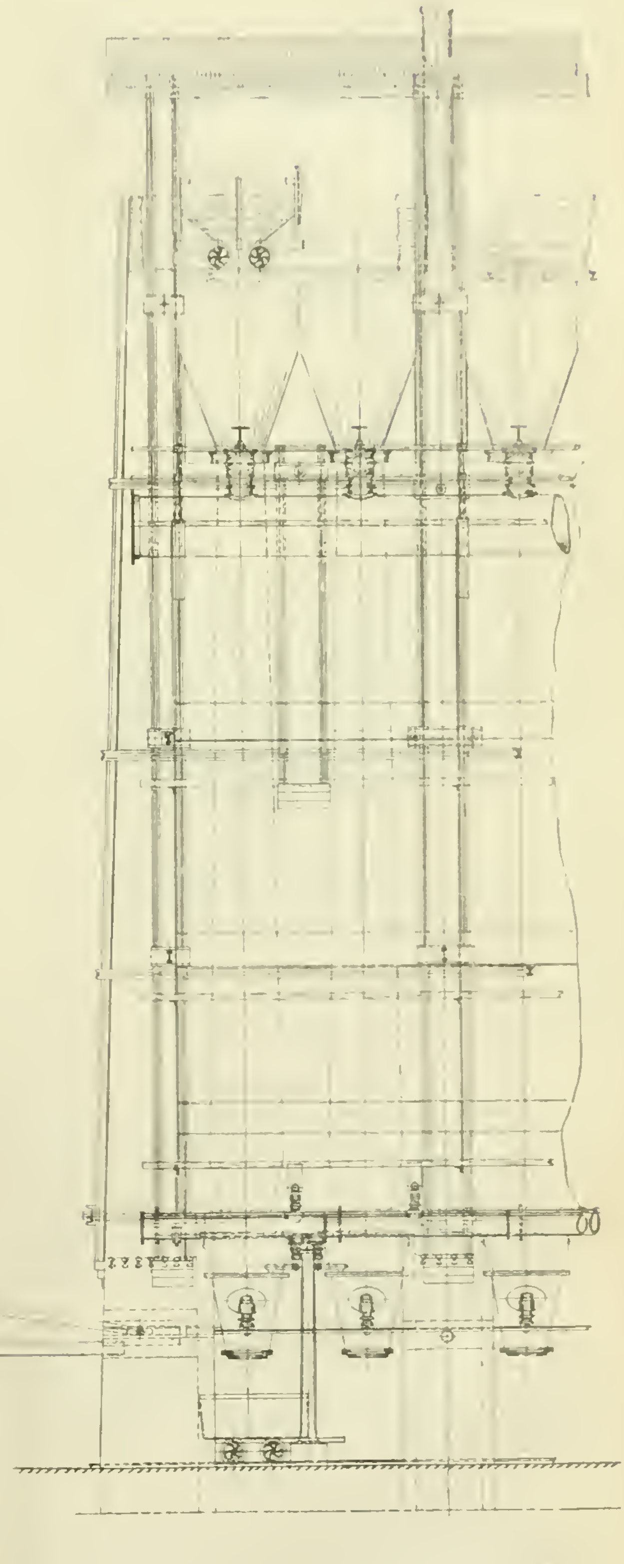
The next point is the strength or stability of the structure, and reference to the illustration will show how that has been carried out, the triangular pillars projecting between the retorts forming heavy and strong abutments to the walls, while the whole structure is firmly bound by horizontal and vertical binders.

Another point, which is by no means unimportant, which we secured by the construction as described and illustrated by the drawings, is that every individual retort is supplied with its own fuel gas and is isolated from its neighbours up to the bottom of the iron part. By this means the heats can be more regularly kept and adjusted, according to the condition under which each retort may be working. The erection of the first bench of these retorts clearly demonstrated the possibility of retorting ordinary shales without more fuel than that derived from the permanent gases returned from the condensers, and at the present moment the Pumpherston Oil Company have 208 of these retorts working without any other fuel than those permanent gases.

Pumpherston Retorts.



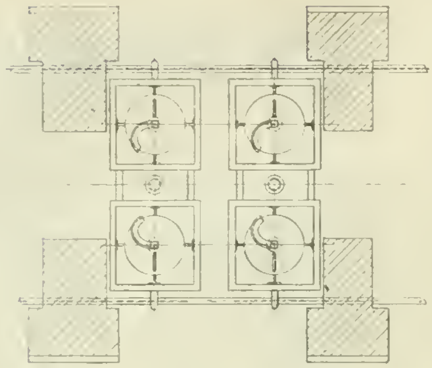
END SECTION



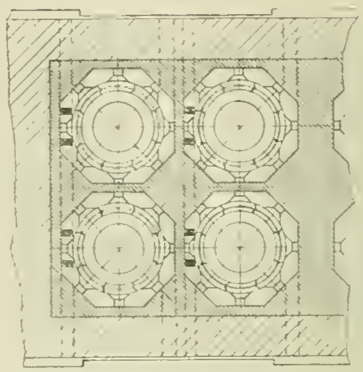
PART ELEVATION

D

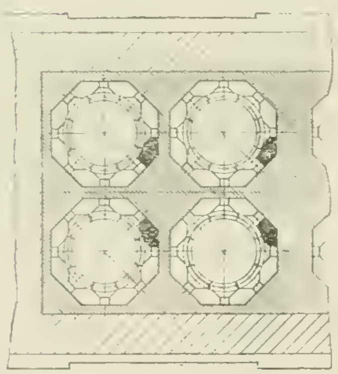
Pumpherston Retorts.



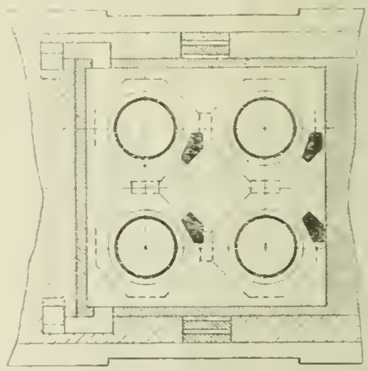
Section on A.B



Section on C D



Section on E.F



Section on G H

The economy which has been effected may be readily understood by comparing the results which have been realised in actual practice extending over a considerable time.

The average cost of retorting with Young and Beilby retorts was 22*d.* per ton, and with the new retort 12*d.* On the other hand, on comparing the value of the products from the shale, we get a difference of about 1*s.* per ton. These two added together give a difference of 22*d.* per ton in favour of the new retort, which in these hard times would be considered a very handsome profit indeed.

The experimental retort which was erected in 1894 has worked 1,230 days, including various stoppages for examination, and during that time has distilled 1,312 tons of shale of various qualities, without requiring repair in any way whatever—an experience, so far as we know, unique in the history of the Scotch oil trade, and one which goes far to give us confidence to go forward with further erection.

Discussion.

The Chairman said that it would have been very interesting if Mr. Henderson had, for purposes of comparison, expressed the actual working costs of his retort in tons, because, as Mr. Bryson had pointed out, the ton was, after all, the unit which had to be dealt with and handled by the workmen. He said that what struck him most in Mr. Bryson's retort was its great diameter and capacity. Mr. Bryson was right in saying that they all would have been startled 15 years ago to have been told that anyone could work a retort 3 ft. in diameter by external heating alone, and he hoped that some of those present who were actively engaged in working automatically drawn retorts would have something to say on this point, as to how it is that the shale in a retort of such large diameter could be satisfactorily heated through and distilled. The old fear was that as the diameter was increased the external temperature would have to be raised, so that the shale next the skin of the retort would be in a fusing condition before the shale inside was cooked. That was the fear long ago, and it was certainly not groundless under the old conditions. When he had the pleasure of seeing Mr. Bryson's retorts at work a few weeks ago, he had been struck with the uniform character of the spent shale which came from the retort. The series of drawings of the retorts of the past 30 years, which was prepared for the last meeting of the Section, showed how the height of the working part of the retort had been steadily increased, but it was only when Mr. Bryson's retort was reached that the diameter and cross section were seriously increased. Of course, the continuous movement of the shale must be credited with the better and quicker distribution of heat, but within this general statement there was room for

speculation as well as experiment on the possibilities and limits of the method.

Mr. A. H. Crichton said that he had been very much pleased with what he had seen of the working of Mr. Bryson's retort, and highly appreciated the frank and spontaneous way in which information had been afforded those visiting the works. He was pleased that Mr. Bryson had been willing to come forward and publicly read a paper on this subject, and asked Mr. Bryson to supplement his description by giving the value of the heating agent he employed, as compared with that used in the Young and Beilby retort. He considered the question of labour a very important one, but thought that, from what he had been able to see at the works, there was no difficulty whatever in arriving at Mr. Bryson's method of economising labour; but that it had always seemed to him that, while the cost of labour for retorting shale per ton was an exceedingly heavy one, in the distilling of shales the great incubus was the cost of fuel. The elimination of fuel from the cost sheets would mean increased prosperity to the Scottish oil trade. Mr. Bryson had gone a considerable way in that direction, in obviating the necessity of providing fuel for the distilling part of the process; and this, he presumed, meant a distinct saving of between 5*d.* and 6*d.* per ton, taking the average of the last few years. He therefore thought it would be very interesting and instructive to have a record of the value as a heating agent of the permanent gases returned from the condensers of the new retorts as compared with the old form of retorts.

Mr. J. Bryson, in reply, said he had some difficulty in answering the question, because of the difficulty which Mr. Henderson had mentioned. He had tried very often to measure the quantity of gas, but had never been able to arrive at anything definite. He usually got more than Mr. Henderson gave, and estimated it at something like 13,000 cb. ft. to the ton. He quoted the results of an analysis given by Mr. Rowan (this Journal, 1891, 443) of the gases from the Young and Beilby retorts, then taking that as a comparison and allowing the quantity to be something like 13,000 cb. ft., which he thought most of the users of the Young and Beilby retort would admit to be a fair estimate. A considerable difference is shown in the heating value of the return gas.

Young and Beilby.	Per Cent.	Bryson. (Sample taken Feb. 27, 1895.)	Per Cent.
Carbonic acid	23·0	Hydrogen	55·56
Carbonic oxide	4·0	Olefines	1·58
Hydrogen	13·4	Marsh gas	3·70
Olefines	1·6	Carbonic oxide	9·77
Marsh gas	19·7	Carbonic acid	22·08
Oxygen	1·3	Oxygen	1·18
Nitrogen	37·0	Nitrogen	6·33
	100·0		100·00

The greater quantity of gas from the Young and Beilby, compared with that obtained from the Henderson retort, he (Mr. Rowan) attributed to leakage—a view which was fully confirmed by the high percentage of nitrogen. He stated that a number of samples from his retort had been taken and tested by their own chemists, and the results had been confirmed by other chemists to whom samples had been sent. The large percentage of combustible matter, 70·46, he thought, explained to a great extent the difference in consumption of fuel between other forms of retort and this larger retort. He had no doubt that a great deal was due to the larger capacity with the less surface for radiation, as with a larger quantity of shale in the retort there was a proportionately very much less surface exposed to radiation and loss. These were the only reasons he could give for being able to eliminate the fuel from the heating of the retorts.

Mr. Henderson said that the Chairman, in expressing his views on the capacity of these retorts, had talked with surprise of the increase in dimensions. He thought there was much to be learned in that way yet, but he had no doubt that the introduction of mechanical movement in the retort enabled them to increase the dimensions of the retort and so to do what had formerly not been possible. He referred specially to the higher percentage of hydrogen in the gases from the Bryson retort, which, as far as he remembered, was about three times as great as in the gases from his own, and thought that the various oilworks managers should have samples of gas taken regularly and analysed, as the results would show whether the retorts were working properly, and would enable them to see that they were not producing gas at the expense of oil.

The Chairman remarked that the results quoted by Mr. Bryson could not be taken as representing the average composition of the Young and Beilby gas. In his experience the nitrogen content given was abnormally high.

Mr. Bryson, in reply, stated that he had in some cases got even a higher percentage of nitrogen.

Mr. Crichton suggested that some of the members having a knowledge of the chemistry of the gases produced in the distillation of shale should supplement what had been said on retorting by giving a paper on the heating of retorts. He did not consider the subject a suitable one for a conference of managers, but thought that it should rather be taken up by one man who was specially fitted for the investigation, and who would subsequently give a good practical paper upon which further discussion and inquiry might be founded.

Mr. John D. Somerville considered that the uniform heating of the shale in Mr. Bryson's retort might be explained on the assumption that the shale at the centre of the retort moved more quickly than that at the sides, which had the resistance of the walls of the retort to overcome, and that as the result of this the shale next the walls of the retort would gradually be moved in towards the centre. He was of the opinion that the shape of the retort, which tapers from above downwards, would favour this turning in of the shale. He further stated that, as this mixing process would account for the uniform nature of the spent shale, it would be interesting to construct a model of the retort, and to study the passage of coloured pieces of shale through it, with a view to testing the correctness of his theory.

DISCUSSION ON THE LABORATORY METHODS OF DETERMINING THE YIELD OF SULPHATE OF AMMONIA OBTAINABLE IN THE WORKS FROM A GIVEN SAMPLE OF SHALE.

Mr. J. Moffat Johnston said that, as he understood the meeting of the Society had been primarily arranged for the benefit of the members of the oil trade, he was of opinion, on account of the probable representative attendance of the chemists connected therewith, that some benefit might be derived from the free interchange of opinion upon the above subject, and he hoped that ultimately a standard method might be agreed upon for general adoption by the trade. He first discussed the method of sampling, referring to the great difficulty of obtaining a fair average sample of shale, and gave his own experience, which had been chiefly derived from the shales of the Oakbank Company. The method which he adopted there, the results of which had been fairly satisfactory on the whole, was as follows. Circular cuttings of all the seams worked by that company were procured, *i.e.*, the actual height of each seam worked, and in these cuttings the total nitrogen was determined by Varrentrap and Will's method of combustion with soda-lime, with the result that he found that from 50 to 55 per cent. of the actual nitrogen present in the shale was obtained in the form of sulphate of ammonia in the Young and Beilby retorts when in fairly efficient condition. In connection with these figures, he remarked that he had additional proof of their general accuracy from a test, which he had the pleasure of superintending, of two separate 50-ton representative lots of Oakbank shale carried out in Mr. Bryson's experimental retort at Pumpherston; the results of these tests gave somewhere about 58 per cent. of the total nitrogen in the form of sulphate of ammonia, and corroborated the accuracy of the claim made by Mr. Bryson for his new retort, that the yield of ammonia is increased by from 5 to 10 lb. per ton of shale.

Before he gave notice of his intention to bring up this subject at the meeting, he had put himself in communication with Messrs. Steuart and Bailey, the respective chief chemists of the Broxburn and the Pumpherston Oil Companies. As a result, he found that the method used in Broxburn was the old-fashioned tube test, which, although old-fashioned, is an excellent mode of arriving at the yield of crude oil in shale, but which can scarcely be regarded as being very satisfactory for ammonia results. This tube or miniature retort was sealed by means of water, the ammonia water titrated, and the result thus obtained multiplied by two, as being approximately what may be expected in the works from the new Henderson retorts. Mr. Steuart informed him that these retorts give about 70 per cent. of the total nitrogen in the form of sulphate of ammonia, which was certainly very satisfactory; but, in view of the results obtained in Mr. Bryson's retorts, he was inclined to think this figure rather exaggerated. Mr. Bailey, of the Pumpherston Company, informed him that he adopted a method which was supposed to be an imitation of a retort. He heated about 30 grms. of the shale in an iron tube by means of an ordinary combustion furnace, passed steam over the red hot shale for about one hour and a half, absorbed the ammonia gas produced in standard acid, and titrated back the excess of acid. Mr. Bailey maintained that his results agreed precisely with those obtained in the works. He (Mr. Johnston) regretted that he had not yet been able to make any trials with this method adopted by Mr. Bailey. Of the three months described, he was personally inclined to favour that of determining the total nitrogen in the shale, but he was naturally anxious that the most reliable be adopted, and thought a discussion on the subject might be the means of putting them in the way of arriving at this desirable end. He had much pleasure in inviting any members present to favour them with their experiences or any suggestions which they might have to make. Mr. Gray explained that he was in the habit of taking a sample from each natural parting in the shale, and thought it was advisable to take a fairly large sample; in some cases he took from four to eight samples from the seam, according to its thickness, and tested each one separately by itself. He used a small experimental retort holding from 20 to 25 lb. of the shale, and treated it just as it is done in the Young and Beilby retort, distilling off the oil and gradually raising the heat till a good ammonia heat was got while a current of steam was passing all the time. The ammonia water was carefully measured and titrated in the usual manner. He found the results to agree well; the yield of ammonium sulphate was about a pound less than what is got from the Young and Beilby retort in good working order.

Mr. D. R. Steuart said that he had for many years accepted as correct, without any suspicion, a sample taken by making a bore through the depth of the seam, but he subsequently found that bores taken within a few inches of each other sometimes varied in regard to the oil to the extent of several gallons per ton, so that if he wished to know the exact value of the shale, he found it necessary to take the trouble of making a cutting of some size through the seam, or of making an average of several bores. He distilled 2 lb. of the shale in a 2-inch wide tube in the old way, catching all the ammonia in cold water; and if an accurate idea of the amount of ammonia was required, he, at the same time, and alongside in the same furnace, carried out a similar experiment, for comparison, using a shale the exact value of which, as an ammonia producer, had been determined in the works. It was, of course, possible to arrive at an approximation to the amount of ammonium sulphate obtainable, as Mr. Johnston had said, by multiplying the result by two; but he did not claim, in his letter to Mr. Johnston, any special accuracy for the result arrived at in that way; he had, however, found the comparative method satisfactory, and had used it invariably. The total nitrogen in the shale might be used as a basis for calculation, or rather for judgment, but he had not found that the results obtained by the soda-lime method always accorded with each other as closely as he could have wished. He had, however, not studied the subject specially.

Mr. E. M. Bailey said that his experience was, that some shales gave an oil containing a much larger quantity of highly nitrogenous matter than that obtained from other shales, so that the amount of nitrogen present in the oil bore a much larger proportion to that obtained as ammonia in some cases than in others. For this reason he considered, that in the case of two shales so differing, it would be incorrect to assume that the same percentage of the total nitrogen in the shale would be obtained as ammonia in both cases. At one time he used Mr. Steuart's plan, but for the last six years he had used the method communicated by him to Mr. Johnston, and had found it perfectly satisfactory.

Mr. W. Carrick Anderson asked if any of the chemists connected with the various works had compared the results of the Kjeldahl method with those of the soda-lime method.

Mr. Johnston, in reply, said that he had not tried the method.

The Chairman thought that one of the most interesting features in connection with this discussion was that it had shown what a very great need there was for a full ventilation of the whole matter of shale assaying, and that as a Society they ought to do what they could to promote a final settlement of the matter. He would therefore propose that a small sub-committee, consisting of Mr. Johnston, Mr. Steuart, Mr. Bailey, and Mr. Gray, all gentlemen who had spoken on this subject, should take up the matter and report further upon it.

Mr. Harris said he had much pleasure in seconding that.

The Chairman said that their time had now expired, but that it was evidently the desire of the members that this meeting should be adjourned for the further discussion of matters of interest and importance to the oil industry. The Committee would therefore arrange for the holding of the adjourned meeting in February, so as to give sufficient time for the preparation of the various matters to be discussed. In bringing the business of the meeting to a close he proposed that they should convey their hearty thanks, firstly, to Mr. Henderson and the local members and friends who had taken so much trouble to ensure the success of the meeting, and, secondly, to Mr. Henderson, Mr. Crichton, Mr. Bryson, and Mr. Moffat Johnston, who had provided the meeting with such interesting and valuable communications.

Mr. Jno. Fyfe gave expression to the interest with which he had listened to the proceedings, and was deeply struck with what Mr. Bryson had said regarding the output from his retort. He thought that Mr. Bryson was wrong in stating the cost of retorting with the Young and Beilby retorts at such a high figure, bringing out a difference of 1*s.* 10*d.* per ton, but pointed out that even supposing the result of the improved mechanical arrangement and the saving in fuel and labour to be 1*s.* 6*d.* per ton, this gain meant not less than 200,000*l.* per annum to the Scottish oil trade.

Yorkshire Section.

Chairman: Thos. Fairley.

Vice-Chairman: Christopher Rawson.

Committee:

H. E. Aykroyd.	J. J. Hummel.
F. W. Branson.	W. McD. Mackey.
J. Cohen.	H. R. Procter.
N. Farrant.	F. W. Richardson.
T. Glendinning.	A. Smithells.
A. Hess.	Geo. Ward.

Hon. Local Secretary and Treasurer:
H. R. Procter (*pro tem.*). The Yorkshire College, Leeds.

Meeting held on Monday, November 29th, 1897.

MR. THOS. FAIRLEY IN THE CHAIR.

NOTE ON THE EXAMINATION OF SEWAGE AND POLLUTED WATERS.

BY WM. MCD. MACKEY AND JAMES MILLER.

The following results are given for the sake of comparison between the albuminoid-ammonia process and the determination of nitrogen by the Kjeldahl process as applied to the analysis of sewage and polluted waters.

The determinations have been made on samples of River Aire water, four marked "A" being taken from above Leeds, and four marked "B" below Leeds, at a point below where the discharge from the Knostrop Sewage Works enters the river.

The albuminoid ammonia process was carried out as follows:—600 c.c. of distilled water was distilled with a little sodic carbonate till ammonia-free, cooled, and 25 c.c. of sample added; the distillation being then continued as in the case of a drinking water. After the addition of alkaline permanganate all the albuminoid ammonia was found to come over in the first 150 c.c.

The nitrogen by the Kjeldahl process was determined in 500 c.c., boiling down with 10 c.c. strong suphuric acid (subsequently deducting nitrogen equivalent to the ammonia found as "free"), and proceeding in the usual way for an organic nitrogen determination.

—	Grains per Gallon.			
	Albuminoid Ammonia.	Equals Nitrogen.	Nitrogen by Kjeldahl.	Nitric Nitrogen.
A. Tuesday, 11.15 A.M., 8th Sept. 1896.	0·137	0·113	0·45	Nil.
B. Tuesday, 4.0 P.M., 8th Sept. 1896.	0·325	0·267	0·85	Nil.
A. Thursday, 12.0 A.M., 29th Oct. 1896.	0·087	0·071	0·64	Not done
B. Thursday, 3.0 P.M., 29th Oct. 1896.	0·157	0·129	0·52	Not done
A. Thursday, 1.45 P.M., 26th Nov. 1896.	0·145	0·120	0·50	0·030
B. Thursday, 4.30 P.M., 26th Nov. 1896.	0·143	0·118	0·50	0·015
A. Wednesday, 11.0 A.M., 24th March 1897.	0·089	0·073	0·31	0·064
B. Wednesday, 11.15 A.M., 24th March 1897.	0·089	0·073	0·58	0·100

The nitric nitrogen in the first two experiments was nil. In the next two it was not determined owing to inadvertence, but except in the last two it could not materially affect the figures for nitrogen, and in these cases to a small extent only; consequently the nitric nitrogen has been disregarded.

Our object was, if possible, to find a ratio between the nitrogen as determined by the Kjeldahl process, and that by the albuminoid-ammonia process, which could be applied to a number of determinations by the latter process, on samples collected since January 1894. In this we have failed, but we considered the figures found worth recording.

Though the albuminoid-ammonia process has proved such a useful method to chemists in the analysis of drinking water it is not suited to that of sewage or trade effluents, a more absolute means for the determination of nitrogen such as the Kjeldahl method affords is required. At the same time, were this process generally adopted a uniform method of procedure among chemists would be desirable, including some modification eliminating any error due to the interference of nitric nitrogen.

Discussion.

Mr. T. Fairley thought that the difficulty regarding the nitrogen due to nitric acid might be surmounted by using Frankland's method of reduction with sulphurous acid.

Dr. J. B. Cohen stated that he had once made a comparison of Wanklyn's, Kjeldahl's, and Dumas' methods operating upon air-dried commercial gelatine and found 10·5, 13·5, and 15·2 per cent. respectively in the same sample. These results were however obtained using the original Kjeldahl method; the modified one might give better results. If commercial sulphuric acid were used for Kjeldahl's method a blank experiment should always be performed, as the acid always contained small and varying quantities of nitrous or nitric acids.

Prof. H. R. Procter said that in recently collating some results of other chemists, he had found that the average quantity of nitrogen in gelatine, whether determined by the Dumas or the Kjeldahl process, ran about 18 per cent. Gelatine solution gave a larger amount of albuminoid ammonia after slight putrefaction than when quite fresh. The estimation of strong solutions by Nesslerising or by any other colorimetric method was very inexact, because the stronger solution must be diluted to a strength approximately, the same as the weaker solution, and any error of observation must thus be multiplied by a large factor. Very considerable differences in the albuminoid ammonia found might be caused by variations in the time of distillation, and the volume distilled; and in factory-effluents matters might easily exist, such as aniline, which yielded "albuminoid ammonia," while containing no putrescible matter whatever.

Mr. Mackey stated that the nitrogen in his sulphuric acid had not been determined. His samples of water had in nearly all cases been taken on the same day of the week, generally Wednesday, so that the results might be more strictly comparable.

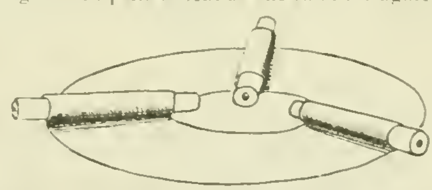
After the discussion on Mr. Mackey's paper, Mr. F. W. Branson exhibited a model of Marconi's instrument for telegraphing by means of Herzian waves and explained the action of the "Coherer," the most important part of the apparatus, by means of which the waves are detected.

Mr. Fairley then showed an apparatus for recovering ether evaporating from open vessels.

A bell jar connected by an elbow tube to a condenser is inverted in a flat crystallising dish over the vessel containing the ethereal solution. Some water is then poured into the outer dish to make the joint tight. The crystallising dish is then set upon the water-bath and the ether distills off.

Mr. Fairley also showed a new substitute for a pipe-clay triangle of his own design.

A ring of iron plate is bent as shown in the figure, when



the pieces of pipe shank which are inserted in the grooves thus formed may be adjusted as required.

New York Section.

Chairman: Charles F. Chandler.
Vice-Chairman: T. J. Parker.
Committee:

H. Clementson.	G. A. Prochazka.
Virgil Coblentz.	Clifford Richardson.
H. Endemann.	Wm. Jay Schieffelin.
W. F. Fuerst.	R. C. Schupphaus.
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F. J. Lederle.	

Hon. Treasurer: R. C. Woodcock.
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Dr. H. Schweitzer, 77, William Street, New York, U.S.A.

SESSION 1897–98.

Meetings will be held on the third Friday after the first Monday in every month.

Meeting held at the College of Pharmacy on Friday, November 19th, 1897.

MR. THOS. J. PARKER IN THE CHAIR.

ON THE SULPHUR CONTENT OF BITUMENS.

BY S. F. AND H. E. PECKHAM.

We have read with greatest interest Dr. Endemann's second paper in this Journal upon the analysis of asphalts (this Journal, Feb. 1897, 121), as well as the discussion thereon, and later, upon the paper contributed by ourselves (*ibid.* May 1897, 424).

Dr. Endemann took up the subject of asphaltums only a few months prior to the publication of his first paper. He then, after an investigation in his own way of Trinidad pitch, a Mexican asphalt: and a Texan maltha, propounded certain general conclusions in relation to solid and semi-solid bitumens, with which conclusions we from first to last disagree. These, as we believe, wholly erroneous conclusions do not detract from the value of his work, which, it appears to us, has brought out much respecting the decomposition products of the bitumen extracted from Trinidad pitch and the Texas maltha. This is the scope of his work and nothing more. So far as our experience goes, it is confirmed in every particular by Mr. Clifford Richardson in his discussion on the second paper.

Dr. Endemann tells us that he adopts the name "asphaltene for the reason that it is the main constituent of the asphalt," and "petrolene for the reason of identity." He further says that the "asphaltene of different asphalts has the same composition and identical properties." He also states that asphaltic acid is the cause of the solution of metallic oxides in chloroform. Of more importance is this statement: "Enough, however, had been learned, namely, that what we call petrolene is a complicated mixture. It contains a series of compounds perhaps not even all belonging to a single aliphatic series, but comprising representatives of several such."

In our former communication, we asked several questions, the answers to which appear to us to be fundamental. To put these questions in other words: Does the word asphalt or asphaltum designate an individual substance or a class of substances? Has Dr. Endemann, or anyone else, determined the constituent substances or compounds that together form any variety of natural asphalt or asphaltum? If so, where are the results published? Refined asphalts are manufactured articles, whether solid or semi-fluid. What name does he give the many other solid bitumens, that are wholly unlike the one he selects in composition? Are they asphalts, asphaltums, or what are they?

He states that he treats his natural asphalt so as to get rid of all extraneous substances. He gets rid of his petrolene, which is a complex mixture of undetermined bodies; of all the sulphur, whether free or combined; of all the nitrogen, if there is any, and finally reaches his asphaltene, which he claims is a constituent of all asphalts, but which we claim is nothing but a residue of decomposition, which may or may not be common to all solid bitumens when subjected to his peculiar process. As we understand him, he regards sulphur as a foreign substance, mechanically mixed with solid bitumen. We do not agree with this conclusion. We believe that sulphur has played a very important *rôle* in the formation of solid bitumens, and are inclined to the opinion that solid bitumens are in many instances largely the result of the action of sulphur upon fluid or semi-fluid bitumens. As we have said previously, sulphur burns out the hydrogen from bitumen, and this reaction may proceed slowly at ordinary temperatures or rapidly at higher temperatures. It is not necessary that the sulphur should be free. It has long been known that sulphates are deoxidised when brought in contact in solution with dead organic matter. If the organic matter has already been distilled into bitumen, the reaction proceeds through double decomposition, a carbonate of the base being formed and hydrogen sulphide, free sulphur, and a sulphur substitution compound remains as a part of the bitumen (Geol. and Chem. Essays, T. S. Hunt; pages 87, 99, 115, 163, 230). In proof of this latter statement we have the direct evidence furnished by Mabery's researches upon the sulphur compounds of Lima petroleum. These are paraffin petroleums, and the sulphur compounds isolated are paraffin derivatives. As nothing has been proved concerning the sulphur compounds of solid bitumen, except the few facts stated in our first paper, there is very little upon which to base any general conclusions concerning the sulphur content of bitumens.

In the first place, no writer who has written upon this subject has a word to say concerning the method that he used for determining the sulphur; consequently we have never had any other instructor than experience. While our tutelage has been extended it has also been thorough. It began several years ago while in Southern California, in an attempt to establish, at the request of the Patent Office examiners, specific differences between a solid asphaltic residuum obtained from California petroleum, the same oxidised by prolonged action of air, and after treatment with sulphur. Nearly all of this work was carried on in association with Dr. Frederic Salathè, who said he had examined a large number of natural and artificial bitumens for sulphur. The residuum and sulphurised residuum were tested for sulphur by boiling with fuming nitric acid in a flask with an inverted condenser, and no sulphuric acid was found in the liquids. This method of testing was not then further investigated. Other reactions showed that a compound was formed, which, when oxidised with nitric acid, produced styphnic acid, which is a trioxynitrobenzol—a compound not only interesting, as showing the action of the sulphur, but also as showing that the residuum consisted of benzols. Neither transmitted air nor direct oxidation with nitric acid converted any portion of the residuum into styphnic acid. Treatment of the residuums by deflagration with $NaCO_3$ and KNO_3 showed sulphur in the asphaltic residuum, but only a trace in the same after treatment with sulphur at a temperature of 400° F. At that temperature the sulphur almost fully burned out with hydrogen, and the bitumen was left dryer and more brittle. This is substantially what Dr. Endemann does to get rid of his sulphur. He has left a decomposition product and nothing else.

Later, we had occasion to determine the sulphur in various bitumens, and recourse was had to boiling with fuming nitric acid, with an inverted condenser. After repeated attempts it was discovered that no quantitative determinations of any value could be made in this way. Addition of potassium nitrate, chlorate, or permanganate was of no avail, and we finally abandoned all attempts in this direction. We believe we have tried every method for the determination of sulphur except the bromine method, and that has never appeared to us to be practicable with solids. Nor has the method of Carius, which we mention here, for fear that by its omission we might be misunderstood. The method which we have finally adopted and now always use, is one that was used for the determination of sulphur in the Pacific Coast coals. (See Geology of California, vol. II., The Coast Ranges, App., Cambridge, Mass., 1882, p. 45.) It is susceptible of great accuracy if conducted with care. Two grms. of the bitumen are intimately mixed in a mortar with 16 grms. each of pure, dry $NaCO_3$ and KNO_3 and the whole projected in small portions at a time into a 2-oz. platinum crucible, heated to dull redness, or no hotter than is necessary to cause the mass to deflagrate. Experience will soon teach the manipulator how best to conduct the operation. After deflagration and complete fusion, the mass is dissolved in water, hydrochloric acid added, and the solution either evaporated for removal of silica or immediately filtered, and the sulphuric acid precipitated in the hot acid solution. The barium sulphate is washed, dried, and weighed with the usual precaution. In illustration of the value of this method of determining sulphur when applied to the practical examination of bitumens, we give below the results of an examination of Turrelite—the bituminous coquina found in Uvalde County, Texas.

The mineral itself is a grey mass, apparently of shells, cemented together with bitumen. It is exceedingly tough and difficult to break—a property that is easily accounted for when the material is deprived of its bitumen by being digested in chloroform. The mineral residue is then dis-

covered to be a coquina or shell limestone possessing considerable stability without the bitumen. The shells are cemented together, and the cavities of many of them contain rhombspar and fragments of other shells, showing that the shell rock had been formed before the bitumen was projected into it.

Analysed by solvents, there were obtained the following results: —

	Per Cent.
Soluble in petroleum ether	9·415
Soluble in spirits of turpentine, after	4·121
Soluble in chloroform	Trace
Soluble in dilute hydrochloric acid	84·894
Sulphur in residue	0·138
Silica and clay	1·432
	100·000

The average total bitumen is 13·536 per cent., of which the petroleum ether soluble is 69·555 per cent., and the spirits of turpentine soluble is 39·415 per cent. This gives a bitumen wholly soluble in spirits of turpentine, of which 69·5 per cent. is soluble in petroleum ether—a very high-grade bitumen. Five grms. of the rock, analysed in duplicate, gave an average of 1·13 per cent. of sulphur in the rock. 1·13 per cent. − 0·1381 per cent. found in the mineral residue, leaves 0·5919 per cent. of the rock, of sulphur in the bitumen, or, of the bitumen 7·328 per cent.

Digestion for several days in water at 60°—70° C., yielded a solution containing 0·126 per cent. of the rock, which consisted of a trace of organic matter and calcium carbonate. There were no sulphates soluble in water. The portion dissolved by dilute hydrochloric acid consisted of calcium carbonate with traces of magnesium, iron, and sulphuric acid. The insoluble residue remaining on the filter consisted of silica, clay, and very small grains of pyrites of appreciable size. Caustic alkalis were without appreciable action on the rock. Fuming nitric acid acted upon it with great energy, dissolving the mineral matter and converting the bitumen into a brittle, charred mass of carbonaceous matter. The bitumen extracted from this rock, when analysed by itself, yielded to—

	Per Cent.
Petroleum ether	71·28
Boiling spirits of turpentine, after	28·72
	100·00

The results are nearly identical with those obtained from the rock. The sulphur in the bitumen amounted to 7·582 per cent. The bitumen is not acted on by water, strong acids, or alkalis. Fuming nitric acid coverts it into a friable, carbonaceous mass.

The above-described work is analytical, as far as it goes. It is admittedly very imperfect, but it tells, nevertheless, its story concerning the constituents of this bituminous rock. There is no reason for believing that there is any oxygen compound in the bitumen contained in this rock. The bitumen is very pure, and while it contains a very large percentage of sulphur, there is no reason for supposing that there are any bases in it made soluble in chloroform by means of asphaltic acid. The bitumen contains only a trace of mineral residue when burned.

We have a large number of determinations of sulphur in solid bitumens. The amount varies from ¼ per cent. to 7½ per cent. We have never found a solid bitumen free from sulphur. While some bitumens, rich in sulphur, have no perceptible odour, others, like that of the Dead Sea, possess a strong odour of garlic, and without any doubt will be found to contain sulphur compounds that confer upon them very important properties. These compounds are not mechanical impurities, but are constituents, and cannot be overlooked or gotten rid of in any process of analysis that analyses rather than decomposes the natural crude bitumens.

The interpretation of these results in relation to technology is not without value. They indicate a very pure bituminous limestone, free from pyrites and organic matter, containing a heavily sulphurised bitumen. We did not for this reason condemn the material, but we called attention to the fact that, like other sulphur bitumens, this one was very brittle, and we advised great care in its use, particularly in reference to overheating.

The compound believed to be a thio-salt of iron that was obtained from the chloroform solution of Trinidad pitch, has been analysed, and contains—

	Per Cent.
C	83·0000
H	7·0170
S	5·8710
Fe	3·711[illegible]
	[illegible]

This corresponds to $C_{111}H_{120}FeS_3$, of which the theoretical composition is:—

	Per Cent.
C	83·0275
H	7·4813
S	6·0000
Fe	3·4912
	100·0000

One of the determinations gave hydrogen, 7·460 per cent. It will require much further research to determine what carbon group has been deprived of hydrogen to form this compound.

We wish further to call attention, in connection with this research of Dr. Endemann, to the great difficulties often attending the correct determination of carbon in compounds of carbon and hydrogen that are solid, or even liquid, with very high boiling point. No one will call in question the high authority of C. M. Warren as an investigator of this class of substances. In several conversations held with him many years ago he assured us that he had often found great difficulty in completely burning carbon, even in oxygen gas, when the combustion was assisted by cupric oxide used in any manner. Our own experience confirms Warren's. While we have found that we could duplicate hydrogen determinations to the third place of decimals, the carbon determinations were persistently short in amount, yet not so widely variable as to discredit the accuracy of the determination. We call attention to these facts to show that extreme care should be exercised when determining oxygen by difference.

We again submit that we will gladly welcome any system or method of analysis that furnishes better results than our own.

Discussion.

Mr. Clifford Richardson said he was very glad to see how many of his own results were confirmed by what what Prof. Peckham had said. He might, however, call attention to the fact that Prof. Peckham's determinations of sulphur were considerably lower than his on the same material, and that, so far as his experience went in the determination of sulphur, the method which Prof. Peckham pursued always gave lower results. It was almost impossible to heat a bitumen with carbonate of soda and nitrate of soda without losing some of the sulphur by volatilisation, whereas the burning of the bitumen in a closed tube in a stream of oxygen could not result in the loss of any which was there in the organic form. Prof. Peckham found 7·5 per cent. in the Uvalde bitumen, as against his 9·8 per cent., and the same ratio in the analysis of the Trinidad preparation which he mentioned. He could also confirm what Prof. Peckham had said in regard to the extreme difficulty of making an ultimate analysis of hydrocarbons such as were found in asphalt. Mr. Byerley, his assistant, has spent months in developing a method which would give satisfactory results. In the first place, carbon and hydrogen had to be determined in the presence of nitrogen and sulphur, which alone made the process decidedly difficult. In the second place, as has been shown by a great many investigators, the attempt to burn these higher hydrocarbons, even in oxygen, frequently resulted, not only in the production of hydrogen and water, but also of acetylene and similar unsaturated compounds. Even with the best and most careful work he and his assistants had been able to devote to such analyses, it was sometimes necessary to make five or six combustions before concordant results could be obtained.

Mr. Edwin F. Hicks said it seemed to him that, had the sulphur determinations been carried out by heating the bitumens with fuming nitric acid in closed tubes, instead of in an inverted condenser at the ordinary pressure, much more satisfactory results would have been obtained. Digestions of this kind could be easily and effectually accomplished, if, after heating the tube for a short time at a comparatively low temperature, and allowing it to cool, the capillary of the tube were broken, thus relieving the pressure; then, upon resealing, the temperature could be raised to the point necessary for complete decomposition with but little risk of the collapse of the tube, which so frequently discouraged the employment of this method. Had Mr. Richardson, in the course of his determinations of sulphur in bitumens, employed nitric acid under pressure?

Mr. Richardson replied that the only check he made on the determination with combustion with oxygen was by oxidation with nitric acid, and that he had found no difficulty in getting results that agreed within 0·1 per cent. on Trinidad asphalt with nitric acid and a little chlorate of potash at ordinary pressure; but he had no doubt that in most cases, if there was any large amount of work done in that way, it would be done in a sealed tube, that being the most desirable way to accomplish difficult oxidations.

Dr. W. Jay Schieffelin inquired whether he had tried connecting several combustion tubes together.

Mr. Richardson replied that if they had been merely highly volatile hydrocarbons he should have done that, but that these were not volatile. He thought that that would be a very good idea, and thanked Dr. Schieffelin for the suggestion.

Mr. Deroode asked, in regard to this method of determining sulphur used by Mr. Richardson, what became of the nitrogen in these compounds. Was it converted into nitric acid and absorbed by the normal alkali?

Mr. Richardson replied that he did not know. He had seen it in potash and in ordinary combustions. The amount of nitrogen, however, was always very small, and would not seriously affect the determination, but it might to a certain extent.

Mr. Deroode said that with nitrogen as high as 0·8 it would seriously affect the sulphur determination.

THE APPLICATION OF CHEMISTRY TO THE STUDY OF THE MAGNETIC PROPERTIES OF IRON.

BY BERTRAND S. SUMMERS.

In the present state of science it is difficult to trace other than general relations between chemical composition and physical properties of substances in general. As the chemistry of iron is developed about as well as any branch of the science, we should naturally look for some deduction more or less useful from the study of it. Although there is much that is wanting in this branch of chemistry, it is often possible to trace relations which are exceedingly helpful in commercial practice.

There are reasons to expect a more intimate knowledge of the subject if the proximate analysis of iron can be brought to the point where it can be termed practical and useful. It would seem that a great many of the secrets of iron are locked up in its proximate composition. The microscope has proved an important factor in this connection, but unfortunately, in the present state of the art, it has its limitations. However, it is an important aid when used in connection with chemistry, and often gives a clue to a mystery where chemistry has failed.

Magnetism and magnetic permeability seem to be properties of the iron itself, but are influenced to a very great degree by the metalloids commonly associated with iron. As the latter property is the one that concerns the engineer the most, and as the writer's investigation has been applied chiefly in this field, the paper will deal almost entirely with this branch of the subject. It can in nearly every case be assumed that magnetism and magnetic permeability are opposed to each other, and that a state of the metal favourable to one will be unfavourable to the other.

In examining an iron for magnetic permeability, it is also quite necessary to take into consideration another property of iron, *viz.* hysteresis, that is, the energy lost in magnetising iron, due to the magnetic inerta of the metal, or the resistance a metal offers to magnetisation. This lost energy is usually rated in watts per pound lost under given conditions.

In general, a metal of high magnetic permeability will usually have a low hysteresis, but there seems to be little or no relation between these two properties. It is not infrequent to find a metal of high magnetic permeability and high loss by hysteresis.

These two properties of iron are what govern its adaptability for dynamo construction and similar purposes. It is with these that the metallurgist is chiefly concerned in the foundry, and as this investigation has been carried on largely with cast metal, most of the deductions are drawn from these varieties of the metal.

In experimenting with cast iron it is exceedingly difficult to draw any trustworthy conclusions, for the reason that the large contents of metalloids make it almost impossible to trace the variations in permeability to any one, or any combinations, of the metalloids. The high percentage of graphite is particularly obstructive in this way, and although it has but little or no chemical effect, its effect on the physical character of the iron makes it difficult to experiment under the same conditions at different times.

After endeavouring to draw some conclusions from numerous tests on cast iron, attention was devoted to making some special alloys. For this purpose wrought-iron borings were mixed with varying proportions of ferro-silicon of the following analysis:—

Silicon.	Graphitic Carbon.	Combined Carbon.	Sulphur.	Phosphorus.	Manganese.
Per Cent. 7·25	Per Cent. 1·95	Per Cent. 0·31	Per Cent. 0·044	Per Cent. 0·597	Per Cent. 0·88

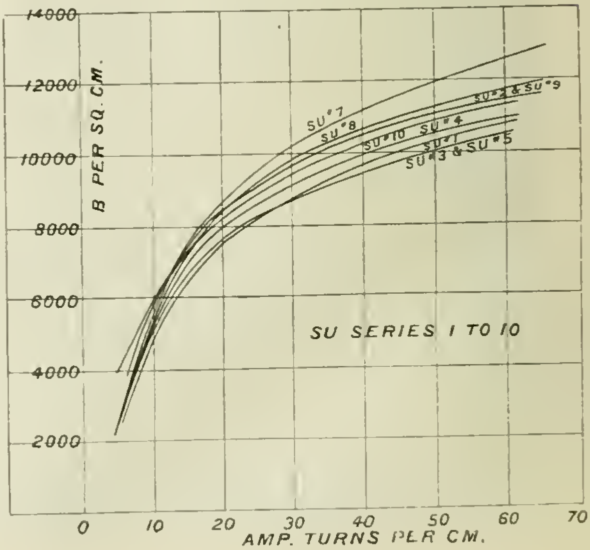
With these two ingredients a series of irons was made. They were placed in a graphite crucible and heated in an ordinary brass-foundry furnace for several hours. When the metal had come to perfect fusion, test bars were cast and tested, and drillings from the same analysed.

These alloys were decidedly peculiar. They did not contain more than a trace of graphite, but the content of combined carbon was usually over 2 per cent. The content of silicon was varied by adding different amounts of the ferro-silicon iron.

A phosphorus iron was made by adding red phosphorus to wrought iron, and when it was desired to raise the phosphorus content, this alloy was added to the mixture of ferro-silicon and wrought iron.

Some of the more characteristic curves are here appended in order to make the comparison more clear. This series was

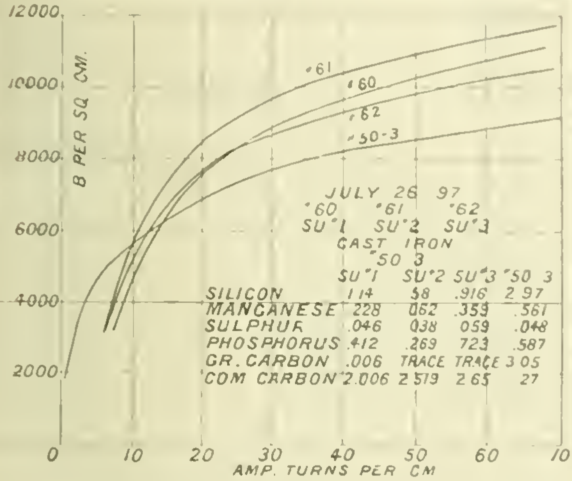
General View of Curves.



designated as SU series, and the curves are so denominated. The alloys were compounded by endeavouring to keep the other constituents constant while the silicon was varied. This plan was a failure, owing to uncertainties of mixing, and the variation of the carbon due to absorption of carbon from the pot. However, by selecting the curves where the least variation is noticed, we are able to draw some deductions.

In comparing these curves, let us first examine them with reference to the variation of silicon. The curve in Plate I.,

Plate I.

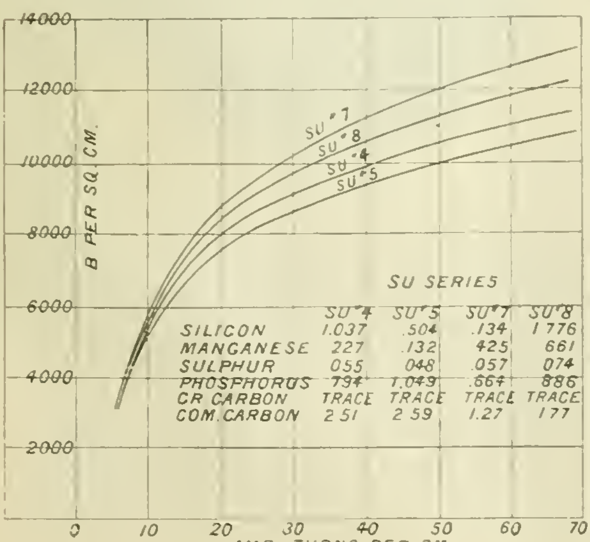


marked # 50—3, is a curve of a very good cast iron, and serves as a means of comparison. It will be noticed that curves SU #1 and SU #3 are more nearly alike than curve SU #2. It might seem at first sight as though this might be due to the silicon content, but a comparison with other curves will show it to be otherwise.

We would naturally expect SU #2 and SU #3 to correspond more closely than SU #1 and SU #3, as the carbon contents are more nearly alike. This difference is due probably to a difference of the state of the carbon and variation in casting condition, which we will discuss later. If we now compare SU #2 with SU #4, we find that their carbon contents are almost identical, while the silicon content in SU #4 is nearly double that in SU #2.

To be sure, the phosphorus varies somewhat, but this does not appear to vary the results, as we will endeavour to show later.

Plate II.

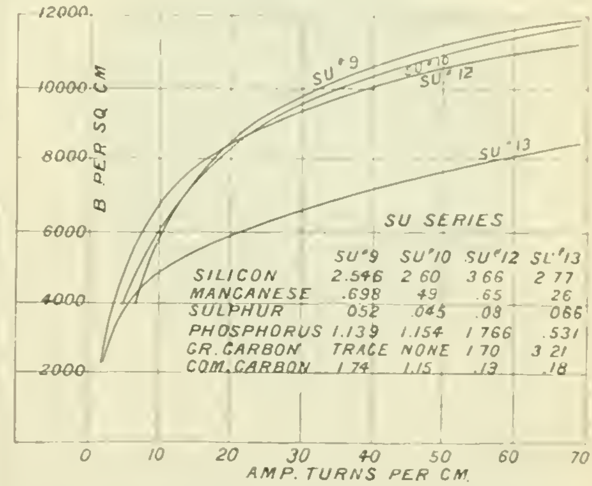


These curves correspond quite closely, showing that this variation in silicon could not have affected the permeability to any marked degree. Finally, if we compare curves SU #8 and SU #9 we notice that the carbon contents are about the same, but that the variation in silicon is about ¾ of 1 per cent. The other constituents correspond quite closely, at least do not vary greatly. It would seem that if the variation of silicon had any marked effect, the variation is so marked in this case that there should be some effect traceable in the curves. The curves, however, are almost the same throughout their entire range. Also SU #3 and SU #5 are identical while the silicon varies considerably. These with numerous other examples have led to the belief that silicon has little or no direct effect upon the permeability of iron within certain limits. The same seems true in regard to phosphorus.

Comparing curve SU #2 with SU #5, we notice that there is not a great variation in anything but the phosphorus. In this there is a variation of nearly 1 per cent. On comparing the curves, we notice that while there is some variation in the curves, there is hardly what would be expected had phosphorus a marked effect on permeability.

Again, comparing SU #8 and SU #9, we notice that the variation in phosphorus has had no more effect than the variation in silicon.

Plate III.



The test bars designed to test the effect of sulphur and manganese were unfortunately so hard that they could not be turned.

It must be here noted that the comparisons above given are taken only as a general indication and not as absolute proof. It is only in connection with other experiments that these ideas are more clearly substantiated.

It now remains to consider the relation of the carbon in the above series. Unfortunately the writer has not had opportunity to distinguish the different state of the carbon in the different test bars, but it would seem from a comparison of the curves that much light would be shed upon the matter if this were done.

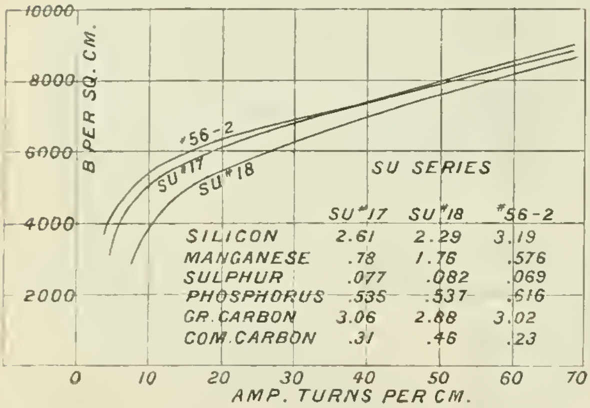
In a general way it will be noticed that the test bars containing the least carbon are a little higher, although the contrary will be noticed in particular cases. Thus SU #2, containing ½ per cent. more carbon than SU #1, is quite a little better than SU #1; the same may be said of SU #3.

These specimens examined under the microscope show a predominance of the structure indicating carbide carbon. The structure is strongly characteristic. The accompanying micro-photographs give a clear idea of them, although in the original the projection is highly coloured. The structure of all the members of this series shows a strong resemblance, but minor differences are noticeable.

Having noticed the results from this series, it seemed desirable to note the effect of manganese and sulphur. In order to discover whether manganese has a marked effect

on permeability, a hand ladle was filled from the regular dynamo cast-iron and a small amount of ferromanganese, containing about 85 per cent of manganese, added. A test bar was then cast and about the same amount of ferromanganese again added, and another bar cast. The first bar contained 0·78 per cent. of manganese, and the second 1·76. The latter was considerably lower in permeability. It will be noticed, however, that the combined carbon has been doubled by the addition of the greater quantity of manganese, and this probably accounts for the lower permeability (see curves SU # 17 and SU # 18). Manganese in all probability effects the magnetic properties through its effect on the carbon.

Plate IV.



This effect is largely governed by the content of carbon and the content of the other elements, particularly silicon. It is safe to say, however, that the influence of manganese plays a very small part when the content is below 1 per cent. This is especially true of high carbon irons, such as cast iron. In general, what is said of manganese may be said of sulphur, as far as the indirect effect is concerned. However, a very small quantity of sulphur has a very powerful effect; this is especially true in low carbon iron.

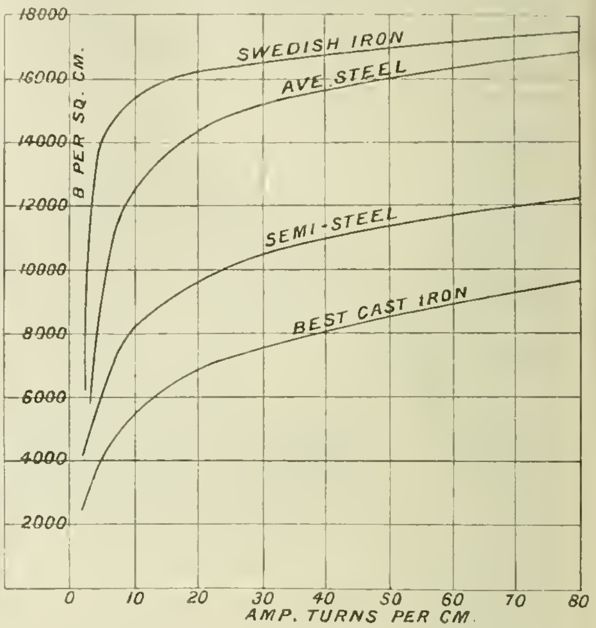
The experiments with sulphur were mostly carried on with a semi-steel containing from 40 to 50 per cent. of steel-rail scrap, and having a total carbon content of about 2 per cent., most of which was in the graphitic state. In these irons when the sulphur rose much over one-tenth of 1 per cent., it was nearing the danger limit, while 13 per cent. was fixed as the maximum for any metal of this composition where even moderate results were expected. It must be noted, however, that a soft graphitic carbon iron could carry this content without much detriment to its permeability. If the sulphur was increased in these high carbon irons a very bad effect is noticeable.

The effect of sulphur is undoubtedly indirect and effects the permeability through its power of promoting combined carbon. One of the test bars of semi-steel, containing about 0·15 sulphur, was so white and hard that it could not be turned, although the other constituents were normal, with the exception of the higher content of combined carbon.

If we return to our consideration of the effect of silicon on the magnetic properties of iron alloys, and consider another alloy of iron, we will find our deduction from the first series confirmed. The alloy selected for these tests was the semi-steel above mentioned, except that the sulphur content was normal.

It will be noted in the accompanying curve the relative position of the metal to cast iron and cast steel. With this alloy the silicon varied about $\frac{3}{4}$ of 1 per cent., and some cases nearly 1 per cent., without any appreciable effect. A variation of $\frac{5}{10}$ of 1 per cent. produced no effect whatever upon the permeability. The silicon in this alloy is always kept above 3·75 per cent., consequently, is always so high that the change in the content could scarcely affect the state of the carbon. However, it was found that the content of silicon had a marked effect on the hysteresis, the loss by hysteresis being very much less when the silicon was high. A variation above 3·75 per cent. had an effect on the hysteresis, where none was noticeable on the permeability.

Plate V.



It would seem fair to presume that silicon has no direct effect on permeability and above the limit necessary for holding the carbon in the graphitic state, it is doubtful if it has any effect. This is also probably true in the irons containing no graphite, but there would undoubtedly be a limit in this case.

In the same way phosphorus can vary greatly in this metal without any noticeable effect. Within the limits that have been experimented with, nothing has been noticed that would lead one to think that phosphorus has any effect on permeability or hysteresis. This limit is over 1 per cent., and covers the range that would be met with in commercial work.

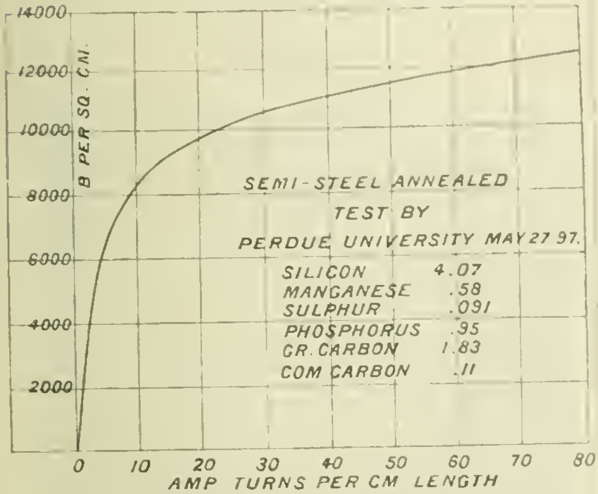
The carbon is undoubtedly the chief factor in the chemical effect on permeability and hysteresis. Practically the same effects were noticed in the semi-steel as in the pot irons previously mentioned. In general a low content of combined carbon indicates high permeability and low hysteresis. It is often noticeable, however, as in the case of the pot irons, that an iron containing more combined carbon than another would have a higher permeability. To discard physical effects this is readily accounted for by the different state of the carbon.

It has occurred to the writer that there was some form of combined carbon that varied in these irons, and the permeability was more affected by the content of this form of carbon than by the total content of combined or graphitic carbon. This form of carbon seems to be that which Prof. Ledebur calls hardening carbon, and is probably the complex crystalline mass isolated by the microscopist under the name of Martensite. Although not having opportunity to investigate this question further, from the meager data at hand, on this phase of the question, it would seem that this is the state of carbon having the most influence in the different varieties of iron in which permeability and hysteresis are important. This is offered merely as a suggestion and the writer has hopes of investigating the matter further.

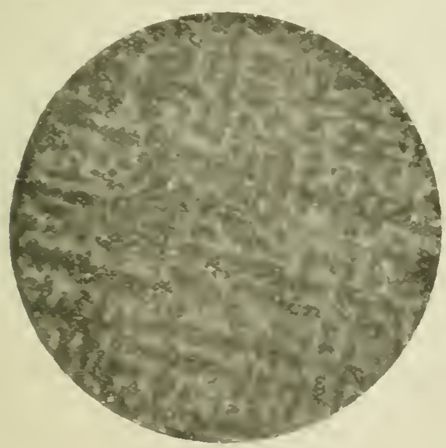
Graphite appears to act as an inert body in regard to magnetic qualities. In the irons where its content is one of the predominating elements, a considerable variation is often noticed without much variation in the permeability. Its effect is that of an inert body except it tends to open the grain of the metal and thereby decreases the density,

which of course effects the permeability. In the case where an attempt is made to close the grain of cast iron for the purpose of raising the permeability, the permeability is more often lowered than raised. While the graphite may be decreased in this way and the density of the specimen raised, the beneficial effects are absent for the reason that the combined carbon is increased and the permeability is lowered by its effect. If the graphite can be decreased without raising the content of combined carbon, the permeability will unquestionably be higher.

Plate VI.



To show the radically different structure of the different specimens discussed in this paper, some micro-photographs will be found of some of the principal alloys. The photograph marked No. 6 is a permanent magnet made of cast



No. 1 (SU = 8).

iron. It is very high in combined carbon. Its structure after etching more closely resembles steel than any of the other irons. The complex formation of this iron is quite apparent. The sample has been hardened, after it was cast, thereby increasing the percentage of combined carbon. Its structure is quite unique compared with the other irons.

The graphite in the photographs of cast iron and semi-steel show very clearly the predominance of this metaloid in these metals. No. 5 illustrates the structure of the test bar giving the curve marked = 50—3 and it is one of the highest cast irons examined.

The other photographs are of the special pot metals and are very characteristic and unique. They show very

No. 2 (SU = 5).

No. 3 (SU = 2).

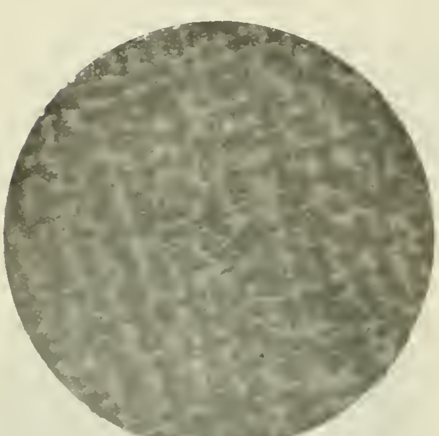
closely the effect of the high combined carbon content. The structure is characteristic of the carbide form of carbon which predominates in all of these pot metals.

Photographs 2 and 7 are of test bar which gave the curves SU = 5, and SU = 3, which curves are identical throughout their range. The minor differences in structure are probably due to difference in etching conditions.

No. 4 (Semi-Steel).

No. 5 (Cast Iron.—Curve # 50—3).

No. 6 (Cast Iron.—Permanent Magnet).



No. 7 (SU # 3).

The physical state of the metal is equally or more potent in its effect upon the magnetic properties of iron, and it is this effect which makes deduction from experiments on chemical effects more or less general. An almost carbonless iron will often be found of low permeability and high hysteresis in comparison with its class. After it is annealed, it will sometimes give results very much better than its class. This seems to be due to an internal stress which has resulted from some heat treatment, and inasmuch as the specimen is nearly pure iron it must be purely physical. This might be attributed to an alotropic state of iron similar to that proposed by Prof. Osmond, of France, but whatever be the facts, the physical condition of the metal must ever play an important part in calculations of this kind. It does not, however, make the chemical study useless. It is often surprising to note how closely one can figure from chemical results, and the chemical study of these irons have been of high commercial importance where it has been pursued.

SULPHURIC ACID AS A REAGENT IN THE ANALYSIS OF FATTY ACIDS.

BY E. TWITCHELL.

When concentrated sulphuric acid is brought in contact with any fat, a decided chemical action is observed. This reaction appears to involve chiefly the unsaturated fatty acids of the fat, and consists primarily of the addition of H_2SO_4 to the unsaturated carbon atoms of these acids. The products of this combination have been studied to some extent, and have found some practical uses; notably in dyeing and calico printing with Turkey red. It is not to the commercial uses of these bodies, which I believe are destined to greater importance in the manufactures, that I wish to call your attention, but to the possibility of developing exact methods for the analysis of fats by the aid of concentrated sulphuric acid.

I do not know that any attempt has been made to employ sulphuric acid as a reagent in any of the quantitative methods which have of late become of such importance in the examination of fats. The nearest approach to such a method is the Maumené thermal reaction, which, indeed gives remarkably exact results considering the imperfect apparatus and manner of manipulation usually employed. The object of this process is clearly to measure the heat of combination of the sulphuric acid with the two or more unsaturated fatty acids of the fat. A thermal effect as definite as this would imply a definite chemical reaction as a cause, and this strengthened my belief that quantitative results could be obtained from the action of sulphuric acid on fatty acids. If this were the case, then a separation of the unsaturated oleic, linoleic, and linolenic acids from the unacted on, saturated palmitic and stearic acids could be obtained, and possibly a difference in the action of sulphuric acid on the different members of the groups of unsaturated acids might show itself. With this in view, the following work was undertaken, during the course of which I observed some peculiarities in the manner in which sulphuric acid acts on these bodies, as well as some properties of the resulting compounds, which I do not think have been published before. For this reason a somewhat detailed description is given of some of my preliminary experiments which, from the analyst's point of view, are failures, but which brought out some of the characteristics of these interesting compounds.

At ordinary temperatures, or even at 100° C., concentrated sulphuric acid has no effect on the saturated fatty acids of ordinary fats. Experiments with pure palmitic and stearic acids have satisfied me as to this. It, however, combines with oleic acid at very low temperatures, and its action on linoleic acid must be very similar to that on oleic acid, as some of my experiments with mixtures which undoubtedly contained large quantities of linolic acid, would indicate.

When sulphuric acid acts on oleic acid at not too high temperatures a compound is formed which has been studied by various chemists and was supposed to be a sulphonic acid, sulpho-oxy-stearic acid—

$$(OH)C_{15}H_{30}CH_2CH(HSO_3)COOH.$$

Later Benedikt has shown that it is more probably a sulphuric ester of oxy-stearic acid, stearo-sulphuric acid, $C_{15}H_{31}CH_2CH(HSO_4)COOH$. This will be seen to be the sum of 1 mol. of oleic and 1 of sulphuric acid. This substance has properties which should make its separation from the fatty acids comparatively easy. It is readily soluble in water. It is a strong acid, dibasic; the acid hydrogen of the HSO_4 group being comparable in strength

with that of sulphuric acid itself, while the hydrogen of the carboxyl group has the properties of that of other fatty acids. This fact furnishes a simple and beautiful method of examining and identifying compounds of this nature, as the HSO_4 group can be sharply titrated with standard caustic soda solution, using methyl orange as indicator, and then phenolphthalein can be added and the carboxyl as accurately titrated. The acid strength of this compound is shown by another remarkable property. If the aqueous solution of stearo-sulphuric acid be salted out with common salt or sodium sulphate the pure compound is not obtained, but large quantities of the sodium salt are formed by double decomposition between the stearo-sulphuric acid and sodium chloride or sulphate, and this even in quite an excess of mineral acid. This fact has been noted by Juillard in his investigation of Turkey-red oil.

I have found also that compounds containing the HSO_4 group are entirely insoluble in petroleum ether, provided no large quantity of fatty acid is present. This last property has proved to be the most useful in purifying these compounds. At first thought it would appear that their solubility in water would furnish a most simple means of separating them from the quite insoluble fatty acids; but it was found that this aqueous solution was a very good solvent for most fatty matters, and, besides, had a strong tendency to form emulsions with the ether or other medium with which it was shaken, thus rendering the separation very tedious. When petroleum ether is used the sulphuric acid compounds need not be dissolved in water, but can be extracted dry, or floating on an acid solution in which they are insoluble.

With the aid of petroleum ether it was attempted to separate from the products of the action of sulphuric acid on a fatty acid mixture that part unacted on. If the combination with the unsaturated acids were complete, this unacted on part would be the saturated stearic and palmitic acids, at least in the case of the more common fats, and we would then have a simple method for the separation of these from oleic and other liquid acids.

To determine the completeness of the reaction, experiments were made with oleic acid. No attempt was made to obtain this absolutely pure, but the commercial product, which had been pressed at a low temperature, was made use of. It had a cold test of 12° C., and could contain but little solid fatty acid. On treating this oleic acid with about twice its weight of 94·7 per cent. of sulphuric acid, adding the acid slowly to avoid rise of temperature, a clear solution was formed. To this was then added an equal volume of water, still keeping the temperature as low as possible with ice water. The whole was then shaken with petroleum ether, which was decanted and evaporated, leaving a residue which, in two experiments, was found to be 49·51 and 42·31 per cent. of the oleic acid taken. This was liquid at the temperature of the room, and slightly more viscous than the original oil. Its mean combining weight, as found by titration with caustic soda, was 328. It was found to contain no sulphur. The oily layer insoluble in petroleum ether was identified as stearo-sulphuric acid, and but little over one-half the oleic acid had been converted to this compound.

As the petroleum ether extract of the last experiment was not simply oleic acid, but gave unmistakable evidence of having been acted on by the sulphuric acid, it seemed possible that the reaction between oleic and sulphuric acids, in the first place, was complete, but that, on adding water, a part of the H_2SO_4 compound was instantly decomposed, yielding the substance soluble in petroleum ether. To decide this point, an attempt was made to extract with petroleum ether the sulphuric acid solution of a fatty acid without adding water; but in no case could anything be removed from the solution, even when a fatty acid was used which was known to contain saturated compounds. It appeared that the solubility of all fatty acids in concentrated sulphuric acid was so great that they could not be extracted from it by petroleum ether.

The following experiment is conclusive:—One $\frac{1}{2}$ grm. of a sample of fatty acid, containing about 40 per cent. of stearic and palmitic acids, was dissolved in 50 c.c. of petroleum ether, and thoroughly shaken with 4 c.c. of 95 per cent. sulphuric acid. It was found that all but 0·02 grm. of the fatty acid had been removed from the ether by this one treatment with so small a quantity of sulphuric acid.

The simplest way to diminish the solvent action of sulphuric acid on the solid fatty acids would be to slightly dilute the former, and if at the same time it remained of sufficient strength to act on the oleic acid, the separation could be undertaken with more hope of success. A number of preliminary experiments led to the conclusion that sulphuric acid of 85 per cent. acted on oleic acid quite as completely as the stronger acid, while stearic and palmitic acids could be readily shaken out of this solution with petroleum ether. Moreover, the rise in temperature was much less, and the mixture could be made without any particular caution.

I now repeated the last experiment with oleic acid, using, however, 85 per cent. of sulphuric acid instead of the more concentrated. A description in detail of this experiment will perhaps best bring out its object and the conclusions to which I arrived.

In a glass-stoppered Erlenmeyer flask of about 300 c.c. capacity was weighed 2·0785 grms. of oleic acid. This was spread over the bottom of the flask and chilled by dipping the flask in ice water. To this was then added 4 c.c. of 85 per cent. sulphuric acid, and the flask allowed to warm in the air. As soon as the oil began to melt the sulphuric acid acted and a clear solution was formed, though without any appreciable disengagement of heat. 50 c.c. of petroleum ether was now poured into the flask, which was well closed, and violently shaken for a few minutes, allowed to settle, and the ether decanted. The contents of the flask were again shaken with 50 c.c. more of petroleum ether. The two extracts were then each transferred to a separatory funnel washed with water to remove any sulphuric acid which they might contain, and separately evaporated. The following residues were obtained:—

1st extract	0·0356 grm.	= 1·71	per cent. of the oil.
2nd „	0·0530 „	= 2·55	„ „

The first extract had a melting point of 37° C., and was therefore not pure stearic and palmitic acid, though it had the crystalline appearance of a mixture of these acids. The second extract was liquid at the temperature of the room, and could contain but little solid fatty acid. Continuing the treatment with petroleum ether, there was always a small quantity of oily substance taken up from the sulphuric acid solution. A more careful examination of this extract made it appear that the petroleum ether had taken up a small quantity of the stearo-sulphuric acid, which on treatment with water was partially dissolved out, while a part decomposed, which we have seen always happened to this compound on adding water. This part went into the petroleum ether solution and gave these small residues.

It is therefore highly probable that sulphuric acid of 85 per cent. acts quantitatively on oleic acid to form stearo-sulphuric acid, which is sufficiently insoluble in petroleum ether to allow of an analytical separation from those fatty acids on which sulphuric acid has no effect.

It now remained to apply this method to fatty acids containing considerable quantities of saturated compounds. Before giving figures I will briefly describe the manner of manipulation which I have adopted for the present:—From $\frac{1}{2}$ to 1 grm. of the fatty acid to be analysed is weighed in a glass-stoppered Erlenmeyer flask, melted, and by moving the flask, chilled in as thin a layer over the bottom as possible; this to facilitate the action of the sulphuric acid by exposing a large surface. On the layer is now poured about 3 c.c. of 85 per cent. sulphuric acid. There is usually no combination until the flask has been slightly warmed. At a certain temperature the combination takes place very rapidly and forms a clear solution. When this occurs the flask is quickly cooled, 50 c.c. of petroleum ether added, the stopper inserted and the flask shaken violently for a minute or two, then allowed to settle and the petroleum ether decanted. The flask is rinsed twice, each time with

10 c.c. of petroleum ether. The washings are added to the first extract and the whole poured into a separatory funnel and washed once or twice with water. The petroleum ether is then evaporated, and the residue, consisting of the saturated fatty acids weighed. The results of the analysis of a few samples of fatty acids are given in the following table:—

Origin of the Fatty Acids.	Solidifying Point.	Amount used.	Petroleum.	Ether Extract.	Melting Point of Saturated Acids.
		Grm.	Grm.	Per Cent.	C.
Lard........	40·75	0·7845	0·3310	42·35	53·5
Cotton-seed oil	36·80	0·6150	0·2005	32·60	..
„ „	31·90	0·5875	0·1405	23·91	53·0

The solidifying points of the original fatty acids are given as an imperfect criterion of what may be expected as to the percentage of solid fatty acids they contain. The saturated acids, being in very small quantity, their melting points were obtained by heating in a small tube attached to the bulb of a thermometer; the point taken was that of incipient fusion, which I have found to correspond quite closely to the solidifying point obtained with larger quantities. As the solidifying point of the composition of stearic and palmitic acids contained in these fats is about 55° C., the melting point shown would indicate a slight impurity.

To decide whether the extraction of the unacted on fatty acids had been complete, the sulphuric acid solution of the last sample of cotton-seed oil fatty acid was shaken a second time with 50 c.c. of petroleum ether. By this treatment 0·0190 grms. were obtained, which, however, did not solidify at the temperature of the room, and, from the former experiment, I concluded to be the decomposition product of a small quantity of stearo-sulphuric acid taken up by the petroleum ether.

The figures of the table lead to another conclusion, namely, that linolic acid is acted on quite as completely as oleic acid to form a compound insoluble in petroleum ether. This is proved by the completeness of the reaction with cotton-seed fatty acids which, if we can trust the iodine absorption test, must contain from 40 to 50 per cent. of linoleic acid. Beyond the insolubility in petroleum ether, I have as yet investigated none of the properties of the compound of linoleic acid with sulphuric acid.

A few words as to the petroleum ether employed: the commercial product even after redistillation contains compounds which, when brought in contact with concentrated sulphuric acid, form products not volatile on the water-bath. When acid of 85 per cent. is used, there is not so much action, but it will be well always to test the petroleum ether by shaking with 85 per cent. sulphuric acid, separating and evaporating on the water-bath. If there is a residue, the ether should be purified by digesting for an hour at 100° C., with about one-half its weight of concentrated sulphuric acid, then washing and redistilling on the water-bath.

The method described will, I believe, be found useful in the analysis of fats, both in the examination of the natural bodies, and as an assay of raw materials and intermediate products in the manufacture of commercial stearic acid. That the saturated fatty acids obtained by this method are not absolutely pure, cannot be denied, but this impurity is of such a nature that it is quite possible a means may be found for its accurate separation or estimation.

If this can be accomplished, we shall have a strictly accurate method for separating two groups of fatty acids, something much needed in the development of the chemistry of the fats.

Journal and Patent* Literature.

I.—PLANT, APPARATUS, AND MACHINERY.

Liquid Ammonia Cylinder, Cause of the Explosion of a. H. Lange. Woch. für Brau. 1897, **14**, [15], 584—585.

In the course of the investigation on the explosion of a cylinder of liquid ammonia used in connection with an ice-making machine, it was found that the cylinder had been placed in a vessel of water heated by steam, and that the valve had been left closed, thus causing a high pressure of gas and expansion of the liquid. All such cylinders in Germany are tested up to a pressure of 100 atmospheres, and must not contain more than 1 kilo. of ammonia in a space of 1·86 litre.

With the object of avoiding the danger of warming the cylinders, the author advocates the introduction of the ammonia as liquid instead of gas into the freezing apparatus.—C. A. M.

Painted Ironwork, Rusting of. E. Simon. Dingler's Polyt. J. 1897, **305**, 285.

See under XIII. A., *page* 1023.

PATENTS.

Lixiviating, Steaming, and Aërating, Impts., in. J. Hargreaves, Farnworth-in-Widnes, Lancashire. Eng. Pat. 23,263, Oct. 20, 1896.

Liquid, vapour, or gas is passed downwards through a series of vessels containing the materials to be treated, each newly-charged vessel being introduced at the bottom or lower part of the series, then gradually raised as each similarly charged vessel is introduced beneath, and ultimately removed when it arrives at the top of the series. —R. A.

Drying Granular and other Material, Impts. in Apparatus for. C. G. J. Moeller, Berlin. Eng. Pat. 25,612, Nov. 13, 1896.

The furnace or other gases employed in the drying process, after they leave the material which is being dried, are conducted back by means of a fan, through a return channel or flue, and are mixed with fresh combustion gases as they issue from the furnace. By this method, the temperature of the

* Any of these specifications may be obtained by post by remitting 8*d*.—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

gases arising from the fire is reduced, whilst avoiding loss of heat, and only as much air is conducted through the furnace as is requisite for the advantageous combustion of the fuel. Several forms of the apparatus are described in the specification, some of which comprise a series of heating registers, which are heated by the warm gases issuing from the drying apparatus, and are saturated with the condensed steam, these registers again yielding up their heat to the fresh air entering the apparatus. The registers thus serve the purposes of utilising the free and latent heat of the exhaust gases, and of collecting the condensation water.
—R. A.

Filtering, Washing, and Drying Crystalline, Granular, and other Materials; Impts. in the Method of and Apparatus for. S. Sherratt and W. A. Richards, both of Northwich, Cheshire. Eng. Pat. 28,289, Dec. 10, 1896.

The material to be treated is delivered on to a rotating filter bed in the apparatus, with or without the addition of water or other washing fluid, and the liquid is drawn through the material on the filter by means of a vacuum apparatus beneath. When it is necessary to subsequently dry the material, air or gas, heated or otherwise, is drawn through the material in the same manner. The latter is removed from the filter bed by a number of curved scrapers, which can be lowered to, or raised from, their operative position either automatically or by hand.—R. A.

Sand, Cement, and other Similar Substances; Impts. in Apparatus for Measuring and Mixing. H. Alexander, Water Lane, Leeds. Eng. Pat. 13,505, June 1, 1897.

The apparatus comprises a compound hopper, having separate divisions for receiving the different materials to be mixed, in combination with a compound drum, having separate sets of adjustable compartments for measuring the materials and delivering them into a mixing trough below. In the trough, the materials are mixed by two sets of oppositely-rotated mixing blades.—R. A.

Preserving Organic Bodies, Impts. in Apparatus for. L. A. M. D. de Belfort, Paris. Eng. Pat. 14,907, June 19, 1897.

The apparatus consists of a number of steel cylinders lined with lead, capable of withstanding a pressure of several atmospheres. These are connected by pipes with each other, and also with a carbon dioxide generator. The materials to be preserved are placed upon shelves in the cylinders, which are then charged with carbon dioxide under a pressure of 5 to 10 atmospheres. After about 15 minutes, the carbon dioxide is displaced by pumping in water containing 0·25 per cent. of formalin in solution. After about 30 minutes the solution is run off, and the articles are removed from the cylinder. Polymerisation of the formalin is prevented by the addition of from 25 to 30 per cent. of common salt or other normal chloride, and when preservation for several months is desired, the liquid, whilst in the cylinder, is subjected to electrolysis, to increase its antiseptic properties.—L. A.

II.—FUEL, GAS, AND LIGHT.

Flame, Non-Transparency of Glowing Carbon in. N. Teclu. J. prakt. Chem. 1897, **56**, [15, 16], 178—180.

Hirn (Ann. Chim. phys. **30**, 319), not being able to prove optically the presence of finely-divided carbon in the luminous gas flame, concluded that at a high temperature, the solid particles become transparent. To determine this point the author made experiments with different flames. The ordinary gas flame, Bunsen flame, and flames of a candle and petroleum lamp, together with the glowing carbon thread of an incandescent lamp were illuminated by electric light, their shadows projected on a white screen and photographed. In the photographs (reproduced in the paper) there was no perceptible difference between the shadows cast by the luminous and non-luminous gas flames, and the same was the case in the shadows cast by sunlight. In the shadow of the candle flame a darkening was faintly visible, and markedly so in the case of the petroleum flame. The shadow cast by the glowing carbon thread was scarcely visible, but became plainly visible in oblique light.

Experiments were made to determine whether there was any difference in the transparency of the gases likely to be present. The shadows cast by layers (of a metre) of carbon monoxide, carbon dioxide, coal gas, acetylene, and air sealed in glass tubes showed that the gases differed but little in this respect.

Considering that the difficulty of optically recognising suspended particles in the gas flame was due to their extraordinarily fine state of division, the author experimented with a thicker layer of burning gas. Twelve batswing flames, 3 cm. broad, were placed one behind the other and illuminated as before with the electric light. The shadow cast on the screen showed very plainly a dark zone, which, in the author's opinion, could only be due to the presence of solid particles. Hence, he considered that Hirn's conclusion was due to his method not being sufficiently sensitive, and that therefore the theory of the transparency of carbon in glowing condition was not justified by the facts.—C. A. M.

Coal-Gas, Action of Sulphuric Acid on. P. Fritzsche. J. f. prakt. Chem. 1897, **56**, 258—265.

Brown substances, ranging in consistency from resin-like solids to limpid oils, are formed when coal-gas is washed with concentrated sulphuric acid. Berthelot (Comptes Rend. **82**, 871) obtained 25 grms. of such condensation products from 100 cb. m. of Paris gas, and thence isolated six bodies of an aromatic nature, apparently formed by condensation of the molecules of methyl-, dimethyl-, and propyl-acetylene. The acid would therefore appear to exert only a contact action on the hydrocarbon, but the author found the volume of the products scarcely diminished when substituted acetylenes were removed from the gas by means of ammoniacal cuprous chloride prior to the washing with acid. Therefore Berthelot's explanation can only apply to a small part of the condensation products. Moreover, the quantity and nature of the products vary with different samples of gas. The author obtained from 5—15 grms. per cb. m. of gas. A portion was dissolved by water, forming a green solution; and, after washing the remainder with solution of potash, about three-fifths of the whole was susceptible of distillation, and passed over steadily between 100°—300° C. Several kilos. were prepared by treating coke-oven gas—freed from benzol and sulphuretted hydrogen—with sulphuric acid under five atmospheres pressure. The brown oil which remained after some crystals had deposited, was washed with slight excess of caustic soda, and the saline solution was separated from oil and concentrated. On cooling, white scales of silky lustre were detected in the dark brown pulp, and by repeated extraction and crystallisation this white salt was separated. From it other salts were prepared. The chief characteristics of the salts are as follows:—

The copper salt, $(C_{15}H_{25}SO_4)_2Cu + 6H_2O$, prepared from the sodium salt and copper sulphate, is pale blue, but, when dehydrated, pale green. The salts of the alkalis lather freely, and dissolve phenols to clear non-alkaline solutions. The ammonium salt, $C_{15}H_{25}SO_4NH_4 + 2H_2O$, is in needles of silky lustre, readily soluble. The sodium salt, $C_{15}H_{25}SO_4Na + 6H_2O$, forms efflorescent plates, less soluble than the ammonium salt, and fuses only with decomposition. The alkaline earths give salts with difficulty soluble in cold, more readily in hot water. The ferrous salt, $(C_{15}H_{25}SO_4)_2Fe + 7H_2O$, is white—yellowish in a moist atmosphere. This salt separated in crystals from the original condensation product, the iron having been derived from the containing vessel.

The acid was prepared by passing sulphuretted hydrogen through water containing the copper salt in suspension, and removing the copper sulphide. It formed white crystals, readily soluble, and was very stable, being unattacked by hot concentrated sulphuric acid, and liberating hydrochloric acid from a hot solution of common salt. In this respect it resembles the sulphonic acids derived from the camphors, and it appears to be the sulphonic acid of a body having the formula $C_{15}H_{26}O$, which is also that of cedar-camphor.

Other acids of a similar nature appear to be present in the condensation products of gas. The mother-liquor of the above sodium salt contains at least one other acid, probably several acids, of which the salts are more freely soluble. On distilling the clear brown oil remaining after washing the condensation product with alkali and water, a crystalline white body was obtained at 200°—220°, but was not examined. The distillate oils boiling below 200° were limpid, and left a grease spot on paper; those boiling between 200° and 300° resembled mineral oils. The hydrocarbon residue in the retort cooled to a brown resin. Clearly, the products of the treatment of coal-gas with sulphuric acid are by no means so simple as Berthelot supposed.—J. A. B.

Gas Lamps, Incandescent; Danger of Fire from. Dingler's polyt. J. **306**, [4], 96.

A notification has recently been issued by the Chief of Police in Berlin, to the effect that, in consequence of the frequency of fires due to the ignition of wooden beams in buildings by the flame of incandescent gas lamps, situated too close to the ceilings or beams, such lamps—which give out a greater amount of heat than is, apparently, generally supposed—must in future be placed at least 40 ins. from the ceiling, or else fitted with a (sufficiently large) shield to protect the woodwork. Such shield must, however, not be affixed to the ceiling, but either attached to the lamp or else suspended from an adjoining brick wall.—C. S.

Cerium. O. Boudouard. Comptes Rend. 1897, [20], **125**, 772.

See under XX., *page* 1035.

Calcium Carbide and Acetylene, Analysis of Commercial; and the Purification of the Latter. G. Lunge and E. Cedercreutz. Zeits. angew. Chem. 1897, [20], 651.

See under XXIII., *page* 1046.

PATENTS.

Fire-Damp in the Atmosphere of Mines and other Places [Gas Battery], Improved Method of and Apparatus for Detecting and Indicating the Presence of. E. L. Mayer, London. Eng. Pat. 23,822, Oct. 26, 1896.

See under XXIII., *page* 1040.

Calcium Carbide, Manufacturing, and for the Reduction of Metals from their Oxides; An Improved Method and Apparatus more especially intended for. H. Maxim, London. Eng. Pat. 25,611, Nov. 13, 1896.

See under XI. B., *page* 1022.

Peat-Charcoal, Impts. relating to the Production of, and to Apparatus therefor. E. T. Zohrab. Eng. Pat. 26,191, Nov. 19, 1896.

Relates to the preparation of weathered peat charcoal, especially useful in blast and other furnaces in the production of iron, steel, or other metals. There are three steps in the process, and three sets of apparatus.

1. *Pulping.*—The peat is passed into a vertical perforated inverted hollow cone, having a tapered Archimedean screw, whereby the peat is forced through the perforations to a lower vertical cylinder, where it is subjected to the action of rotating and fixed knives, and forced through lower openings into horizontal tubes, also provided with Archimedean screws and rotating knives, which force the pulp through perforated end plates, to suitable moulds for forming blocks.

2. *Drying.*—The blocks are fed to the top member of a series of endless bands, arranged in a drying oven, so that the blocks are continually and slowly moved forwards from one end to the other of the top band, then, falling to the next lower band, travel backwards, and so on to the lowest band.

3. *Charring.*—The dried blocks are filled into a box on wheels, and run into a kind of muffle furnace. The gaseous products are collected in a hydraulic main.—R. S.

Enamelling Process, Impts. in, for Electric Heating Apparatus. [Insulation.] R. E. B. Crompton and E. J. Fox, London. Eng. Pat. 26,267, Nov. 20, 1896.

To prevent the resistance wire coming into contact with the metal plate in electric heating apparatus manufactured according to Eng. Pat. 17,091, 1892 (R. E. B. Crompton), the plate is first given a thin coating of enamel, and while this coat is still in a plastic condition, it is covered with a layer of granulated porcelain, or similar infusible and non-conducting substance, which is again covered with a layer of enamel, and the whole fused together. The resistance wire is then attached as described in the previous patent. When very great insulation is required at very high temperatures, the layer of granulated material is replaced by a thin sheet of porcelain, or similar substance, which may be either continuous or moulded into hexagons or other convenient forms, which can be laid closely side by side, and is then covered with a layer of enamel.—G. H. R.

Electric Furnaces, Impts. in. [Fixed Arc.] W. S. Horry, Michigan, U.S.A. Eng. Pat. 22,521, Oct. 1, 1897.

The furnace consists of a bottomless hopper of fire-resisting material, supporting standards so as to hang over the space between the carbons in which the arc is struck, and between the flanges of a rotary spool or reel, to which movable cover plates can be attached. The carbons rest on the fire-proof material of the hopper, which screens their electrical connections from the heat of the furnace, and their edges are bevelled so that the space between them is vertical. The length of the arc is constant, and not variable, as in previous furnaces. A mixture of the coke and lime is fed into the hopper and converted into calcium carbide in the arc, and when the ampèremetre rises, the spool is rotated to remove the product from the furnace, the action of which is thus continuous, fresh material being fed into the hopper as that converted into calcium carbide is removed in a plastic sheet.—G. H. R.

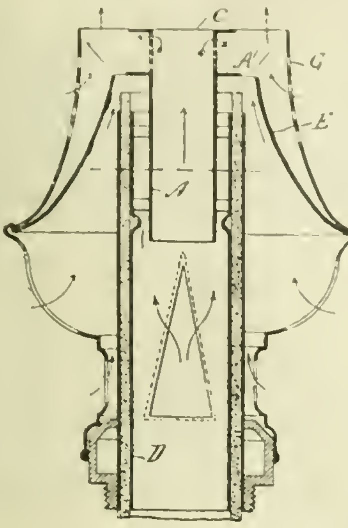
Heating or Evaporating by means of Hydrocarbon Liquids of any Density, Impts. in Apparatus for. P. J. E. E. Chambost, Paris. Eng. Pat. 29,000, Dec. 17, 1896.

The hydrocarbon liquids are passed through an externally heated horizontal retort or cylinder, provided with a central shaft on which are a number of partitions. Within this retort the hydrocarbon is vaporised, the coke being deposited in the retort, which is purposely arranged so as to be easily taken apart for cleaning. Preferably, two retorts are used, so that whilst one is in use the other is being cleaned. The vaporising retorts are enclosed within one or more refractory and non-conducting jackets, to prevent loss of heat. Part of the vapours may be used to heat the retorts, the rest being employed for the special heating device for which it is required. The burners used for these vapours consist of a valve-box, in the bottom of which are one or more holes for the escape of the vapours. A perforated thin screwed cap preferably forms the bottom. The regulating plug has one bearing in this screwed cap, the other in a stuffing-box.—R. S.

Oil Burners applicable for Incandescent Lighting, Impts. in. W. P. Thomson, London. From The Continental Gas Glühlicht Actien Gesellschaft "Meteor," vormals Kroll, Berger, and Co., Berlin. Eng. Pat. 28,853, Dec. 16, 1896.

The patentee claims an oil-lamp burner, which produces a blue flame suitable for incandescence lighting, as the result of four distinct air currents, *viz.*, a sluggish current rising inside the burner tube and passing to the outside over the edge of the circular wick, to develop therefrom combustible gas; a second current rising outside and past the outer edge of the wick, to carry the gas upwards; a third current of comparatively large volume rising centrally and being deflected to enter the flame at the point where it burns; and, finally, a fourth current rising outside of the second current and commingling with it, to change the direction of the combined other currents. The form of lamp preferred is of the Argand pattern, within the central air tube D of

which is fixed an inner tube A of smaller diameter, having a flange A′ projecting over the wick, and a head with upper cover C and perforated sides for the escape of the central



air draught. Only a relatively narrow annular space is left between the tubes A and D, so that only a sluggish current passes outwards over the wick. A strong current passes between a cone E and the wick tube, and through the perforations in an outer cone G, there passes the above-mentioned fourth current from the base of the chimney.
—H. B.

Incandescent Lighting or Heating Purposes, An Improved Vaporiser for Liquid Combustibles for. F. V. C. Brokk, Berlin. Eng. Pat. 28,290, Dec. 10, 1896.

The vaporiser claimed, consists of an annular retort containing a wick, from which hydrocarbon vapours are distilled off by a small central heating flame, the vapours mixing with air and burning beneath a mantle as usual. The waste gases of the small flame escape by a pipe which rises centrally through the vapour nozzle, the burner-tube, and the mantle, and as it is always hot throughout its length, it prevents the condensation of the vapours surrounding it. The annular retort is perforated with one or two passages, to admit air to the heating flame and to serve for igniting the flame, the passages being opened or closed by means of a revolvable ring.—H. B.

Incandescent Burners, Impts. in. A. Bandsept, Brussels. Eng. Pat. 29,597, Dec. 23, 1896.

In the improved burner a central gas jet issues from a nozzle, the interior of which becomes gradually constricted. A little below its orifice the nozzle is surrounded by an annular gas passage or by a number of annularly arranged gas jets. The gas is directed upwards into a truncated conical chamber, at the base of which air is drawn in and mingles with the gas. Issuing upwards from this chamber the gas ascends a gradually widening tube, drawing a second supply of air from apertures at its base. The top of the expanding tube, forming the mouth of the burner, is covered with wire gauze, from the centre of which depends an inverted deflecting cone. The air-inlets may be regulated by a ring-valve, the expanding tube may be adjusted as to height by a telescopic arrangement, and to more effectively mix the gas and air, the deflecting cone may be lengthened and fitted with helical blades.—H. B.

Incandescent Oil Lamps, Impts. in and connected with. M. Graetz, Berlin. Eng. Pat. 29,697, Dec. 24. 1896.

"In a petroleum incandescence lamp . . . . the combination is claimed with a duplex burner and with a device for raising the upper part of the said burner and fixing it in its raised position, of a means for detachably securing the upper part to the elevating device; of a flame-disc situated in the plane of the upper opening of the cap of the upper part of the burner, and a cylinder supported by said part, and having at least twice the diameter of the wick tube"; also of the means for holding the mantle above the flame.—H. B.

Incandescent Oil Vapour Burners, Impts. in. M. Bernstein and M. Ehrenbacher, Berlin. Eng. Pat. 21,913, Sept. 21, 1897.

Within a circular wick is arranged a central shaft, up which a current of air passes, the air being heated by passing through a concentric radially disposed system of thin metal plates. The primary flame, burning at the top of the wick, is divided into "a number of superheated, heating and illuminating flames," by feeding the combustible mixtures through a perforated flame distributor which is surmounted by a perforated hollow cap, in which part of the mixture is superheated. The superheated mixture blow outwards against the exterior flame circle, meeting with a supply of air which has been previously heated by passing through a perforated hollow metal casing. The burner is surmounted by a mantle as usual.—H. B.

Carburetted Air for Incandescent Gas Light, Process and Apparatus for the Production of. O. Kahns, Hamburg. Eng. Pat. 20,571, Sept. 7, 1897.

Carburetted air for incandescent gas lighting is produced by leading a current of air through a closed tank in which are suspended partitions of wick-like, absorbent fabric, kept saturated with a hydrocarbon liquid. This hydrocarbon liquid may be a cheap product of the process of petroleum refining, having a sp. gr. of 0·71.—H. B.

Acetylene Gas, Impts. in the Production of, and in Apparatus therefor. L. N. V. Bablon, Paris. Eng. Pat. 25,224, Nov. 10, 1896.

A distributing screw at the base of a receptacle for ground carbide is rotated by a gas meter through which passes the gas flowing from the holder to the burners, and the carbide falls into water and produces acetylene at a rate determined by the speed of rotation of the screw. The rise and fall of the holder may control a variable speed gear or a friction clutch device between the meter and the distributor. Or the meter may directly operate the distributor, and the holder control an outlet valve on the carbide receptacle, or a valve on a by-pass main, which permits gas in case of over-production to slip the meter.
—J. A. B.

Acetylene Gas, Impts. in Apparatus for Generating. J. Wetter, London. From G. Meyer, Zürich. Eng. Pat. 27,212, Nov. 30, 1896.

Water is discharged from a chamber into a generating vessel containing carbide, when the gasholder descends, and, by means of lugs, releases a weighted lever which opens the discharge valve. The water at each discharge barely suffices to generate sufficient acetylene to fill the holder. The water chamber is refilled automatically, and is provided with an overflow pipe.—J. A. B.

Acetylene Gas, Impts. in or relating to means for Regulating the Production of, and in Lamps therefor. E. Gossart and H. Chevallier, both of Bordeaux, Gironde, France. Eng. Pat. 27,574, Dec. 3, 1896. (Under Internat. Convention.)

An auto-regulating lamp is formed of a base containing a basket in which is calcium carbide, on which drops from one or more capillary tubes—bent to increase their length in a small compass—water from a cistern forming the cover.—J. A. B.

Acetylene Gas, Apparatus for Producing. E. Richard-Lagerie, Roubaix, France. Eng. Pat. 29,168, Dec. 19, 1896.

In the crown of a bell gasometer is arranged a bell-shaped receiver submerged in water in an annular tank fixed to the gasometer crown. A pipe with cocks places the bells in communication. A carbide basket covered by an inner

bell-shaped receiver acting as a float, is fixed by bolts in the outer bell-shaped receiver. The water in the gasometer tank serves for the reaction, which ceases when the pressure in either bell increases and lifts the carbide above the water.—J. A. B.

Acetylene Gas, An Improved Apparatus for the Production, Drying, Purifying, and Storage of. W. H. Dargue, Newcastle-on-Tyne. Eng. Pat. 29,768, Dec. 28, 1896.

The generator is cylindrical and is water-jacketed, or within the water tank of a bell gasholder, whence, or from an independent cistern, water is supplied to the carbide under the control of a valve operated by the movements of the bell. Suitable connections and taps for relief generators are described.—J. A. B.

Acetylene Gas, Apparatus for Producing. G. Lebrun and F. Cornaille, Paris. Eng. Pat. 512, Jan. 7, 1897. (Under Internat. Convention.)

Water from a closed reservoir falls on to carbide in a wire-gauze cylinder, which is mechanically rotated above a tray for the reception of the residual lime. The flow of water is determined by the pressure of gas admitted to the top of the reservoir by a pipe, on which is a valve controlled by the movements of the gasholder into which the acetylene passes.—J. A. B.

Acetylene Gas, Impts. in Apparatus for the Production of. W. P. Thompson, Liverpool. From P. P. H. Macé and L. L. H. Gérard, both of Paris. Eng. Pat. 3013, Feb. 4, 1897.

In the crown of the weighted and balanced bell of a gasholder is fixed a carbide receptacle, provided with a discharge opening of a doubly tapering form. Two cones on a weighted and guided rod act, one above, the other below, this opening, and open and close it in concurrence with the falling and rising of the bell, which arrest and release the rod. Pulverulent carbide falls through the opening into the water in the gasholder tank. Two methods of arresting the movements for recharging with carbide are described.
—J. A. B.

Acetylene, Impts. in Apparatus for the Production of. E. Jimeno, Barcelona, Spain. Eng. Pat. 14,090, June 9, 1897.

A water vessel is placed above a vessel about double its capacity containing carbide. A capillary communication permits water to flow to the carbide and generate acetylene. Various methods of regulating the pressure on the escape orifice, of automatically closing the communication, and of governing the pressure at the burners, are claimed.
—J. A. B.

Acetylene Gas, Improved Apparatus for the Production and Storage of. R. F. Carter, Niagara Falls, Canada. Eng. Pat. 17,148, July 24, 1897.

Water is supplied through a ball-cock valve to a large cistern pivoted within a larger tank, in such a manner that the inner cistern tips automatically when the gasholder falls, and discharges a limited volume of water into the outer tank, whence it flows on to carbide in a generator; and the acetylene evolved, raises the gasholder and restores the inner cistern to its original position. Twenty claims are made.—J. A. B.

Acetylene Gas, Impts. in Apparatus for the Production of. A. Rieffel, Paris. Eng. Pat. 17,938, July 30, 1897.

The bell of a gasholder is, by one or other of several specified devices, increased in weight as it rises, and thereby the pressure of gas in the generator varies with the rise and fall of the bell. Water is supplied to the generator under a constant head, which is such that it flows thereto when the pressure therein is low and ceases to flow when it is high. Thus the generation of gas only occurs when the gasholder is nearly empty. Several modifications of various parts are claimed.—J. A. B.

Acetylene Gas, Improved Apparatus for the Production and Storage of. H. J. Bell and Niagara Falls Acetylene Gas Machine Company, Ltd., both of Niagara Falls, Canada. Eng. Pat. 19,411, Aug. 23, 1897.

Attached to the side of the bell of a gasholder by a rood is a float which sinks into a water chamber when the bell descends, and expels water from the chamber into a spout surrounded by a sleeve which may be tilted, and is thus caused automatically to discharge its contents into a tip tank, which empties itself into a tank, whence a pipe conveys the water to carbide in a generator. The water chamber is refilled automatically when the bell rises. The generator used is that protected under Eng. Pat. 2284 of 1897 (this Journal, 1897, 431). Fifteen claims are made.—J. A. B.

Acetylene Gas, An Improved Device for Producing and Burning. J. Windmüller, Cologne. Eng. Pat. 21,464, Sept. 18, 1897.

The lower part of a large tube is partially occupied by a smaller tube, which leaves an annular space. This space and the upper part of the apparatus are filled with water, which percolates through a hole near the base to meet the carbide compound in the inner tube, and develops gas, which passes through a hollow rubber ball for regulating the gas pressure, to the lamp, in which an air chamber is provided between the casing and the disc, the burner being so adjusted that it only partially reaches or dips into the casing.—J. A. B.

Acetylene Gas, Impts. relating to the Production of, and to Lamps for Generating and Burning the same. H. H. Lake, London. From Wizard Manufacturing Company, Chicago, U.S.A. Eng. Pat. 21,831, Sept. 23, 1897.

Refers particularly to lamps for bicycles, &c. A stick of carbide is pressed against an absorbent pad, to the under side of which water is admitted from the surrounding water receptacle through a passage, of which the size is fixed by a stem valve. The gas passes through a similar passage to a lava burner tip provided with an air passage. The burner is in a suitable combustion chamber.—J. A. B.

Blow Torch Burners, Impts. in. J. C. Dupee, Chicago, U.S.A. Eng. Pat. 18,830, Aug. 14, 1897.

These burners, intended for the use of plumbers, braziers, &c., are designed for burning hydrocarbon oils of low specific gravity, as kerosene or crude petroleum. Each burner has an oil reservoir, and an air pump for producing pressure in the reservoir, whereby the oil is forced upwards to an annular retort, wherein it is vaporised, and from which the vapour passes to a nozzle having a needle valve. From the nozzle it passes to the cavity within the retort, where it mixes freely with air, and burns in a combustion chamber fixed thereon. This combustion chamber is arranged so as to cause eddies, by forming it of a pear shape, having a restricted opening at its outer end, an enlargement in the body thereof, and a series of openings in its walls. By this arrangement a portion of the main flame is reversed and forced back through the openings on to the retort, the main flame passing upwards or outwards to heat the brazier's or plumber's pot.—R. S.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Petroleum, Origin of: Spontaneous Polymerisation of Hydrocarbons. C. Engler. Ber. **30**, 2358—2365.

Referring to Sadtler's preparation of hydrocarbon gases from vegetable oils (this Journal, 1897, 727), the author points out that similar results have been previously obtained by him (Ber. **21**, 1816; **22**, 592) from pure stearic and oleic acids. Moreover, that in attributing an animal origin to petroleum deposits in general, he does not imply the impossibility of other methods of formation in isolated cases.

The presence of heavy lubricating oils in natural petroleum has been considered to militate against the theory of animal origin, because the products of the distillation of fat

under pressure consist largely of light ethylenes. However, recent experiments made by the author or under his direction, tend to show that a spontaneous polymerisation of the hydrocarbons occurs in petroleum distillate, coal-tar distillate, &c., which probably explains the development of the heavy oils from the "protopetroleum" first resulting from the distillation of animal matter, &c.

For example, a distillate from a high-boiling Java petroleum was found to have a sp. gr. averaging 0·8799, which, however increased on standing for some time, to 0·8917. In a second sample from Java, the sp. gr. rose in a few days from 0·8912 to 0·9008. That other petroleums also exhibit the same tendency, though in different degree, was shown by a distillate (b. pt. 300°—320° C.) from Galician residuum, the sp. gr. increasing in one case from 0·8601 to 0·8612, and in another—after heating the residuum for three days under a reflux condenser and then distilling at 320° C.—from 0·8334 to 0·8349 (in 20 days).

A similar behaviour was noticed by Spilker in coal-tar distillates, crude benzene giving an increase of 0·001 in 20 days, and the so-called light oil an increase of 0·00173 in eight weeks. Also in brown-coal-tar distillates an analogous behaviour was discovered by W. Eisenlohr, though here the increase in sp. gr. during the period occupied by the experiments (30 days) was very slight. On the other hand, samples of the fish-oil distillate prepared for the author by Krey, in 1888, and which had an initial sp. gr. of 0·8105, now exhibit, after nine years' storage, densities varying from 0·8221 to 0·8295; and an increase of 0·002 was found to ensue in 30 days in the case of a sample newly distilled from fish oil.

From these samples the author considers it permissible to suppose that the heavy oils in petroleum have gradually been elaborated by spontaneous polymerisation, from the lighter hydrocarbons first formed by distillation; such polymerisation being probably feasible, though at an exceedingly slow rate, at low temperatures. It may be that the change in question is in turn succeeded by intramolecular transpositions. The prevalent temperature and pressure would influence the character of the resulting products, and determine the preponderance of saturated, unsaturated, open-chain, or cyclic hydrocarbons, and may thus account for the differences in composition exhibited by natural petroleums.—C. S.

Petroleum, Japanese. Eng. and Mining J. 1897, **64**, [11], 394.

The oil districts of Japan extend from Hokkaido to Akita in the north, traverse the provinces of Echigo and Shinano, and reach the Totomi Province. Previous to the year 1890, wells were sunk in an unscientific manner to a depth of about 600 ft., but since that time American boring machinery has been introduced, and oil is now being obtained from a depth of 800 to 2,000 ft. The method of refining has also been improved to such an extent that Japanese petroleum is at present equal in quality to foreign petroleum.—A. S.

Petroleum in Texas. Eng. and Mining J. 1897, **64**, [14], 398.

There has been discovered at Corsicana, Texas, at a depth of 1,040 ft., a seam of bluish sandy shale, about 20 ft. thick, saturated with petroleum. The ground above and below this stratum consists of a hard clay. The oil was found, on analysis, to contain about 90 per cent. of volatile matter. In the following table, the Texas petroleum is compared with the oils from other districts:—

Crude oil from	Specific Gravity at 17° C.	Began to boil at	Came over below 150° C.	Between 150° and 300° C.	Above 305° C.
		° C.	Per Cent.	Per Cent.	Per Cent.
Texas-Corsicana	0·821	80	34·6	40	15·8
Pennsylvania.	0·818	82	21·0	38	40.7
Galicia........	0·824	90	26·5	47	26.5
Baku.........	0·859	91	23·0	38	39·0
Alsace........	0·907	135	3·0	50	47·0
Hanover......	0·899	170	..	32	68·0

—A. S.

Petroleum; Is the Occurrence of Nitrogen in, an Unassailable Proof of Organic Origin? K. W. Charitschkoff. Chem. Rev. Fett- u. Harz-Ind. 4, [21], 289—290.

The author—who supports (J. Russ. Phys. Chem. Ges. **29**, 151) the inorganic theory of origin—considers that the presence of nitrogen compounds in petroleum is capable of explanation without resorting to an organic theory of origin, since modern research is continually affording instances of the direct combination of nitrogen with various substances, particularly in processes of combustion occurring in presence of this element. He believes the formation of petroleum to be effected at depths not very remote from the earth's surface, and that, under such circumstances it is not impossible for the nitrogen compounds to have resulted from subsidiary reactions going on in presence of nitrogen either in a free state or dissolved in sea water. The accuracy of this view will, he believes, be confirmed as future research enlarges our knowledge of the chemistry of nitrogen.—C. S.

Petroleum, Refining, with Fuming Sulphuric Acid. Fr. Boleg. Chem. Rev. Fett- u. Harz-Ind. **4**, [19], 263.

The author states that the process patented by the Badische Anilin und Soda Fabrik (this Journal, 1897, 901) has been—in principle—employed by him during the past ten years, and consequently cannot be regarded as novel, though the apparatus is new. The same results can, however, be obtained by using an ordinary lead-lined, steam-blast agitator, but working it with compressed air and leading the acid into the air-pipe, which is fitted with an atomising jet.—C. S.

Mineral Oil, Refining with Water-Glass. J. R. Michler. Chem. Zeit. **21**, [82], 853.

As a means of preventing the formation of the troublesome emulsions occurring in the alkali treatment of heavy mineral oil distillates, the author recommends the employment of alkali silicate (water-glass), either alone or in conjunction with sodium carbonate or caustic soda, for neutralising the oil after the acid treatment. The advantages claimed for this reagent are that a smaller quantity of acid is needed, since the dark oil then obtained is bleached by the action of the precipitated silica, and that this body at the same time disperses any emulsion produced in the oil.—C. S.

Petroleum Spirit [Benzine] from Grosni Petroleum. Sulphur Compounds in. K. W. Charitschkow. Trudy bak. otd. imp. russk. techn. obschtsch. 1897, **12**, 272; through Chem. Zeit. Rep. 1897, **26**, [74], 203.

The fraction of the Grosni petroleum boiling up to 150° C. is in many external characteristics similar to Ohio petroleum, and has a disagreeable smell, which is not removed by purification with the usual reagents. The author has therefore examined the sulphur compounds present, in the belief that they gave rise to the odour in question. To determine the presence of these compounds, the benzine was treated with a 5 per cent. solution of corrosive sublimate, or with weak platinum chloride solution, after removing a trace of hydrogen sulphide by means of lead acetate. The substances obtained after the sublimate treatment reacted like mercaptans, but contained not more than 7—10 per cent. of sulphur. Further research showed that some hydrocarbons (of the acetylene series) as well as the sulphur compounds, entered into combination with the mercuric chloride. For the determination of sulphur in fractions containing but little of this substance, both Heusler's method and Engler's modification of it proved to be ill-adapted: too much of the distillate had to be burned. The author only determined the sulphur in the compounds separated by means of mercuric chloride, and then used the Heusler-Engler method to prove qualitatively that the whole of the sulphur had been removed by the mercuric salt. The sulphur found in the benzines examined, varied between 0·003 and 0·019 per cent. No regularity could be traced in the distribution of the sulphur compounds between the fractions of different boiling point; all that may be safely stated is that sulphur compounds are always present in the petroleum

spirit (sp. gr. 0·685), whilst the naphtha (ligroin) distillates (sp. gr. 0·75 to 0·77) appear to be free from sulphur.
—W. G. M.

Olefines, Naphthenes, and Benzene Hydrocarbons; Formation of, by the Distillation of Fats under Pressure. C. Engler and Th. Lehmann. Ber. **30**, [15], 2365—2368.

By treating the various fractions of fish-oil distillate by the bromination method, the purified bromides were found to correspond to hexylene bromide in the fraction 50°—60° C., whilst in the fraction 70°—80° C. they approximated to heptylene bromide. The remaining fractions were apparently mixtures of higher and lower homologous olefine bromides.

The detection of naphthenes was attempted by examining the composition of various fractions after the removal of the olefines, aromatic hydrocarbons, &c., with the result that, whilst the fractions 68·5°—69·5°, 78°—80°, 98°—99°, 123°—124°, 149·5°—150·5°, and 160°—162° C. appeared to consist mainly of paraffins, the presence of naphthenes was also indicated. The quantities of substance employed were, however, too small to permit of the isolation of these hydrocarbons.

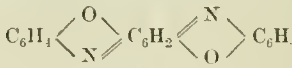
Benzene hydrocarbons were detected without difficulty by nitration, *viz.*, benzene in the fraction 76°—83° C., toluene in the fraction 106°—114°, *m*-xylene in that between 130° and 138°, and mesitylene and pseudocumene in the fraction 160°—169° C.

Pure glycerides and other fats and oils gave, on the whole, analogous results.—C. S.

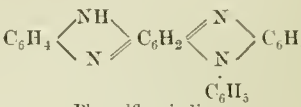
IV.—COLOURING MATTERS AND DYES.

Aposafranine, Action of Bases on. O. Fischer and C. Giesen. Ber. **30**, 2489—2494.

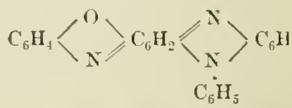
In continuation of Fischer and Hepp's work on the formation of Induline and Fluorindine from Aposafranine (Ber. **29**, 361; this Journal, 1896, 536), the authors have investigated the action of methylamine, *p*-anisidine, ethylene diamine, *o*-amidophenol, and *o*-naphthylene diamine on Aposafranine. They find that the monamines form Indulines, whilst the diamines yield Fluorindines in addition. Methylamino-aposafranine is obtained by heating together in a sealed tube to 100° C. for 5—6 hours, 4 grms. of Aposafranine hydrochloride, 2 grms. of methylamine hydrochloride, 10 c.c. of a 33 per cent. methylamine solution, and 40—50 grms. of alcohol. The solution is filtered, acidulated, evaporated, the residue extracted with water, and salted out, preferably with potassium bromide. The bromide of the induline crystallises from water or dilute alcohol in brownish needles. The solution in water is orange-red, which is turned magenta-red by concentrated hydrochloric acid. An analogous compound is formed by boiling together 5 grms. each of Aposafranine hydrochloride, *p*-anisidine, and *p*-anisidine hydrochloride in 100 grms. of alcohol. The product crystallises in greenish crystals, which dissolve to a brown solution. By the action of ethylene diamine on Aposafranine, two different substances are formed, one of which shows the reactions of an induline, whilst the other belongs to the class of the fluorindines and can be obtained by oxidation of the former. The Induline is formed by heating 5 grms. each of Aposafranine hydrochloride, ethylene diamine, and ethylene diamine hydrochloride with 80 grms. of alcohol under an inverted condenser for two hours. The solution is then made alkaline with potash and precipitated with water. The brown powder so obtained, after crystallisation from dilute alcohol, forms acicular crystals with a bluish-green reflex. The base dissolves to an orange-red non-fluorescent solution, which is turned violet by dilute mineral acids. The hydrochloride dissolves in hot water and in alcohol to a yellowish-red solution, and is a strong dyestuff. The base melts with decomposition, being partially converted into the fluorescent compound. This conversion is easily effected by oxidising agents, especially by mercuric oxide in nitrobenzene solution. The fluorindine can also be obtained directly by heating the original melt to 150°—160° C. for some hours. The shining greenish crystalline product so obtained, only dissolves with difficulty in the usual solvents; the yellow solutions in benzene, xylene, &c., have a greenish-yellow fluorescence. Two possible formulæ are given. By heating equimolecular proportions of Aposafranine hydrochloride, *o*-amidophenol hydrochloride, and *o*-amidophenol in an alcoholic solution under an inverted condenser for about 12 hours, the magenta solution gradually turns blue. The boiling solution is then treated with alcoholic ammonia and the brown base recrystallised from benzoic ethyl ether. The product forms brownish-red needles, which dissolve in alcohol to a cherry red, in benzene and toluene to yellowish-red solutions, which have a blood-red fluorescence. Apparently a product is formed which is at the same time an oxazine and an azine, and hence stands intermediate between triphendioxazine and phenylfluorindine—



Triphendioxazine.



Phenylfluorindine.

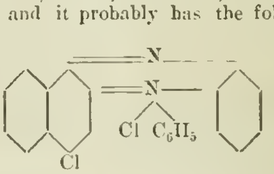


Triphenoxazine-N-phenylazine.

A similar substance is obtained by the action of *o*-naphthylene diamine hydrochloride on Aposafranine in an alcoholic solution. The substance is very sparingly soluble; the alcoholic solution is red with a blood-red fluorescence; on acidifying, the solution turns blue and the fluorescence greenish.—T. A. L.

Rosindone and Aposafranone, Action of Phosphoric Chloride on. O. Fischer and E. Hepp. Ber. **30**, 1827—1831.

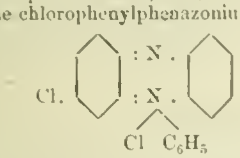
When one molecular proportion of rosindone is dissolved in five times its weight of phosphorus oxychloride and one molecular proportion of phosphorus pentachloride is added, a reaction occurs spontaneously. After distilling off the oxychloride, the yellow residue is washed with ether, and when recrystallised from alcohol, yields reddish-yellow shining metallic plates. The substance has the formula $C_{22}H_{14}N_2Cl_2$, is easily soluble in water, tolerably easily in alcohol, with a faint greenish fluorescence, but insoluble in ether or petroleum ether. It can be precipitated from its aqueous solution by salt, and it dissolves in concentrated sulphuric acid to a cherry-red solution, which is not dichroic. On dilution this solution turns yellow. The substance is unstable to alkalis, and is reconverted on standing with dilute caustic soda lye or sodium carbonate, or on boiling with sodium acetate, into rosindone. It reacts with potassium bromide, iodide, or nitrate, forming the corresponding salt, and it probably has the following constitution:—



that is to say, is chloronaphthophenazonium chloride, and the authors suggest that a similar transformation takes place here to that in the case of the diazotisation of the green rosinduline salts according to Kehrmann, which yields naphthophenazonium derivatives. They admit that this reaction of rosindone points rather to the anhydride than to the *p*-quinone formula of rosindone, although, of course, a transformation may have been effected by means of the phosphoric chloride. The salts of chloronaphthophenazonium readily react with bases. Thus, on dissolving the hydrochloride in an alcoholic solution, together with an excess of aniline, and warming slightly, phenylrosinduline

can be obtained from the melt after driving off the aniline with steam. It is formed quantitatively, and melts at 236° C. In a similar manner methyl rosinduline can be obtained by the action of methylamine, and crystallises from a mixture of benzene and petroleum spirit in greenish prisms melting at 181° C. On replacing the methylamine by ethylamine, ethylrosinduline is produced, which is identical with that previously obtained from benzene-azo-α-ethylnaphthylamine (Annalen, **256**, 237). It crystallises from dilute alcohol in greenish needles melting at 184° C. (see also this Journal, 1889, 877).

The action of phosphorus pentachloride on Aposafranone yields a product analogous to that obtained from rosindone. Finely powdered Aposafranone is dissolved in phosphorus oxychloride in the cold, and one molecular proportion of phosphorus pentachloride is carefully added. After standing about an hour with occasional agitation, a current of air is passed through the greenish-yellow solution at 40—50° C., to remove the phosphorus oxychloride. The residue is then taken up with water, and, after filtering and concentrating, the chlorophenylphenazonium chloride—



is salted out. Like the rosindone derivative, it reacts with aniline, and is converted by digestion with the base in an alcoholic solution into phenylaposafranine, $C_{24}H_{17}N_3$, which melts at 201° C. and gives a characteristic nitrate.
—T. A. L.

Azo Dyestuffs, The After-treatment of, with Copper Salts. C. Rumpf. Färber-Zeit. **8**, [19], 293.

As is now well known, dyestuffs which contain dianisidine as a component (such as benzo-azurine) can be made faster to light on the fibre, and, in a lesser degree, also to soap, by treating the dyed material with copper salts. The colour, however, is changed at the same time from a pure blue to a greenish shade. The author has treated with copper salts benzopurpurine 10 B, which also contains dianisidine, and obtained a reddish-blue. The fastness to soap was thereby increased, but not the fastness to light. From this and other like experiments with other dyestuffs, all of which gave varying results, the author concludes that the other components of the dyestuffs — besides the dianisidine—must have a share in the reaction when treated with copper salts.—I. S.

Bixin. K. G. Zwick. Ber. **30**, 1972.

The author describes a method by which he has separated Bixin, the colouring principle of Annatto, in a crystalline form. Well-dried Annatto is ground to a fine powder, moistened with chloroform, and extracted for 24 hours. After distilling off the chloroform, the residue is dried on a water-bath, ground, and extracted in a Soxhlet with petroleum ether of low boiling point. The undissolved residue, after drying, is again extracted with chloroform until the extract is colourless. The crystals which separate from the chloroform are, after drying, washed with petroleum ether, again extracted with chloroform, and separated by fractional crystallisation. They are then washed with a little chloroform, and dried, first in the air, and finally on a water-bath, and correspond in crystalline form and composition with those obtained by Etti.—T. A. L.

Maclurin, Dyeing Properties of. L. Schnell. Mitth. k. k. Techn. Gew. Museums in Wien, 1897, **7**, 171—172.

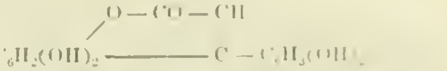
Two preparations of the compound were employed in the experiments: one, a pale yellow, crystalline powder, which was recrystallised from water; the other, the isolation of which had been effected by Herzig, an almost white product. They were found to behave similarly.

Maclurin dyes iron-mordanted cotton faint brownish-grey, alumina-mordanted cotton pale brownish-yellow, while it produces scarcely any change upon the same fibre when mordanted with chrome. The colours of the lakes produced are exceedingly pale.

To show that the dyeings are not due to the presence of traces of Morin in the samples, a portion of one of them was fractionally precipitated from aqueous solution by lead acetate. The various portions of the compound liberated from the several fractions, in dyeing, behaved alike.

Maclurin exhibits close analogies with the pyrogallol tannins: thus, it is precipitated by tartar emetic and albumin, and is partially, if not completely, absorbed from aqueous solution by hide powder, besides having dyeing properties similar to that class of compounds.

Ciamician has obtained by the action of sodium acetate and acetic anhydride on Maclurin, a coumarin compound of the formula—



This theoretically should possess greater colouring power than Maclurin. It does not, however. But its dibromo derivative is a dyestuff of some value.—E. B.

PATENTS.

Sulpho Acids, and Colouring Matters [Browns, Blacks] therefrom; Impts. in the Production of. Read Holliday, Sons, and Co., Ltd., J. Turner, and H. Dean, Huddersfield. Eng. Pat. 26,020, Nov. 18, 1896.

By reducing the 1.2′, 1.3′, or 1.4′-nitronaphthylamines with normal or acid sulphites soluble in water, they are converted into sulphonic acids of the corresponding naphthylene diamines, which yield tetrazo compounds capable of combination to form dyestuffs. 100 kilos. of 1.3′-nitronaphthylamine are boiled with 700 kilos. of concentrated sodium bisulphite until dissolved. The solution is acidified with hydrochloric acid, and boiled until all the sulphurous acid is expelled, when 1.3′-naphthylene diamine sulphonic acid crystallises out on cooling as a sandy, green precipitate. To obtain a brown dyestuff, 34 kilos. of the sulphonic acid are converted into the tetrazo compound and added to 36 kilos. of α-naphthylamine hydrochloride in solution. After filtering off the dyestuff, it is converted into the sodium salt, which dyes brown shades on vegetable fibres, and may be diazotised and developed thereon, yielding shades of considerable intensity. According to another process the patentees proceed as follows: 21·2 kilos. of tolidine are converted into the tetrazo compound, and run into a solution containing 68 kilos. of 1.2′-naphthylene diamine sulphonic acid made alkaline with soda. The dyestuff thus produced, gives brown shades on unmordanted cotton, and may be further diazotised and developed on the fibre. For the production of a black dyestuff, 18·4 kilos. of benzidine are converted into the tetrazo compound and added to a solution containing 34 kilos. of 1.2′-naphthylene diamine sulphonic acid containing just sufficient soda to neutralise the free acid. The intermediate product formed, is then run into a solution of 24 kilos. of amidonaphthol sulphonic acid G, dissolved in soda. After standing a few hours, the dyestuff is filtered off, pressed, and dried. It gives violet-black shades on vegetable fibres, which, after rediazotising and developing, yield deep ingrain blacks.
—T. A. L.

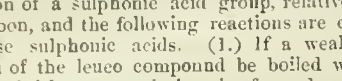
Basic Disazo Dyestuffs [Reds], Manufacture of. O. Imray, London. From "The Farbwerke vorm. Meister, Lucius, and Brüning," Hoechst a M., Germany. Eng. Pat. 27,896, Dec. 7, 1896.

The patentees find that disazo dyestuffs containing an aromatic amido ammonium base possess the important property of dyeing wool and cotton simultaneously from an acid bath. Such dyestuffs have been obtained by diazotising aromatic amido ammonium bases, and combining them with other components. Dyestuffs having similar properties may also be obtained by diazotising amido-azo compounds, and combining them with aromatic oxyammonium bases, such for instance as 2.2′-oxynaphthylene trimethyl ammonium. The diazo compound from 23·3 kilos. of amido-azobenzene hydrochloride, 18 kilos. of hydrochloric acid (20° B.), and 7 kilos. of sodium nitrite, after adding 22 kilos. of crystallised sodium acetate, is run

into a solution containing 24 kilos. of 2.2′-naphthol trimethylammonium chloride. The dyestuff is salted out and purified by re-dissolving. It forms red flakes, and dyes wool and cotton cherry red from an acid bath.—T. A. L.

Dyestuffs of the Series known as "Patent Blue," Manufacture of. O. Imray, London. From "The Farbwerke vorm. Meister, Lucius, and Brüning," Hoechst a/M., Germany. Eng. Pat. 29,706, Dec. 24, 1896.

The properties of dyeing evenly and of fastness possessed by dyestuffs of the Patent Blue series, depend upon the ortho position of a sulphonic acid group, relatively to the methane carbon, and the following reactions are characteristic for these sulphonic acids. (1.) If a weak mineral acid solution of the leuco compound be boiled with ferric chloride, a bluish-green solution is formed, which, on saturation with ammonia, gives an intensely blue filtrate. (2.) The Patent Blue dyestuffs on boiling with caustic soda lye, split off alkylamine and form violet to yellowish-red solutions of phenol dyestuffs. The dyestuffs hitherto known, belong to the diamidotriphenylmethane series. In the present specification, dyestuffs of this group are described which are derivatives of triamidodiphenyl-*o*-tolylmethane—



They may be obtained by condensing alkylated diamidodiphenyl carbinols with the sulphonic acids of monalkylated *o*-toluidine and oxidation of the leucosulphonic acids thus produced. If benzyl-o-toluidine be employed, it is to be noted that the first sulphonic acid enters the tolyl residue, and this acid is produced if the solution of the base in sulphuric acid monohydrate be mixed with fuming sulphuric acid. The disulphonic acid (the second sulphonic group being in the benzyl residue) is formed when the base is dissolved in fuming sulphuric acid. The condensation is carried out as follows:—27 kilos. of tetramethyl-diamido diphenylcarbinol are dissolved in 35 kilos. of hydrochloric acid (30 per cent.) and 100 litres of water, and mixed with 40 kilos. of the calcium salt of benzyl-*o*-toluidine disulphonic acid in 50 litres of water. The whole is heated for 10—12 hours on the water-bath until a sample is completely soluble in ammonia, indicating the disappearance of the carbinol. The solution is then treated with sodium carbonate until it has an alkaline reaction, filtered from calcium carbonate, and the leuco salt is salted out from the filtrate and dried. In order to oxidise it, 10 kilos. of the above salt are dissolved in 300 litres of water, the solution is heated to 60°—70° C., mixed with the calculated quantity of lead peroxide and acetic acid, and 6 kilos. of sodium sulphate are subsequently added to precipitate the lead. The dyestuff is salted out from the filtrate, or this latter is evaporated *in vacuo*. The new product forms copper-coloured crystals easily soluble in water to a blue solution, which is dichroïc, being bluish-red by transmitted, and bluish-green by reflected light. The solution is unaltered by ammonia or sodium carbonate, but turns violet when boiled with caustic soda lye. The dyestuff gives blue shades with a green reflex on wool and silk from an acid bath. The shades do not appear redder in artificial light.—T. A. L.

Brown Colouring Matter [from Dinitrocresol], Production of a. S. Pitt, Sutton. From L. Cassella and Co., Frankfort-on-Main, Germany. Eng. Pat. 29,828, Dec. 28, 1896.

When dinitrocresol is heated with sulphur and an alkaline sulphide, it yields a melt containing a dyestuff of unknown constitution, which gives brown shades on unmordanted cotton in presence of sulphides or strong alkalis. The following quantities are given:—25 kilos. of dinitrocresol are heated with 125 kilos. of sodium sulphide and 50 kilos. of sulphur for 1 hour to 100° C. The temperature is then gradually raised to 160° C., and maintained thereat until the melt has become solid, which takes about 2—3 hours. The melt, after having been broken up, can be used directly, or its solution may be carefully mixed with hydrochloric acid until the reaction is neutral. The precipitated dyestuff is filtered off and made into a paste or dried, but in this case it must be dissolved in soda lye or sodium sulphide before dyeing.—T. A. L.

Inks [Fugitive Plate] for Use in Printing from Engraved Plates, Impts. in. R. Webb, London. Eng. Pat. 26,992, Nov. 27, 1896.

1 drm. of sodium carbonate and 2 or 3 drms. of yellow soap are dissolved by boiling with 10 oz. of water, and the solution is well stirred into 4 oz. of a mixture of four parts of flour and one part of magnesia, the whole being tinted to shade with a soluble dye. In cold or damp weather a little dextrin may be added to assist the drying. When used on a "security" paper containing a sensitive substance the colour of which it is liable to change, some alkaline carbonate (preferably the ammonium salt) should be incorporated with the ink to prevent this action. The completed material forms a "security" or "Docteur" plate ink for printing from engraved (*i.e.*, intaglio) plates; and it is principally employed in the production of bankers' cheques, &c., to prevent fraud. [See also Eng. Pat. 19,783, below.]
—F. H. L.

Printing Inks, Impts. in the Method or Process of Making. D. J. Ogilvy, Cincinnati, U.S.A. Eng. Pat. 18,533, Aug. 10, 1897.

The essential feature of this patent consists in the statement that the pigment and vehicle of a (black) printers' ink can be effectually mixed together by agitation with high-pressure or superheated steam, so that the varnish is prepared, deodorised, and the ink finished all in one operation. The oil (rosin, petroleum, shale, linseed, &c.), the solid rosin, and the pigment (lampblack or carbon black) are placed in a closed vessel connected with a condenser, and the steam is turned on "until the result is satisfactory." "When sufficiently treated, the product is passed, if desired, through a straining or grinding apparatus . . . ." The proportions of the several ingredients depend partly on the weather and the class of trade for which the ink is intended, and partly on the amount of volatile matter driven off by the steam.
—F. H. L.

Printing Inks [Fugitive Letterpress], Impts. in. R. Webb, London. Eng. Pat. 19,783, Aug. 27, 1897.

Eighty-three parts of dextrin are mechanically mixed with 17 of treacle or golden syrup, 50 parts of glycerin added, and then sufficient aniline colouring matter to give the desired tint. The proportions vary somewhat, according to the state of the weather, and in summer it may be necessary to use a little phenol or salicylic acid as an antiseptic. The finished material forms a "fugitive" or "security" letterpress ink [for printing from type or relief].—F. H. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

Yarns, Enamelled. C. D. Rice, jun. Textile Colorist, 1897, **19**, 289—290.

Enamelled yarns are a device for producing various effects in woven designs. The yarn is impregnated with a hot paste, consisting of flour, tallow, grease, &c., with or without colouring matter; and, when cool, is passed between rollers to distribute the paste evenly. When dry, the yarn receives two or three successive coatings of a varnish or "enamel." "Flake effects" are produced by applying ground mica while the enamel is yet moist.—I. S.

Fatty Acids, Volatile; Production of, from Wool Washings. A. and P. Buisine. Comptes Rend. 1897, **125**, [20], 777—780.

When wool washings are allowed to stand, a spontaneous fermentation takes place, in which the volatile fatty acids, from acetic to caproic, are produced. It is suggested that these washings might serve as an abundant source of these acids, some of which are rare and difficult to procure. The

washings give the largest yield in volatile acids after a week's fermentation. The following table gives their composition at this period :—

Density	1·079		
Dry residue	153·4 grms. per litre.		
Ammonia (as carbonate)	1·5	"	"
Total nitrogen	1·5	"	"
Carbonate of potash (ready formed)	7·6	"	"
Volatile acids (expressed as H_2SO_4)	16·0	"	"
Fatty matter	15·5	"	"
Mineral matter	77·4	"	"
Total carbonate of potash	66·5	"	"

The fermented liquid is boiled to drive off ammonia, then acidified, and the volatile acids distilled off in a current of steam. The higher acids pass over first; the longer the distillation is continued the more acetic acid is obtained. The residual solution, on concentration, deposits potassium sulphate, and the calcined residue is rich in carbonate of potash. If carbonate of lime be added before calcination, all the potash is obtained as carbonate. The nitrogenous matters give ammonia, which may be collected.

The distillate contains the fatty acids in the following proportions:—

——	Per Cent. of the Mixture.	Per Litre of Washings. Grms.
Formic acid	Traces	Traces
Acetic "	60	10·7
Propionic "	25	5·4
Butyric "	5	1·3
Valeric "	4	1·2
Caproic "	3	1·0
Caprylic "	Traces	Traces
Benzoic "	3	1·0
Phenol	Traces	Traces

The washings of different wools vary little in composition, or in the yield of acids obtained. The crude mixture of acids is suitable for the preparation of acetone, methyl ethyl ketone, and the acetone oil used for the denaturing of alcohol. By the addition of the proper amount of carbonate of lime, followed by steam distillation, pure acetate of lime is obtained, the other acids coming over with the water.

The wool washings of Roubaix and Tourcoing amount to 500 cb. m. per day. By treatment of these, 10 kilos. of acetic acid, 5—6 kilos. of propionic acid, and 20 kilos. of ammonium sulphate might be obtained per cb. m. in addition to the carbonate of potash now produced.

—A. C. W.

Textiles, Soaps for. Leipziger Färber- u. Zeugdr. Zeit. **46**, [11], 491.

See under XII., *page* 1023.

PATENTS.

Textile Fabrics, Impts. relating to the Manufacture of. V. Bona, Carignano, Turin, Italy. Eng. Pat. 25,362, Nov. 11, 1896.

This invention relates to the use of the outer waste silk of the cocoons, silk-floss or "spelaia," as it is called, in the manufacture of mixed woollen goods. The material is ungummed, dried, carded, and combed. It is then mixed with shoddy or wool in the desired proportion, and treated as usual. The addition of 15 per cent. of floss-silk greatly increases the tensile strength and fineness of the thread that can be obtained from shoddy or wool, and a material improvement in the goods is effected.—H. I.

Silk-like Gloss Effects on Cotton or Linen Stuffs by means of Printing, Production of Local Durable. O. Imray, London. From The Farbwerke vormals Meister, Lucius, and Brüning, Hoechst a Main, Germany. Eng. Pat. 29,832, Dec. 28, 1896.

These are produced by the local mercerising of the fabric by caustic soda, according to a modification of the process of Thomas and Prevost. The material is printed with a composition containing, for example, 70 pts. of British gum and 930 pts. of soda lye of 40° B., with or without the addition of dyestuffs or mordants. It is then washed in "a stretched state until the inner tension of the fibre has ceased." For resist styles the fibre is printed with substances, such as albumin, casein, various oxides, acids, and salts, which will resist or neutralise the mercerising action of the caustic lye. The fabric is then mercerised and washed under tension as before. By this treatment, a permanent gloss is imparted to the cotton, and the affinity for dyestuffs and mordants is increased. See also Ger. Pat. 85,314 of Thomas and Prevost.—H. I.

Textile Plants [Ramie, &c.], Impts. in the Treatment of, to remove Fibre therefrom. C. L. Bachelerie, Paris. Eng. Pat. 12,511, May 20, 1897.

A quick method of retting is described in this invention. The freshly-cut plants (Ramie) are introduced, on trellis-work supports, into a large gas-tight chamber, and subjected for some minutes to the action of gaseous carbonic acid under a pressure of about two atmospheres. After removing the pressure, the plants are removed and dried. They are then passed through breaking rollers, when the plant gum is said to fall off and leave the fibre clean, supple, and brilliant, ready for the combing operation. By introducing sulphurous acid or chlorine into the gas, bleaching may be effected as the retting progresses.—H. I.

Nitrocellulose, Impts. in Treating, for the purpose of Coating or Impregnating Textile and other Fabrics. D. M. Sutherland, Sunbury-on-Thames, and W. McLaren, London. Eng. Pat. 28,613, Sept. 14, 1897.

This invention relates partly to solutions of nitrocellulose and analogous substances, such as parkesine, ivorine, xylonite, and celluloid, and partly to the methods of applying such substances to the purpose of coating textile fabrics. In one method of producing the solvent, pyroligneous acid is first prepared, by distilling off the spirit from crude acetic acid obtained by the destructive distillation of wood. The spirit is redistilled three times, the last two distillations being conducted in copper stills heated by steam coils, taking care that the temperature does not exceed 180° F. For the last distillation, from 1 to 1½ per cent. by weight of sodium carbonate is added, in order to neutralise any free acid that may remain; the acid in the distillate existing in the form of methyl and dimethyl acetate. The mixture used by the patentees is of a strength of about 42 overproof at 50° F. It is free from acidity, and rich in methyl alcohol and methyl acetate.

In preparing a suitable solution, nitrocellulose is then agitated with the spirit in suitable closed vessels, and with the addition of castor oils and gums.

The solution may be used for coating textile fabrics or for coating paper, plain or printed, felt, leather-cloth, oil-cloth, linoleum, and all other materials in web form having a fairly even surface, by using well-known spreading machines. After drying, suitable designs are stamped on the material.

—D. B.

Fibres, Vegetable, for Textile Purposes; Improved Process and Apparatus for Preparing. J. S. Brown, Bally, Bengal, India. Eng. Pat. 26,185, Nov. 23, 1897.

The stems of plants of the urtica or nettle family are placed in a cage and boiled for 1—1½ hours in a caustic alkaline solution, to which a proportion of kerosene oil has been added. After cooling and washing, the stems are fed into a straightening machine. This consists of a large, pegged drum into which 10 smaller pin-rollers work, removing from the large drum such stems as lie transversely and obliquely to the direction of motion. These stems are returned to the drum so that they lie parallel to the others. From the straightening machine, they pass between two endless aprons of wire-net, through a washing machine, where the remaining epidermis and gummy matters are removed; the stems are finally dried.—H. I.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Dyeing, The Theory of. L. Vignon. Comptes Rend. 1897, **125**, [6], 357—360.

The author investigates the nature of the dyeing process when cotton is dyed with the substantive or "direct" colouring matters, and seeks to establish which molecular groups confer this property of direct dyeing. He has estimated the amount of various substances, chemically related to the direct dyestuffs, taken up by cotton from boiling solutions. This estimation was effected either by the increase in weight of the well-washed cotton, or by the amount of the substance remaining in the bath after the operation. The following table gives the amount of each substance absorbed, per 100 grms. of cotton, after 15 minutes' boiling. The neutral bath consists of 1 grm. of substance dissolved in 250 c.c. of water; the alkaline bath contains in addition 3·5 grms. of sodium carbonate, 1 grm. of cotton being used in each case. The bases are employed in the form of neutral hydrochlorides.

——	Neutral Bath.	Alkaline Bath.
Ammonia	0·2—0·4	0·2
Hydroxylamine	0·0—0·3	0·2
Hydrazine	1·2	1·7
Phenylhydrazine	3·6	2·9
Diphenyl (dissolved in petroleum spirit).	0·0	..
Azobenzene (dissolved in petroleum spirit).	0·0	..
Aniline	0·1	0·1
Dimethylaniline	0·0	0·0
Diphenylamine	0·4	0·4
o-Phenylenediamine	0·4	0·6
m-Phenylenediamine	6·4	2·4
p-Phenylenediamine	6·7	3·2
Benzidine	5·7—6·2	5·6
Tetramethylbenzidine	7·0	6·3
Benzidine sulphonic acid	7·4	4·8
Diamidostilbene disulphonic acid	3·5	3·6
Dianisidine	6·9	5·7
Diamidonaphthalene	0·1	1·7

From these results, conclusions may be drawn as follows:—The fixation of the direct colours is evidently due to chemical combination, and depends upon molecular grouping. This property is not due to the presence of a benzene nucleus, to free nitrogen atoms, or to two nitrogen atoms joined to form the azo group, since diphenyl, ammonia, hydroxylamine, and azobenzene are not absorbed. However, the diamines (except *o*-phenylenediamine) and the hydrazines are absorbed to a considerable extent, and the absorption appears to be independent of the degree of substitution of the azotised molecular groups. Apparently the property of direct dyeing is due to the groupings $>N - R - N<$ or $>N - N<$ *i.e.*, to two hydrazinic nitrogen atoms, united either directly or by means of aromatic residues. The two nitrogen atoms may be combined with H, CH_3, or with other N atoms forming azo groups. These N atoms in the direct colouring matters can unite with the cellulose molecule, and thus become pentatomic—

$$\underset{\wedge}{>N} - \underset{\wedge}{N<}$$

This theory is supported by the fact that benzidine and tetramethylbenzidine are absorbed by the cotton, while the methyl iodide compound of tetramethylbenzidine, in which the nitrogen atoms are already pentatomic, is not absorbed in the least.—R. B. B.

Silk, The Dyeing of Artificial. Leipziger Färber- u. Zeugdr. Zeit. **46**, [11], 501.

The author directs attention to the researches of Kuhlmann on the dyeing of pyroxylin, which, though made in 1856, are of interest in connection with the dyeing of artificial silk. It is pointed out that moisture at a high temperature affects the strength of the fibre, and that, therefore, the temperature of the dye-bath should not exceed 60° C. Alkalinity or acidity of the bath should also be avoided, as tending to weaken the strength of the fibre. The most suitable dyestuffs are the basic colouring matters, which are applied in a cold and neutral bath.—I. S.

Indigo on Cotton Fabrics, Printing with. C. Kurz. Leipziger Färber- u. Zeugdr. Zeit. **46**, [11], 479—480.

The author describes the difficulties of printing with indigo according to the well-known method of Schlieper and Baum, and recommends the following simplified process:—10 grms. of pure indigo are dissolved in 200 c.c. of aniline, by digesting it in the latter at the boil for 15 minutes. The clear solution is decanted and allowed to cool. A finely divided indigo paste is thus obtained, well adapted for the purpose of printing. For thickening purposes the following preparation is used:—In 10 litres of soda lye of 38° B. are dissolved 4,800 grms. of sodium hydrate, to which are then added 10 litres of a dextrin solution, 1:1. To 8 parts of this thickening are added 2 parts of the indigo paste as above described, and the same is then printed on the fabric that has previously been padded in a solution of glucose of 5° B. After printing and drying, steam for two minutes in a Mather and Platt ager, and wash. 40 grms. of indigo per litre of printing colour is a proportion said to be sufficient for very deep shades.—I. S.

Lactic Acid as a Solvent for Dyestuffs. Leipziger Färber- u. Zeugdr. Zeit. **46**, [11], 486—487.

The use of lactic acid for dissolving dyestuffs which are insoluble in water, has been patented in France. For spirit-soluble Indulines and Indigo, about four times the weight of acid is taken, and the dyestuffs may then be used for printing on cotton or wool.—I. S.

Starch, Soluble. A. Wroblewski. Ber. 1897, **30**, [14], 2108.

See under XVI., *page* 1028.

Starch, Soluble. W. Syniewski. Ber. 1897, **30**, [16], 2415.

See under XVI., *page* 1029.

Catechu. Textile Colorist, 1897, **19**, 298.

See under XIV., *page* 1026.

PATENTS.

Bleaching Textile Vegetable Fibres, Yarns, and Fabrics; Impts. in. A. F. B. Gomess. Eng. Eng. Pat. 28,266, Dec. 10, 1896.

The ordinary caustic lye is replaced, according to this patent, by a mixture of caustic soda solution and zinc (dust), in the proportions necessary for the solution of the zinc oxide formed.—H. I.

Paranitraniline Red, Impts. in the Production of. A. G. Green, Heaton Moor, Stockport. Eng. Pat. 28,499, Dec. 12, 1896.

The process consists in "mercerising" the cotton fibre before impregnating it with the alkaline β-naphthol solution. The red so obtained is said to possess greater fulness, evenness, brilliancy, and blueness of shade than when dyed by the ordinary process, and it is also claimed to be faster to light. The cotton is immersed in cold caustic soda lye (30° Tw.) for a half to one hour. It is then washed and grounded with β-naphthol, which may be used of half the usual strength, *viz.*, 1 lb. instead of 2 lb. to every 8 galls. of water. The mercerising process can be shortened by employing a stronger caustic lye, such as one from 40°—50° Tw.—T. A. L.

Machine for Aniline Dyeing Cotton, and Drying Wool, Cotton, and the like; A New or Improved. T. Ender, A. Kleindienst, J. Procner, Pabianice, Russian-Poland. Eng. Pat. 19,081, Aug. 18, 1897.

The impregnated cotton is introduced into a large rotating drum or cylinder of wire gauze, in which are fitted longitudinal blades or lifters. By means of hot air, supplied by

a fan, the cotton is dried, a lagging or covering is placed over the drum, and the black is developed by blowing steam on to the cotton from a perforated pipe running outside the drum, but parallel to its axis of rotation.

—H. I.

ERRATUM.—This Journal, 1897, 908, col. 2. The numbers of the two Figs., I. and II., should be reversed.

VII.—ACIDS, ALKALIS, AND SALTS.

Sulphuric Acid Manufacture: Reduction of Chamber Space. E. Hartmann. Chem. Zeit. 1897, **21**, [848], 877.

To reduce the chamber space, Burgemeister considers it practicable to introduce a large number of vertical lead pipes, 40 to 50 cm. in diameter, into the first chamber. These, reaching from floor to roof, induce, in the first place, a stream of cold air, and, in the second place, offer to the gases, surfaces for impact and condensation.

The author, fearing leakage from the many joints, especially on the floor of the chamber, has devised a modification, which he has employed with success since 1893; according to this, these floor joints are avoided. The chamber shafts he makes 1·50 × 1·90 m. in section, and each is luted at the bottom by a water lute. These shafts, open top and bottom, exert a cooling effect, and aid the process by condensing the water vapour to form the water or weak acid necessary to bring about the decomposition of the nitrosulphonic acid.

Several years' experience, it is said, has shown that this modification increases the production of a system by about 20 per cent., and that with it, no difficulty is experienced in working off per 0·9—1 cb. m. of chamber space, 1 kilo. of sulphur in 24 hours. Compared with other arrangements for effecting a like purpose, it possesses the great advantage of being extremely cheap in construction.—J. T. C.

Sulphuric Acid [in Sulphates], Volumetric Determination of Combined. Félix Marboutin. Bull. Soc. Chim. **17**, [22], 953.

See under XXIII., *page* 1041.

PATENTS.

Lixiviating, Steaming, and Aërating; Impts in. J. Hargreaves, Farnworth-in-Widnes, Lancashire. Eng. Pat. 23,263, Oct. 20, 1896.

See under I., *page* 1001.

Zinc Chloride, Production of, or of Zinc and Chlorine. E. Hoepfner, London. Eng. Pat. 24,573, Nov. 3, 1896.

ZINC sulphide ores are treated with an acid, or with a solution of cupric chloride or ferric chloride, to extract silver and lead, especially if lead be present in any such proportion as to be injurious in the roasting process which follows. The sulphurous acid evolved in this roasting process, is converted into sulphuric acid, which is heated with sodium or potassium chloride to obtain an alkali sulphate and hydrochloric acid, which acid is digested with the roasted ore. It is preferred that the strong acid should act first upon ore that has already been subjected to acid treatment, in order that ferric chloride may pass into solution, which facilitates the solution of the precious metals, to aid which, chlorine may also be passed in. Finally, the acid solution is digested with excess of the untreated roasted ore. From the solution thus obtained, metals electro-negative to zinc are precipitated by known means, but preferably by electrolysis. Iron and manganese are then separated by chloride of lime, for instance, and the resulting solution of zinc chloride is electrolysed to obtain chlorine and pure zinc, the solution being concentrated when necessary. Chloride of sodium, potassium, calcium, or magnesium, should be present in the solution to increase its conductivity. The application of either the exhaust steam of the steam engines, or of the steam produced on concentrating the solutions, in feeding the sulphuric or other acid-producing apparatus, is claimed.

—E. S.

Soap, Dry, or Soap Powder; Impts. in the Manufacture and Production of. W. W. Wright and The United Alkali Co., Ltd., Liverpool. Eng. Pat. 27,906, Dec. 7, 1896.

See under XII., *page* 1023.

Sulphuric Acid [Spent Acid in Nitroglycerin Making], Impts. in Purifying and Concentrating; in part applicable to the Manufacture of Nitric Acid. G. Kynoch and Co., Ltd., and A. T. Cocking, Witton, Warwick. Eng. Pat. 28,891, Dec. 16, 1896.

See under XXII., *page* 1040.

Cyanides and other Products, Impts. in Obtaining, from Blast Furnaces. A. K. McCosh, Coatbridge, Lanark N.B. Eng. Pat. 381, Jan. 6, 1897.

See under X., *page* 1020.

Soda, Caustic, or Potash, Caustic; Impts. in the Manufacture of, from Carbonate of Soda, or Carbonate of Potash. P. J. Worsley, Clifton, Bristol, and The United Alkali Company, Ltd., Liverpool. Eng. Pat. 591, Jan. 8, 1897.

IN Loewig's process for the manufacture of caustic alkalis from their carbonates by strongly heating these with iron oxide, "it has hitherto been necessary to lift the materials out of the atmosphere of carbonic acid which is generated," but the inventors have found that this is unnecessary if the materials be well agitated on the bed of the reverberatory furnace. A furnace having a rotating horizontal bed and provided with mechanical agitators, such as a Mactear carbonating furnace, is preferred for this process.—E. S.

VIII.—GLASS, POTTERY, ENAMELS.

Glass, Colouring with Silver ("Silberlasur"), and the Cause of the Irregular Behaviour of different Glasses under this Operation. R. Zsigmondy. Sprechsaal, **28**, [39—45]; Dingler's Polyt. J. **306**, [4], 91—95.

FOR colouring small glass articles yellow, the author finds that the ordinary process of applying a mixture of clay and silver oxide to the surface, and then exposing to heat in a muffle, can be replaced by dipping the warmed glass in a flux composed of 20—30 parts of sodium metaphosphate and 4 parts of silver nitrate. As the glass cools down, the coating of colourless flux scales off, leaving the surface of the glass coloured a more or less decided yellow, according to the kind of glass used. The method is unsuitable for large glass articles, the necessary rapid cooling being in such case impossible of attainment.

With regard to the actual condition in which the metal is capable of colouring glass, the author showed, by heating a silvered tube in a current of hydrogen, that the silver is not absorbed in a metallic condition. On the other hand, when heated in a current of air, the oxidised silver is taken up by the glass, but without producing any coloration, this result not ensuing until the oxide is reduced to metal by re-heating in a current of hydrogen. Moreover, experiments with a large number of glasses of different composition, demonstrated the fact that no coloration is developed by the above phosphatic silver flux (or by the aforesaid ordinary method) in *pure* glass, the colour making its appearance only when ferrous oxide or some other reducing agent (such as metallic lead dissolved in the glass, arsenious salts, carbon, silicon, dissolved metallic sulphides, stannous oxide, &c.) is present. Ferric oxide, in a proportion as large as 2·4 per cent., produced no coloration, whereas the latter developed in presence of 0·96 per cent. of ferrous oxide.—C. S.

Glass Containing Silver, Surface Lustre in. —Zsigmondy. Dingl. Polyt. J. 1897, **306**, 68—72.

THE author some time ago endeavoured to make a glass with metallic lustre, by covering the surface with silver resinate, burning this off in a muffle in an oxidising atmosphere, and then reducing the surface. He has now obtained better results by fusing small quantities of silver nitrate with the materials for making a glass (either borate, silicate, or a mixture of the two), and, while the melt was still

hot, reducing the surface by short exposure to the vapour of alcohol. Borates of sodium, of lead, and of sodium and lead, as also those of cadmium and of bismuth, gave glasses with a fine lustre, some of which also exhibited a blue fluorescence. The borates of barium, calcium, cerium, and antimony gave glasses of too high fusing point to be experimented with, but some of the phosphates gave glasses with lustre. Silicate glasses of the composition—

$K_2O.PbO.6SiO_2$; $K_2O.2PbO.6SiO_2$, and $K_2O.PbO.5SiO_2$

were prepared, and fused with 1 per cent. of silver nitrate. In all cases the lustre was less pronounced than in the case of the borates, and not so easily or certainly produced. Mixtures were then made of lead borate, PbB_4O_7, and borax in equal proportions, to which were added varying proportions of the first of the above silicates; and to the glasses so obtained, silver nitrate was added. The best result was yielded by a mixture of 60 grms. of silicate, 20 grms. of the borates, and 1 grm. of silver nitrate. A smaller proportion of silver nitrate gave poor lustre, whilst a larger proportion of borate gave a very fusible and mobile glass, in which, by any overheating, the silver was easily reduced in the crucible, making the glass turbid and preventing the formation of any lustre. The same results were obtained when, instead of adding the silver nitrate to the ready-formed glasses, the whole of the raw materials—sand, litharge, boric acid, silver nitrate—were mixed and melted together. Mixtures of one of these glasses with 1 or 2 per cent. of various metallic oxides (copper, cobalt, manganese) gave coloured glasses, which also showed, on reduction by alcohol, beautiful surface lustre.

The author's view is that the silver in these glasses exists as silicate or borate; that the yellow colour which some of them show by transmitted light is probably due to the reduction of a minute quantity of silver which remains dissolved in the glass; that the lustre is due to silver reduced in a state of almost molecular division in a thin layer of glass, so nearly solid as to prevent the coalescence of the reduced molecules; and that the turbidity so easily produced by too long reducing action at a high temperature or in a very fluid glass, is due to larger particles of silver formed by the coalescence of the molecules first reduced.—J. T. D.

Glass, Sheet; Melting. R. L. Die Glashütte, **27**, [13], 147.

A useful formula for sheet glass is: Glauber salt, 50 parts; sand, 100; lime or powdered marble, 24; coke or charcoal, $2\frac{1}{2}$ parts. If the furnace work well, and good fireproof pots be available, an extra 5 parts of sand will improve the quality of the glass, but any larger quantity will make it opaque, hard, and brittle. An excess of lime also produces hard and brittle glass, liable to fracture and difficult to work. On the other hand, an increase of alkali renders the glass softer, more elastic, and easily workable, and is particularly advisable for the cooler classes of furnace. The coke should have been well roasted before grinding, and the quantity must be accurately controlled, an excess causing the glass to froth up during the melting, and to subsequently assume a dark bluish coloration, whilst if too little be taken, the glass will not be clear.

The furnace will require about $2\frac{1}{2}$ hours to become thoroughly heated. The well-mixed glass mass should then be piled up in the centre of the pots, to minimise the risk of cracking the hot sides. Opening wide the flue damper when filling the pots, lowers the temperature considerably and retards the melting. In 6 or 7 hours the second charge of glass mass may be put in the pots, and the remaining portions at subsequent intervals of 3—4 hours. Cullet is thrown in to assist the sandiver to separate, and when this latter makes its appearance (in about 1 hour), it should be burned off by means of a smoky reducing flame, unless the quantity (as a result of an insufficiency of coke) be very large, in which case it must be skimmed off. When this has been effected, the glass is well agitated by the air-blast to assist clarification. At this stage the temperature requires careful watching, and any pots giving signs of injury must be drawn. As soon as the glass is clear, the admission of gas to the furnace is stopped, and the latter allowed to cool down sufficiently for drawing the charge.
—C. S.

Glass, Dull [Frosted] Decorations on Graded. Diamant, **19**, [21], 475—477.

There are several ways of producing these decorations. One of them consists in slightly frosting the surface of the glass by applying thereto a mixture of flux, 500 parts; calcined borax, 100; and minium, 300 parts (ground to the proper consistency with oil of turpentine and thickened oil), by the aid of a brush, and then fusing this coating on to the glass at a moderate red heat. An ordinary muffle is less suitable for firing than the continuous furnaces of the Fürbringer type, or the continuous glass-annealing furnace. In the latter, a truckload of coated glass is kept in the first compartment for 10 to 15 minutes, and then passed on to the annealing compartment, where the actual firing takes place. The coating gradually loses its initially red colour and becomes lustrous, the operation being then complete. By dipping the articles in dilute acid for 1—2 minutes (1 vol. of hydrofluoric acid to 4—6 vols. of water is the best medium), the coating and dirt are removed, and the glass is left with a delicate fine-grained dull-lustrous surface. The second stage of the process is effected by means of the sand-blast, the portions to be left in their actual condition being protected from the sand by any of the methods usually employed.—C. S.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Blast-Furnace Slag Cement, Notes on the Manufacture and Properties of. A. D. Elbers. Eng. and Mining J. 1897, **64**, [18], 515.

In this communication the author describes and criticises an article by H. Détienne (Rev. Univ. des Mines, Sept. 1897). The cement experimented with consisted of granulated basic slag which had been dried by heating, and then ground fine in admixture with 15—20 per cent. of "dry-slaked" lime. The composition of the slag should not vary beyond the following limits:—SiO_2, 27—32 per cent.; Al_2O_3, 12—22 per cent.; CaO, 49—55 per cent.; and it is preferably dried by heating to about 500° C., or to a dark red heat, this operation involving a consumption of coke amounting to about 9 per cent. of the weight of dried slag. The dried slag is powdered and passed through a No. 80 sieve (76 meshes to the linear inch). The lime is slaked by immersion, all superfluous water being allowed to drop off, so that the slaked mass becomes thoroughly pulverulent; and it is then stored for 8—10 days, in order to dry it so thoroughly that it will easily pass through a sieve of 76 meshes to the inch (it is first passed through a coarse screen, in order to remove the small percentage of incompletely calcined limestone). The cement is prepared by simple dry-mixing. The chief defect in slag cements is the presence of soluble sulphides, and this cannot be overcome by treatment of the slag with nitric acid, as in the case of less basic slags (this Journal, 1897, 680, 800, and 915). Détienne states, with regard to the presence of sulphides, that the granulated slag, as used in the manufacture of the cement, contains from 1—3 per cent. of sulphur in the form of sulphate of calcium and sulphides of manganese and calcium; but in contact with the water used in "gauging" the cement, the calcium sulphide reacts with ferric oxide and forms calcium hydrate and ferrous sulphide. The latter is the cause of the greenish tint observed on the fractured surfaces of fresh briquettes made with slag cement. In contact with the air, the ferrous sulphide is oxidised, but no deterioration is said to occur on this account, since no alteration in volume or texture was observed in briquettes of slag cement after the lapse of four years. The author disagrees with this reasoning, and states that granulated slag, even under the most favourable circumstances, always contains several per cent. of calcium sulphide, and that only a very small portion of the latter is converted into calcium hydrate by the reactions which take place during "gauging," since these

reactions are merely superficial. He also states that if the briquettes had been kept under water for four years, or had been frequently subjected during that time to varying atmospheric influences, a diminution of strength would have taken place. On admixing small quantities of soda with blast-furnace slag cement, the rapidity of setting is increased (within a range of from 1—2½ per cent.) in proportion to the amount added; an admixture of 1¼—1½ per cent. of Na_2CO_3 is found to give the best results.

—A. S.

Old Mortar, Examination of. M. Gary. Mitt. aus den Königl. techn. Versuchsanstalten zu Berlin, 1897, 246.

In connection with the proposed addition to the middle tower of the church of St. Mary at Mühlhausen—a 15th century structure, partly burnt down in 1689—analyses were made of the old mortars. Samples were taken from different parts, and the following results were obtained:—

I. Clay mortar from a part of the foundations.

II. *a* and *b*. White lime mortars taken from the walls. The mud obtained on levigation showed, after drying at 100° C., the following composition:—

———	North and South Walls.	East Wall.
Insoluble in dilute hydrochloric acid..	13·98	9·69
Loss on ignition	37·25	39·81
Lime	43·66	46·66
Magnesia	0·59	0·81
Oxide of iron, alumina, and silica	4·28	2·96

The added grit consisted of calc-tuff sand.

III. Gypsum mortar from a part of the foundations and superstructure. Dried at 100° C., this gave results as follows:—

Insoluble in dilute hydrochloric acid	18·64
Loss on ignition	12·80
Lime	28·25
Magnesia	2·61
Oxide of iron, alumina, and silica	1·95
Sulphuric anhydride	32·39

The added material in the mortar appears to have consisted for the most part of broken tiles, &c., mixed with a clayey calcareous tufa sand.—H. H. B. S.

PATENTS.

Cement, The Manufacture of an Improved. La Société Metallurgique de Champigneulles et Neuves-Maisons, Pont Saint-Vincent, France. Eng. Pat. 1795, Jan. 22, 1897. (Under Internat. Convention, July 11, 1896.)

Blast-furnace slag is first granulated by running it into cold water as it leaves the blast furnace. It is then, whilst still moist, mixed with slaked lime to the consistency of mortar, and left to harden; after which the mass is crushed, baked, and ground. The result is a cement of high resistance, which sets extra rapidly. This is further used for the manufacture of cements which set less rapidly, by grinding it with dried granulated slag and lime.—H. H. B. S.

Joints, Pipe and other; A Composition or Cement for Making. S. R. Thompson, Liverpool. Eng. Pat. 27,975, Dec. 8, 1896.

The composition, which, it is claimed, will expand or contract with the expansion or contraction of the parts joined together, is made of 12 parts of Paris white, 5 of red ochre, 2½ of Venetian red, and ½ part of plumbago or graphite, ground, sifted, and mixed, and made into a stiff paste with boiled linseed oil.—H. H. B. S.

Wood, Impts. relating to the Impregnation of, and Apparatus therefor. G. F. Lebioda, Paris. Eng. Pat. 30,055, Dec. 30, 1896.

This invention consists of an injector, by means of which wood can be impregnated with solutions for staining purposes, &c., without the necessity of boiling, which is said to ruin the texture of the wood. The apparatus consists of a compressing chamber and a suction chamber, which are superposed, but separated by a perforated partition. The blocks of wood to be impregnated are fixed in an upright position by means of tightening screws in the compressing chamber, with their lower ends resting upon the perforated partition. The holes in the partition not covered by the wood are stopped by plugs, and melted pitch or similar substance is poured in, so as to run between the blocks and cover the partition to the depth of a few cm. By this means a tight joint is formed between the lower edges of the wood and the partition. The liquid is then forced longitudinally through the pores of the wood by the combined and simultaneous action of the compressing and suction chambers, operated by a suction and force pump.

—H. H. B. S.

Sand, Cement, and other similar Substances; Impts. in Apparatus for Measuring and Mixing. H. Alexander, Leeds. Eng. Pat. 13,506, June 1, 1897.

See under I., *page* 1005.

X.—METALLURGY.

Titaniferous Iron Ores, Utilising. A. J. Rossi. Eng. and Mining J. 1897, **64**, [19], 550. Read before the Amer. Inst. of Mining Engineers.

This paper relates to the author's U.S. patent for the utilisation of titaniferous iron ores in connection with the smelting of ores rich in phosphorus. It is claimed that by the admixture, prior to smelting, of a certain quantity of titaniferous ore, a strong, tough iron can be obtained, even when the amount of phosphorus present amounts to as much as 3 per cent.—A. S.

Iron, nearly Pure, Influence of Sudden Cooling on. A. Sauveur. Eng. and Mining J. 1897, **64**, [17], 489—490.

The author traverses the statement by Arnold (Engineering, July 6, 1897) that the critical thermal points Ar_2 and Ar_3 are, as such, without influence on the mechanical properties of steel, and quotes Arnold's own experiments as proof of the direct contrary; and further, he uses the same figures in controversion of Arnold's statement that between 500° and 900° C. the increase of tenacity is proportional to the quenching temperature. As a matter of fact, the temperatures 650°, 780°, and 887° C. are evidently marked out as special points; and this is conclusively shown by the following table:—

Quenching Temperature.	Tenacity after Quenching in Tons per sq. in.	Position of Quenching Temperature with regard to Critical Points.	Total Increase of Tenacity in Tons per sq. in.	Mean Increase of Tenacity for an Elevation of 1° C. of Quenching Temperature, in Lb. per sq. in.
° C.				
15	21·42	Below Ar_1	1·80	7
400	21·59	"	1·80	7
525	22·16	"	1·80	7
600	23·02	"	1·80	7
650	25·56	Ar_1 occurs here	2·54	114
705	26·24	Between Ar_1 and Ar_2	0·68	28
780	29·79	Ar_2 occurs here	3·55	106
820	29·16	Between Ar_2 and Ar_3	− 0·63	− 18
887	32·63	Ar_3 occurs here	3·17	106
928	31·35	Above Ar	− 1·28	− 70

—W. G. M.

Molybdenum-Steel, Some Properties of. W. v. Lipin. Stahl u. Eisen. 1897, **17**, 572; through Chem. Zeit. Rep. 1897, **26**, 205.

The influence of molybdenum on the properties of steel is commonly supposed to resemble that of tungsten. The author has compared two blocks of crucible steel, one containing 3·72 per cent. of molybdenum (with 0·55 C, 0·1 Si, 0·13 Mn, 0·021 P, and trace of sulphur) and the other 3·82 per cent. of tungsten (with 0·55 C, 0·07 Si, and

0·13 Mn). The metal did not rise on pouring; the slag of the molybdenum steel was half vitreous, greenish-grey, and showed a play of colours. For smith-work, both steels must be heated. Molybdenum steel exhibits a perfectly smooth surface, whilst the harder tungsten steel often shows minute cracks. Tests of the two steels were made, after treatment in various ways, by hardening and annealing. Strongly annealed molybdenum steel is softer than tungsten steel of the same composition. Heating to a very high temperature, such as is good for chrome or carbon steels, is unsuitable for molybdenum steel, whilst hardening in oil and subsequent annealing, which improves other kinds of steel, is almost useless for it. The usual method of hardening in oil has more influence on tungsten steel than on the sample containing molybdenum, but more energetic hardening in water acts in the opposite direction.—W. G. M.

Argentiferous and Auriferous Lead, Notes on the Sampling of. A. C. Claudet. Read before the Inst. of Mining and Met., Oct. 20, 1897.

It has been found that different parts of the same bar of lead carrying silver or gold, vary very much in their contents of these metals, and this circumstance affects the sampling of argentiferous and auriferous lead bullion on the large scale. The surface of a bar is, as a rule, poorer in silver and gold than the bottom, but when the gold contents become high, the reverse appears to be the case. The ordinary methods of sampling are as follows:—(1.) Cutting chips out of the tops and bottoms of the bars. (2.) Sawing, by a circular saw, through the bars. (3.) Melting the bars in mass into clean lead and dross.

In *System* (1), chips are cut by a gouge or chisel out of the top and bottom of every bar, or alternately the top of one bar and the bottom of the next. The chips are melted, either together into one bar, or those from the tops and bottoms separately into two bars. On assaying the bars, the results are compensated for enrichment on melting, if necessary. This method should only be employed with lead containing 10—20 oz. of silver per ton.

System (2).—The bars are sawn transversely, either half way or right through. The sawings are either melted into small bars and assayed, or they are assayed without melting; in the latter case, the result must be corrected for impoverishment, due to the oil which has to be used when sawing the bars. The teeth of the saw should be rather small, to prevent too frequent breakages and to produce small-sized sawings. This method may be satisfactorily used with lead containing silver up to 100 or 200 oz. per ton.

System (3).—The lead is melted in large iron pots, holding from 10—30 tons; the charge is heated and skimmed until the molten lead is quite clean; the temperature is then raised as high as possible, the lead well stirred up, and a "dip" sample taken. The dross is broken down when cold and a sample taken. The two samples are then assayed, and the results corrected and calculated with regard to the original lead charge. This method takes the longest time, but is stated to yield the most reliable results; it should be employed in all cases where the silver is above 200 oz. per ton, and where the gold is above 1 or 2 oz. per ton. Nine plates accompany the paper, fully illustrating the precise methods of obtaining samples by cutting or sawing the bars.—A. S.

Gold, Action of Potassium Zinc Cyanide Solutions on. W. J. Sharwood. Eng. and Mining J. 1897, **64**, 396, 426, 460—461.

The author, after a large number of experiments, arrives at the following conclusions:—The gold-dissolving power of a solution of potassium zinc cyanide increases with a rise of temperature, slightly with strength of solution, and rapidly with increased access of oxygen; the presence of the latter is the most essential condition. It is also increased by the addition of alkali, the increase being greatest in solutions to which oxygen has free access. Gold dissolves in the solution of double cyanide to a clear solution at first, but after some time a white precipitate forms, gradually coating the metal and retarding further action. The amount of precipitate formed is not proportional to the gold dissolved, but is relatively less when the amount of solvent is large; it consists of a somewhat variable mixture of the cyanide and oxide of zinc. The solvent action of a solution of potassium zinc cyanide is less than that of a solution of potassium cyanide containing the same amount or half the amount of cyanogen. The considerable increase in solvent power observed when caustic potash is added (oxygen being easily accessible) to potassium zinc cyanide, seems to indicate the formation of free potassium cyanide, and consequently that in dilute solutions (0·3 to 0·6 per cent.) potassium zinc cyanide is partially decomposed by alkalis; this is confirmed by the fact that on mixing solutions of alkali and potassium zinc cyanide, a slight fall of temperature occurs (Ellis, this Journal, 1897, **16**, 116). Reference is also made to the articles of W. R. Feldtmann (this Journal, 1894, 218 and 952); W. H. Virgoe (Eng. and Mining J. 1894, **58**, 314; this Journal, 1894, 739); G. A. Goyder (this Journal, 1894, 523); J. S. C. Wells (Eng. and Mining J. 1895, **60**, 584, and 1896, **61**, 179; this Journal, 1896, 116); N. Anderson (Proc. Colorado Scient. Soc., April 1895); and Bettel (Chem. News, 1895, **72**, 298).—A. S.

Gold Extraction, By-Products from. C. Butters. Proc. Chem. and Metall. Soc. of S. Africa, Meeting of Sept. 18, 1897, 14—20.

By-Products from Stamp Mills.—Inside and outside copper plates, the screens through which the pulp passes, rags and brushes used in cleaning the plates, screen frames, mortar-boxes, shoes and dies, all tend to retain amalgam. The clean-up gives plate-scrapings, including gold amalgam, particles of iron and copper, sand, iron pyrites (especially magnetic pyrites when present), a little grease, and coloured films and other sulphur compounds. The amalgam is cleaned by grinding and washing, generally in an amalgamating barrel, with such chemicals as lime or cyanide of potassium. The ashes from the rags, &c. (which are burned to remove organic matter), together with scalings and any other matter that may contain amalgam, are placed also in this receptacle, which is usually a grinding barrel, and the mixture ultimately yields fine silica, slime, and water containing chemicals. At the best mills these products are further treated, because some of the mercury will have floured, some of the hard amalgam will have become finely crushed, and some of the rest will have become floured in the grinding of the pyrites, whilst in the presence of cyanide, some gold will have been dissolved. Hence the slimes are carefully settled, and the liquid is run into a collecting tank containing any scrap iron that is likely to carry gold, and is here acidified with sulphuric acid. The sludge from this tank, together with the slimes, will contain from 50 to 300 oz. of gold to the ton; the former is cleaned up once or twice, the latter twice or four times in the year, according to the size of the mill, the products being sent away for treatment. From 40 to 50 oz. of gold may thus be easily obtained per mensem from a 100-stamp mill, although in many mines these materials are run to waste. The screens, after being rusted and beaten, retain about 2 oz. of gold per ton; they may be used as blast-furnace flux, or worked up for ferrous sulphate in chlorination works, the resulting sludge being afterwards treated. Worn-out copper plates, even after most careful scaling, should always be refined, as the value of the gold contained, is at least equal to that of the copper. Iron battery chips also contain amalgam even after cleaning. These chips, screens, and old iron generally may be efficiently and cheaply rusted by placing alternate layers of screens, chips, and clean concentrates (containing as much sulphur as possible) in a warm place, near a boiler-furnace, or under a glass frame (like a gardener's forcing-house), and watering the heap frequently with warm water and salt for from four to six weeks. The whole is then dried and run through a ball-mill, and the product may be used as blast-furnace flux, any iron that is not rusted through, being so deeply scaled as to be free from amalgam.

By-Products from Cyanide Works.—These by-products are much less in quantity from fresh than from oxidised pyritous ores. In the latter case ferrous and ferric salts are

formed, and tend with cyanide and alkali to produce ferrocyanides and hydroxides, which diminish the efficiency of chemical precipitation and increase the amount of sludge from electrical precipitation boxes. Moreover, the lime required for acid ores passes into solution as hydroxide, carbonate, or sulphate, and these crystallise out in the precipitation boxes. Lead cathodes thus become crusted with lime salts, which gradually slip off and carry gold with them into the sludge, and increase the proportion of gold carried in suspension. In zinc precipitation any deposit in the boxes is treated in the clean-up, but the complex cyanides, clay, humus, and flocculent precipitates held in suspension always carry gold with them, which may be in part deposited in the sump. With fresh ores the filtration of the still suspended residue should not pay, but with acid ores the material may retain from 1 to 20 oz. per ton. The sludge from the sumps should be removed at intervals, air-dried, and sold to smelting works. Except after roasting or other suitable treatment, such precipitates do not readily yield their gold to cyanide solutions.

By-Products from Siemens-Halske Precipitation Boxes.—The iron anodes gradually oxidise and form Prussian blue and ferric hydroxide. These partly sink to the bottom and partly float through into the sump, where by degrees they become peroxidised. The sacking surrounding the anodes, becomes saturated with the same deposit and, after a time, rotten, and is removed and burnt, whilst the anodes are carefully scaled. In a sand plant, working with clear solutions, all this Prussian blue may contain from 5 to 50 oz. of gold per ton. But in a slime plant the precipitate becomes contaminated and has a lower value. Any precipitation process is handicapped by the mass of suspended matter, and the author is now constructing sand filters for clarification, prior to precipitation. When sand and slimes are worked together, as at the Bonanza, clarification can be largely effected by passing the solutions through the sand vats. The precipitates formed in the vats are far less prejudicial than silica or clay, 1 oz. to the ton of silicious slime sufficing so to coat anode and cathode that gold does not adhere properly and a higher voltage is required.

Siemens and Halske Melting-Room By-Products.—When the lead is fairly clean and free from silicious mud or iron salts, the melting in the reverberatory furnace (5 ft. by 2 ft. 6 in.) is rapid, and a little coal is spread over the surface, while the temperature is urged to the utmost possible extent. Clean lead with 5—10 per cent. of Au is obtained, together with either a liquid litharge slag or (with dirty strips) pulverulent "skimmings." If more than one melt be required for a clean-up, the old skimmings are sweated of their lead contents, as far as possible, in the second charge. The hot liquated skimmings are then drawn, cooled, ground under a Chilian edge-roller, and screened through a mesh with eight holes per linear inch, whereby two grades are formed, known as "coarse metallics" and "ground skimmings." The former are melted in iron pots and run into bars, which are counted as lead bullion, whilst the skimmings form by-products. The lead from the reverberatory is also re-melted, and the skimmings from these pots are added to those from the furnace. The amount of gold in the skimmings may, with care, be reduced to under 1 per cent. The cupellation of the bullion produces about ½ per cent. of "refinery by-products."

Chlorination Works By-Products.—The only by-product here is the sludge, cleaned up once or twice a year, from the second set of precipitation vats. Ferrous sulphate precipitation from chlorine solutions is incomplete in the presence of some salts, and there is always gold in suspension. In some cases the liquid is drawn off after 24 hours, in others after 48, but in the author's works after 72 hours; even then the assay value is from 4 to 12 grains, unless there is much copper or lime present, when it may even be as high as 2 dwt. Hydrogen sulphide precipitation gives the lowest and therefore the best results in this respect, and the acid liquor drawn off from the gold and placed in tanks filled with scrap-iron, decomposes rapidly, yielding a precipitate of gold, copper, and basic iron sulphates and hydroxides. In a cold climate the vats should be enclosed in a house and slightly warmed by steam. The liquor may be run to waste, assaying less than 2 grains after a week. The sludge from these final precipitation vats is worth from 1s. to 8s. per ton of ore treated, and in California yields from 2 to 2 per cent. of the total gold obtained in the chlorination works. The by-products from melting the slimes, which should be nearly pure gold, are necessarily few, the slag produced being treated of in the next division.

By-Products from the Melting Room.—The slime taken to the melting room after sulphuric acid treatment and washing, may contain from 25 to 90 per cent. of gold. Loss is incurred in handling this or the precipitate from the zinc boxes (on both the hands and the tools in mixing with fluxes), in transferring to the crucible, in the slag, in the mould, and in the sink where the bar is scrubbed, but this loss may be for the most part temporary. Every melting room should be provided with two 100 to 200 gallon tanks, into which all washings from hands or tools, and from the sink, are thrown. After four or five months the liquid is assayed and drawn off, and the precipitate is cleaned up; 200 oz. of gold have thus been recovered from one tank. The pots may be scaled and the scalings melted after grinding, but even then the pots will retain 20 to 40 oz. per ton. Ashes and flue-dust from the melting furnaces may be cleaned up, the former averaging 5 oz. per ton, and being (with the pots) the most refractory material to be treated. Slag from melting zinc-gold slimes assays from 25 to 200, but averages 50 oz. of gold per ton. When the zinc slimes have been treated by the acid process, the thin lead matte found on the gold after pouring, contains about 11 per cent. of Au. The melting or roasting furnace itself becomes saturated with gold, and the old masonry of a renovated furnace will commonly assay 20 oz. per ton; where, however, only mill-gold is melted, only the immediate lining of the furnace is valuable. In panning ground slags for coarse gold, 20—25 per cent. of the slag form slimes, which are frequently allowed to run to waste, but which should be saved by allowing the water to stand in tanks until subsidence is complete, for the fine material is as rich in gold as the coarse. The floor of the melting room should either be of iron or be cemented, and should be kept scrupulously clean, the sweepings being saved, as they always assay over 5 oz. of gold per ton.—W. G. M.

Copper, Impurities in Raw. M. Schlagdenhauffen. Comptes Rend. **125**, [16], 573—574.

Filings of crude copper, such as Chili bar or English blister, were allowed to remain in water for several days, when, after filtering and slightly acidifying, the water yielded, with hydrogen sulphide, yellow and orange-yellow precipitates of sulphides of arsenic and antimony. If the same sample, after complete exhaustion with water, be treated with potash or dilute hydrochloric acid, a further and larger quantity of arsenic and antimony is dissolved out. Thus arsenic and antimony may probably be present in the form of oxides.

When similar filings were heated in a combustion tube in a current of carbon dioxide, a crystalline sublimate was obtained, containing arsenic and antimony. Further experiments, however, tend to show that the arsenic and antimony are not wholly present as oxides.

With other samples of copper the author has detected selenium in the sublimate by means of the red precipitate with sulphurous acid and by the red ring produced on sublimation in the presence of ammonium chloride. The author believes this to be the first time that selenium has been detected in raw copper. Also by heating copper in a tube, he has obtained a sublimate of cubical crystals of lead sulphate.—A. W.

Crucibles, Ground Graphite; Notes on the Assay of. A. F. Crosse. Chem. and Metall. Soc. of South Africa. Proc. of Meeting of Sept. 18, 1897, 11—12.

See under XXIII., *page* 1041.

Ferro-Silicon, Commercial. E. Donath and M. Haissig. Stahl u. Eisen, 1897, **17**, 670; Chem. Zeit. Rep., 1897, **26**, 205.

See under XXIII., *page* 1042.

Sulphur Chloride, Analysis of. G. A. Le Roy. Rev. de Chim. Indust. 1897, **8**, [94], 294.

See under XXIII., *page* 1040.

Iron Ores, Method for Estimating the Reducibility of. J. Wiborgh. Chem. Zeit. 1897, **21**, [75], 750.

See under XXIII., *page* 1041.

Copper, Quantitative Analysis of Commercial. Hampe. Berg- u. Hüttenm. Zeit. 1897, **56**, 201 and 297.

See under XXIII., *page* 1043.

PATENTS.

Lead, Zinc, Gold, Silver, and other Metals; Impts. in the Extraction of, from Sulphides, Refractory or other Ores, or Mattes. J. Armstrong, London. Eng. Pat. 22,361, Oct. 9, 1896.

Refractory sulphide ores or mattes containing zinc are heated with carbonate or nitrate of soda or potash, lime, and carbon in a reducing atmosphere, in a furnace with a sloping bed. The charge melts and separates into three layers, *i.e.*, metal, a slag of calcium silicate, and a top brittle layer of a mixture of zinc, zinc salts, and soda salts. The latter is further distilled in a closed furnace for zinc, and the residue therefrom extracted with water to recover sodium sulphide, which is re-converted into carbonate for further use. The lime slag is also lixiviated with water for the same purpose.—A. W.

Furnaces, Cupellation; Impts. in the Beds or Tests of, and of Assay Cupels and the like. T. B. McGhie, London, and E. G. Ballard, Chester. Eng. Pat. 26,493, Nov. 23, 1896.

Barium sulphate is to be substituted for bone-ash; the mineral barytes is crushed, and moulded under pressure, with or without water. To prevent cracking of the finished cupel, the material should be calcined before use; it may, with advantage, be mixed with 5—10 per cent. of Portland or other lime cement. For the lining of improving furnaces or the like, 3 parts (by measure) of barytes may be mixed with 1 part of cement, the mixture being gauged with water and laid on with a trowel. Sodium or potassium sulphate may be used as a binding medium. When the tests are saturated with lead, they are fluxed with fluorspar, and smelted.—W. G. M.

Aluminium, Impts. in the Purification of. P. E. Placet, Paris. Eng. Pat. 28,727 (under Internat. Convention), Dec. 15, 1896.

Molten aluminium is placed in a crucible or furnace lined with magnesia, and is there stirred vigorously with potassium bichromate. The impurities in the aluminium are thus said to be removed in combination with the oxygen of the bichromate, and the chromium thus introduced into the aluminium improves the qualities of the metal. When the reaction is complete, a flux of chlorides, chromates, or fluorides is to be added. Other bichromates may be substituted for the potassium salt, the bichromates of nickel, copper, or other element being employed when one of these latter metals is ultimately to be alloyed with the aluminium. Permanganates or chlorates may be employed, or nitrates, excepting those of potassium, sodium, or ammonium (which act prejudicially upon the aluminium).—W. G. M.

Furnaces for Smelting Ores and Melting Metals by means of Liquid Fuel, Impts. in. L. Pietraschewski, Borissoglebsk, Russia. Eng. Pat. 29,467, Dec. 22, 1896.

A reverberatory furnace is arranged with a vertical shaft and chimney at one end, and a sump with tap hole in the centre, the bed sloping gradually from the shaft to the sump. At the other end of the furnace is a heating chamber with a row of liquid fuel burners projecting the fuel along the main axis of the furnace. The fuel is supplied from a high-level reservoir at one side of the furnace, and is passed through a regulator, which enables the pressure of the liquid to be maintained constantly a little higher than the back pressure in the furnace. Air is blown in through the burner by a fan-blower, and may be pre-heated if desired. The ore or metal is fed into the shaft through a charging door, and the molten material afterwards flows into the sump, from which it is tapped as required. In this furnace, cast iron may be melted in about 20, or steel in about 40 minutes from charging. A furnace capable of melting 1,600 kilos. per hour requires only two attendants. Where a metal is to be decarburised, an automatically regulated stream of liquid fuel is to be turned into the shaft. The regulator consists of a float with a spindle valve at its end, in combination with an outlet having a valve seat, for the liquid fuel.—W. G. M.

Furnaces, Crucible; Impts. in. J. S. V. Bickford, Falmouth. Eng. Pat. 29,705, Dec. 24, 1896.

The crucible is placed in a circular chamber with an outlet to a flue at one side, near the bottom. A lid covers the chamber, and through it, a vertical blowpipe is passed so that its flame is projected into the middle of the crucible. The blowpipe consists of a T-tube, with the cross tube vertical, one end passing through the furnace lid and remaining open, whilst the other end is plugged, except for an air injector of smaller bore which passes into it. The lateral tube is supplied with a mixture of air and gas by a second injector. Around the central aperture in the furnace lid is a series of smaller holes (which may be opened or closed at will) for the admission of air, so that a flame may play around as well as into the crucible.—W. G. M.

Crucible Melting Furnaces, Impts. in. W. Kirkham, Sheffield. Eng. Pat. 29,709, Dec. 24, 1896.

The object of this invention is to dipense with separate or detached regenerators by so constructing the part of the gas-heated crucible furnace in front of the pot holes, that the heat from the latter is utilised in heating the air and gas previous to combustion. This is carried out by arranging a series of pot holes side by side with a straight front, along which is constructed, in the brickwork, the tubes supplying the air and gas, with suitable valves for shutting off or directing the gases into any particular pot hole as may be required.—A. W.

Cyanides and other Products, Impts. in Obtaining from Blast Furnaces. A. K. McCosh, Coatbridge, Lanark, N.B. Eng. Pat. 381, Jan. 6, 1897.

Apertures are made through the boshes of the blast furnace at a height of from 8 to 12 ft. above the twyer level, through which gases are withdrawn and washed, ammonia and cyanides being obtained from the liquors. The pipes passing through the apertures open into a large pipe, encircling the furnace, connected to a main leading to the washing apparatus and fan, and also through a stop valve to the furnace blast pipes, so that, if required, air can be admitted from the pipes.—E. S.

Aluminium, Impts. relating to. H. Griffith, jun., Birmingham. From V. Coppée and A. E. Kemplen, Paris. Eng. Pat. 6991, March 17, 1897.

The resistance of aluminium is to be so increased that the metal shall be available for the construction of cycle components, &c. The aluminium is melted in a steel crucible in such a way that no flame shall come into contact with the surface of the metal; about 0·5 to 2 per cent. of tungsten, which has been mixed into a stiff paste with borax, and moulded into cakes, is plunged to the bottom of the crucible. So much gas is thus evolved that when the maximum amount of tungsten is to be added (to produce the most highly resistant metal), it should be introduced in two batches. When the agitation of the metal has ceased, the contents of the crucible are well stirred and poured into moulds.—W. G. M.

Aluminium, Brazing of; Impts. relating to. H. Griffith, jun., Birmingham. From V. Coppée and A. E. Kemplen, of Paris. Eng. Pat. 6993, March 17, 1897.

50 parts of zinc are fused with 10 parts of tin or bismuth, and cast; the resulting plates are then rolled to the thickness of about 2 mm., and electro-plated with nickel or silver, after which they are subjected to hammering or

compression. The parts of aluminium to be brazed are heated sufficiently to melt the ends of the soldering strip when placed in contact with them. When they have thus been coated with the solder, they are held together at a temperature sufficient to flush the solder, and are finally cooled.—W. G. M.

Metals [Antimony, &c.], Improved Method of and Apparatus for Extracting, from their Ores or other Compound Bodies containing them. W. G. M. Foote, Sydney, Australia. Eng. Pat. 14,138, June 15, 1897.

The ore is mixed with lime (either anhydrous or hydrate) and carbon, and heated in long muffle-shaped fireclay retorts from the outside. The beds of the retorts, instead of being flat, slope downwards from the centre towards a longitudinal channel at each side, and the whole retort slopes from one end to the other, so that any molten portion of the charge will flow along these channels to the lower end of the same. At this end, which is at the back of the furnace, is constructed a well with a tapping hole at its base, which acts as a reservoir for the molten metals and slags during the periods between the tappings. It is not necessary that all the gangue should fuse into slag, as the retorts may be used for processes, such as antimony extraction, where the metal is obtained by liquation.—A. W.

Sulphide Ores [Zinc], Impts. in the Treatment of Complex, and Apparatus therefor. F. Ellershausen, London. Eng. Pat. 16,808, July 15, 1897.

In treating complex sulphide ores, 100 parts are mixed with about 50 parts of oxide of iron or manganese, and 25 parts of carbon, and the whole is maintained at a bright red heat in a reverberatory or other suitable furnace. Fumes of lead, zinc, and sulphur dioxide are produced, and are carried, together with the fire gases, into a chamber, where they are met with air and water, by which means the metallic fumes are converted into soluble zinc sulphate and insoluble lead sulphate. The former is separated by solution, and the latter, which may contain silver, is mixed with the fused residue of the furnace charge to produce a mixture suitable for smelting in the blast furnace.—A. W.

Aluminium, An Improved Process of Soldering. F. George, Laeken-lez-Bruxelles, Belgium. Eng. Pat. 18,731, Aug. 12, 1897.

This process is to solder aluminium by means of aluminium itself. A bath is prepared consisting of 1 part of sea salt or common salt, and 2 parts of turpentine or other hydrocarbon. The parts to be soldered are heated to dull redness and cooled in this mixture, which treatment is said to modify the molecular condition of the metal. After cooling, the parts are lightly powdered with borax, and aluminium in a state of fusion is interposed, whereby the soldering is effected.—A. W.

Amalgamating Pan, An Improved. E. H. T. Plant and S. Dellow, both of Charters Towers, Queensland, Australia. Eng. Pat. 19,180, Aug. 24, 1897.

The pan is made of cast iron with a slope at the centre, then a flat bottom about half an inch in depth, and a deeper recess round the circumference to collect the amalgam. A metallic muller, slightly less than the diameter of the pan, floats on the mercury which covers the bottom, and rotates by means of arms which are keyed to a revolving shaft which rises through an opening in the centre of the pan. The crushed ore and water are fed into a cylindrical casing above the centre, on to the sloping portion of the pan, and thence between the muller and the flat portion containing the mercury, to a sloping shoot at the outer edge.—A. W.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(A.)—ELECTRO-CHEMISTRY.

"Nitrogurete." H. Pauli. Zeits. f. Elektrochem. **4**, [5], 1897, 137—139.

Under the above title, Grove described in the Phil. Mag. for 1841 a series of remarkable metallic compounds containing nitrogen, which were supposed to have been obtained by electrolysis. The electrolyte contained an aqueous solution of an ammonium salt. As positive poles he used successively zinc, cadmium, and copper. The object was to liberate at the cathode the metallic ions simultaneously with ammonia and hydrogen, and to cause them to unite. No details of the current-density are given, but as a source of current he used six of the cells known by his name. The salt of ammonium employed, was sal-ammoniac (in concentrated solution). Grove's results are quoted without question in various text-books, &c. (e.g. Gmelin-Kraut, Thorpe, Pogg. Ann., Watts' Dict.). But Aslanoglou (Chem. News, **64**, 313, 1891), in endeavouring to prepare the copper compound according to Grove's method, arrived at quite different results. At Prof. Landolt's suggestion the author has made experiments on the supposed zinc compound, and he confirms Aslanoglou's results, to the effect that the supposed copper-nitrogurete consists simply of copper and cuprous oxide, its colour varying from dark brown to red.—D. E. J.

PATENTS.

Electrical Batteries, Impts. in. [Zinc, Lead.] C. E. O'Keenan, Paris, France. Eng. Pat. 27,167, Nov. 30, 1896.

Negative plates which have been charged in the usual way are exposed to the air to oxidise them. They are then placed in dilute sulphuric acid until the formation of sulphate of lead ceases, when they are rinsed in water, which may contain a little carbonate of soda to neutralise any free acid that may adhere to them, and they are then set up in a battery, in which the acid is replaced by sulphate of zinc and the peroxide of lead by zinc plates. When the E.M.F. falls, the lead plates are again exposed to the air to oxidise them, and treated as before. The electrodes may be formed in the battery itself, if lead plates containing chloride of lead pastilles are employed. When the reduction is complete, the plates can be used as ordinary and well-formed plates. As the battery works, the density of the sulphate increases, and the dense liquid is displaced into a system of pipes communicating with a supply of water and the battery, so that it is diluted to the proper extent, the equilibrium of the liquids in the tubes being in inverse ratio to their densities.—G. H. R.

Accumulators, Lead; Impts. in. A. Dodd, Wolverhampton. Eng. Pat. 28,139, Dec. 9, 1896.

"A plate . . . . composed of lead strip or ribbon, having, preferably, corrugated surfaces, or surfaces of like character mechanically produced, with several thicknesses of the strip or ribbon placed vertically together, face towards face, and their upper ends joined with a bar or suspender by soldering or burning, or by casting the bar or suspender thereto," &c. &c.—J. C. R.

Batteries, Secondary, Electric Accumulators [Perchloric Acid]: Impts. in the Manufacture of. L. Lucas, Hagen, Germany. Eng. Pat. 28,511, Dec. 12, 1896.

"The process of forming accumulator plates . . . consisting in employing therefor—instead of chloric acid, hydrochloric acid, and the like—the more stable perchloric acid or its salts . . ." "The perchlorate is added in such proportion to the sulphuric acid that the solution shall contain about 5 to 10 grms. of perchloric acid liberated by the sulphuric acid (from perchlorate of potassium, sodium, or calcium)."—J. C. R.

Electric Accumulators, Impts. in the Manufacture of. [Grid.] F. Pescetto, Turin, Italy. Eng. Pat. 18,430, Aug. 7, 1897.

The holes in the grid are jagged three times, at 90° apart, on both sides of the plate, so that the metal projects inwards over the openings, which are then filled with a paste composed of minium or litharge, mixed with 5 per cent. to 10 per cent. of ulmate of ulmin, preferably prepared according to Eng. Pat. 13,092, 1887. The ulmate,

which should be of the consistency of putty, must not exceed "1·5 degrees of acidity."—G. H. R.

Electrolytic Apparatus [*Tanks, &c.*], *Impts. in.* H. S. Jones, London. From E. Balbach, jun., Newark, New Jersey, U.S.A. Eng. Pat. 19,035, Aug. 17, 1897.

1. "The combination of a cathode case, composed of a trough having its bottom divided into two portions, one of which is horizontal and the other of which is inclined, so as to slope down to and connect with the horizontal portion, throughout its length, and provided with a cathode plate; and an anode suspended therein, arranged to cover the horizontal portion of the bottom only." The object is to construct apparatus so "that the metals which it is desired to separate can be easily and readily removed from the bath, without in any way interfering with or stopping the electrolytic separation."—J. C. R.

Electric Furnaces, Impts. in. [*Fixed Arc.*] W. S. Horry, Michigan, U.S.A. Eng. Pat. 22,521, Oct. 1, 1897.

See under II., *page* 1006.

(*B.*)—ELECTRO-METALLURGY.

Separation of Metals, Studies on the Electrolytic. A. Waller. Zeits. f. Elektrochem. 1897, **4**, [10], 24.

See under XXIII., *page* 1042.

PATENTS.

Magnesium and its Alloys, Impts. for the Manufacture of certain Metals and Alloys, especially for the Manufacture of. A. Le Redotte, Paris, France. Eng. Pat. 21,976, Oct. 3, 1896.

In this process for the electrolytic production of magnesium, lithium, chromium, and their alloys, which is specially described with reference to the first-named metal, special care must be taken in the drying and fusion of the magnesium chloride, which is assisted by the use of hydrate of ammonium in a manner fully described. The fused chloride is run into the electrolytic vessel, which is provided with a luted cover, through which a movable anode passes, the cathode being fixed to the bottom of the vessel or formed by a carbon lining. An arc is struck between the two electrodes in the body of the electrolyte, and the production of magnesium is carried on both by electrolytic action and by the dissociation produced by the high temperature of the arc. To prevent the reunion of the gases with the metal, they are carried off through a flue formed by the interspace between the anode and a surrounding sheath of some refractory substance, preferably carbon. This sheath is perforated to allow of the introduction of an inert gas, such as nitrogen, to sweep away the gas evolved in the electrolytic process; and means are provided for the introduction of fresh salts, and the removal of the molten metal, which is kept surrounded by an inert atmosphere. —G. H. R.

Calcium Carbide, Manufacturing [*Electrolytically*], *and for the Reduction of Metals from their Oxides; An Improved Method and Apparatus more especially intended for.* H. Maxim, London. Eng. Pat. 25,611, Nov. 13, 1896.

The claims relate to (1) "Establishing electrical connection between an electric incandescence heating conductor and an electrode connected with one of the poles of a source of electrical energy, whereby a yielding or sliding contact is provided." (2) "An electric incandescence heating conductor extending through and capable of longitudinal movement in a tube or other suitable material for establishing electrical connection between such conductor and the said tube or receptacle, or other suitable electrode or terminal, whilst permitting the required longitudinal movement of the said conductor." (7) "The process of making calcium carbide, which consists in fusing the carbide-forming material by the heat of combustion, and converting the said material into carbide by the aid of electricity." (8) "The superheating of previously heated fuel-gas and air by the combustion of a portion of such heated or superheated gases, &c." (9) "In the manufacture of calcium carbide, the herein described process, consisting in heating separate currents of air and fuel-gas [water-gas, &c.] by passing them through heating furnaces, in the first of which fuel-gas is burned, and in the second or succeeding furnace, they are superheated by the combustion of a portion of the highly heated or superheated air and fuel-gas, and then burning the superheated gases or the remainder thereof for heating the carbide-forming materials."—J. C. R.

Zinc Chloride, Impts. in Apparatus for use in Electrolysis, more especially intended for use in the Electrolysis of. [*Rotary Cathodes.*] F. Hurter and the United Alkali Co., Ltd., Liverpool. Eng. Pat. 28,764, Dec. 15, 1896.

The apparatus consists of a tank divided by means of frames into a number of compartments alternately open and closed at the top, the former containing the cathodes and the latter the anodes, which are separated from them by suitable diaphragms. The cathodes consist of vertical cylinders, preferably of iron, provided with pivots at each end, resting in sockets, and so arranged as to be capable of turning round, or partially round, a vertical axis. Connection is made with the carbon anodes through the sides of the tank, and openings are provided in the respective compartments for the circulation of the electrolyte and the removal of the gases. The deposit is stripped from the cathodes by submerging them in a molten bath of the same metal.—G. H. R.

Cathodes [*Plumbago*], *Impts. in or relating to, employed in Galvanoplastic or Electrotyping Processes.* A. Julien, Paris, France. Eng. Pat. 88, Jan. 1, 1897.

In place of oil or fat it is proposed to employ essence of turpentine, benzine, "petroleum essence," petroleum, alcohol, xylene, or the like, as an admixture to plumbago for the coating of cathode surfaces, preferably in the proportion of 100 to 250 grms. of plumbago to 1 kilo. of the "essence of turpentine." This suffices for deposits on plane or semi-spherical surfaces, but for round or spherical surfaces it is better to add to the mixture a proportion of resin (colophony), gum arabic, yellow wax, paraffin, Canada balsam, or the like, according to the hardness and adhesion required.—G. H. R.

Batteries, Secondary [*Cadmium, &c.*]; *Impts. in.* C. E. Lee, Chicago, U.S.A. Eng. Pat. 10,439, April 27, 1897.

A cell is described with a "porous cup" containing the anode of lead surrounded with the active material (lead oxide), and a cathode of amalgamated copper gauze contained in an outer jar, the whole arranged concentrically. The electrolyte employed is a [" neutral salt without any associated acid "] solution of cadmium sulphate "of 27° to 30° B." "Metallic cadmium is produced, and deposited on the negative element evenly."—J. C. R.

XII.—FATS, OILS, AND SOAP.

Earth-Nut Oil. S. P. Sadtler. Amer. J. Pharm. 1897, **69**, 490—492; Analyst, 1897, [260], 284.

The author states that when earth-nut oil is freed from the acid which is present in the raw state, it does not tend to become rancid so readily as olive oil, and he recommends its use in pharmacy, as the products are equally good, whilst the cost is much less. The bulk of the Castile soap manufactured in Marseilles is stated to be made from African earth-nut oil.

The cold-pressed oil is of a pale yellow colour. About 38 per cent. is obtained from Virginian earth-nuts by the ordinary treatment, and nearly 10 per cent. more by a second hot-pressing.

The following table gives the results of the analyses of oil derived from various sources :—

—	Oil from Virginian Nuts.	Oil from Spanish Nuts.	Oil from African Nuts.	Oil from Pondicheri.	Commercial Oil.
Specific gravity at 15° C.	0·917	0·9175	0·911	0·920	0·9209
Saponification value.	192·53	190·68	194·0	193·1	192·1
Iodine value	91·75	94·17	85·6	95·0	98·4
Hehner value...	94·87	95·31	..	..	95·86
Reichert - Meissl value.	0·48	1·60	..	..	..
Free acid as oleic, per cent.	0·55	0·79	0·62	..	6·20
Cold test of the oil.	+ 3° C.	+ 3° C.	+ 2° C.	..	+ 10° C.
Maumené test ..	56·75° C.	..	..	49° C.	45·5° C.
Melting point of fatty acids.	29° C.	34° C.	30° C.	29° C.	28° C.
Solidifying point	27·5° C.	32·5° C.	29° C.	25° C.	25° C.

—A. S.

Cerasin Industry, Notes on the. B. Lach. Chem. Zeit. 1897, **21**, [88], 916—917.

Among recent improvements, the introduction of an emulsifier is of special interest. This consists essentially of two flat plates revolving in opposite directions, and between which the hot ozokerite, mixed with the requisite quantity of acid, is introduced by means of a central opening in the axis of the upper plate. It is stated that a more thorough admixture can be effected in this way than by any other means, and that there is a considerable saving of acid and an accelerated output. Many attempts have been made to discard sulphuric acid altogether in the preparation of cerasin, but these have speedily been abandoned. By extracting the ozokerite with benzene after treatment with bleaching powder, lycopodium powder, and magnesium silicate, there is an increased yield (up to 96 per cent.), but the product is inferior, being greasy and often having an unpleasant smell. A method of increasing the yield, but little known, consists in adding to the ozokerite from 5 to 10 per cent. of a very heavy mineral oil. This gives a more wax-like character to the cerasin, but the addition is objectionable when a white product is required. Attempts to extract the filter-press residue in the filter-press itself, instead of in an extractor, have not given satisfactory results, owing to the following disadvantages :—Loss of solvent, tediousness, and danger of manipulation, and the necessity of having filter-presses of special construction.

The ukase forbidding the use of cerasin candles in the churches in Russia has been repealed, to the great advantage of the Russian cerasin industry. Austria has now to take a second place to Germany in the export trade in cerasin, owing largely to the Austrian Land Bank monopolising the sale of ozokerite and considerably raising its price.—C. A. M.

Textiles, Soaps for. Leipziger Färber- u. Zeugdr. Zeit. **46**, [11], 491—492.

The author discusses the different purposes for which soap is used, and points out that each process requires a soap specially adapted for the purpose ; and hence a soap that may be excellent for, say, milling, might be unsuitable for printing or scouring. This difference in the behaviour of soaps is ascribed to the relative proportions of fatty acids and alkali therein. In a good, hard soap for printing purposes there should be 65 per cent. of fatty acids to 6 per cent. of soda : for dyeing purposes an olein soap, composed of 61 per cent. of fatty acids to 7·5 per cent. of soda, is recommended.

For silk-dyeing, a soap composed of 65 per cent. of fatty acids to 8 per cent. of soda should be used, some preferring nearly as much as 10 per cent. of soda : and for wool-scouring, a proportion of 61 per cent. of acids to 9 per cent. of soda. The same rules apply to soft soaps.—I. S.

Cotton-Seed Oil, A Characteristic Reaction of. G. Halphen. J. Pharm. et de Chim. 1897, **6**, [9], 390.

See under XXIII., *page* 1045.

Sesame Oil, Some Substances in, and their Relations to the Characteristic Colour Reaction of the Oil. V. Villavecchia and G. Fabris. Ann. del. Lab. Chim. Centr. delle Gabelle, **3**, 13—26 ; Chem. Centr. 1897, **2**, [11], 772.

See under XXIII., *page* 1015.

Unsaponifiable Oil in Greases with a Lime Base, Determination of. H. Bailey. Chem. News, 1897, **76**, 174.

See under XXIII., *page* 1017.

Oils and Fats, Animal and Vegetable ; Purity of Commercial. D. Holde. Chem. Rev. Fett- u Harz-Ind. **4** [20], 271.

See under XXIII., *page* 1016.

Linseed Oil, Amsel's "Water Reaction" for, &c. W. Lippert. Zeits. angew. Chem. 1897, [20], 655.

See under XXIII., *page* 1015.

Fatty Acids, Volatile ; Production of, from Wool Washings. A. and P. Buisine. Comptes Rend. 1897, **125**. [20], 777.

See under V., *page* 1012.

Beeswax, Examination of. K. Dieterich. Chem. Rev. Fett- u. Harz-Ind. **4**, [19], 259.

See under XXIII., *page* 1017.

PATENTS.

Oils, Crude : Impts. in the Method of and Apparatus for Brightening. W. R. Harrison and E. Stephenson, both of Hull. Eng. Pat. 26,097, Nov. 18, 1896.

Essentially, the process consists in breaking up the oil into minute particles either by passing it through a perforated coil or rose on to a revolving cone or cones, or by passing it through a screen or screens, or by means of a combination of these. Claim is also made for a special apparatus for thus aërating and brightening the oil.

—C. A. M.

Soap, Dry, or Soap Powder ; Impts. in the Manufacture and Production of. W. W. Wright and The United Alkali Company, Ltd., Liverpool. Eng. Pat. 27,906. Dec. 7, 1896.

"Roaster Ash" (crude sodium bicarbonate freed from ammonia) is mixed with ordinary soap and water, and the mixture dried by the addition of a further quantity of roaster ash. A good dry soap or soap powder is said to be produced by employing, for every 20 parts by weight of soap, from 60 to 65 parts of roaster ash and from 10 to 15 parts of water.—C. A. M.

Soap, Resinous : Impts. in the Manufacture of, and Apparatus therefor. F. Arledter, Perlen, near Luzern, Switzerland. Eng. Pat. 19,129, Aug. 23, 1897.

The lather ascending in the resin boiler is subjected to the continuous action of a stirring apparatus fitted with cutting knives or planes, whilst the particles of resin ascending with the lather are melted by means of slotted hot metallic plates. The boiling process is thus shortened, owing to the expulsion of the generated gases, and a resinous soap is obtained containing a large quantity of liberated resin. Claim is also made for the employment of two varieties of apparatus in which this process may be carried out.

—C. A. M.

XIII.—PIGMENTS, PAINTS ; RESINS, VARNISHES ; INDIA-RUBBER, Etc.

(A.)—PIGMENTS, PAINTS.

Painted Ironwork, Rusting of. E. Simon. Dingler's Polyt. J. 1897, **305**, 285.

It is usually believed that the formation of rust on the surface of iron which is supposed to be protected by a layer of paint, is due to the passage of moisture and carbonic

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acid through minute fissures in the oily covering, that owe their origin to the different rates of expansion under the influence of heat, possessed by the metal and the dried linseed oil. In order to test this point, the author has prepared a number of detached paint skins by coating gelatinised glass with different kinds of paint, and when dry stripping the films from the support after softening the gelatin substratum by immersion in cold water. After experiments with sea-water, &c., he finds that such skins, though absolutely free from distinct holes, are hygroscopic, and when swollen by damp, are readily permeated by water and gases.

Instead, therefore, of two coats of an oil paint forming a satisfactory protection for exposed ironwork, at least three or four coats should be applied; and, moreover, provided the material adheres well to the iron and possesses good covering power, that paint is the most efficacious which contains the highest percentage of varnish in its composition, and which accordingly allows the largest amount of oil to be applied to the unit of area.—F. H. L.

Driers and Varnishes. M. Weger. Chem. Rev. Fett- u. Harz-Ind. **4**, [21], 285—289. (See also this Journal, 1897, 922—923.)

The only effective driers hitherto discovered for increasing the siccative properties of drying oils are all compounds of either lead or manganese, the calcium preparations sometimes sold for this purpose having no action beyond making the varnishes dry harder, whilst zinc salts merely improve the colour, and the value of copper compounds is confined to the prevention of rust and "fouling" on ships' bottoms.

From experiments made by the author, it appears to be practically immaterial—so far as the rate of drying is concerned—whether the driers of the older class or the so-called "soluble driers" are used, provided the quantity of metal *dissolved by the oil* is the same in either case.

The chief points of advantage offered by the use of the "soluble" driers are: prevention of sediment, lightness of colour, and reduced cost and danger of production, the three latter being due, it is said, to the lower temperature required in the preparation of the varnish.—C. S.

PATENT.

Non-corrosive, Preserving, and Fire-resisting Paint, Improved. "The Non-corrosive, Preserving, and Fire-resisting Paint Co., Ltd.," F. White, and J. Thomson, Christchurch, New Zealand. Eng. Pat. 23,294, Oct. 20, 1896.

As a fire-resisting composition for use on wood, 8 oz. of linseed oil are bodied up with sufficient flake white or other pigment to yield a paint of normal consistency, and with it are mixed sulphuric acid, $\frac{1}{4}$ oz.; turpentine, $\frac{1}{2}$ oz.; and ammonia, $\frac{1}{4}$ oz. If the material is to be applied to ships' bottoms, &c., 1 dwt. each of chloride of copper, chloride of zinc, nitrate of mercury, and cyanide of potassium are also added.—F. H. L.

(*B.*)—RESINS, VARNISHES.

Resins, Exudation. M. Bamberger and A. Landsiedl. Monatsh. für Chem. 1897, **18**, 481—509.

The resin from fir trees, treated in alcohol solution with caustic potash, yields a potassium derivative, from the aqueous solution of which, acids precipitate a phenol-like substance, readily crystallisable, called by the authors *pinoresinol*, and having the formula $C_{17}H_{12}O_2.(OCH_3)_2.(OH)_2$. The diacetyl, dimethyl, and diethyl derivatives of this body were prepared. Nitric acid acts on pinoresinol, producing a body which crystallises from petroleum spirit in large golden-yellow crystals, which stain silk or the skin an intense yellow. From these crystals were prepared a potassium salt in red needles, a red barium salt, and an amido derivative. Analysis showed the nitro-compound to be dinitroguaiacol, $C_6H_2.OH.OCH_3.(NO_2)_2$. Bromine gives with pinoresinol the dibromide of a dibromo-derivative.

Pine-tree resin gives a pinoresinol identical with that from fir trees. Ether dissolves about 80 per cent. of the crude pine-resin; saponification shows this portion to consist of the pinoresinol esters of abietic and paracumaric acids, the former in the greater proportion; and by the action of hydrochloric acid gas on the mixed ethereal solutions of pinoresinol and abietic acid, the soluble part of the pine-resin was artificially produced. The portion insoluble in ether has all the properties of a tannol: from it was isolated *pinoresinotannol*, $C_{30}H_{30}O_6(OCH_3)_2$.

Larch-resin yielded a resinol similar in character to, but not identical with, pinoresinol; this *lariciresinol* has the formula $C_{14}H_{10}(OCH_3)_2(OH)_3$. A triacetyl derivative was prepared by the authors, but the substance is still under investigation.—J. T. D.

Varnish, "Unboiled" Linseed. W. Lippert. Chem. Zeit. 1897, **21**, 775.

The author does not approve of H. Amsel's expression "unboiled" (this Journal, 1897, 924) as applied to varnishes made by dissolving metallic resinates or linoleates in linseed oil, for the temperature necessary to effect solution is only some 70° or 80° lower than that required when the corresponding oxides are employed. The term is only suitable for such drying oils as are prepared (usually by the painter himself) by mixing raw linseed oil with a liquid drier, that is to say, with a ready-made solution of a resinate or a linoleate in oil of turpentine. When it is desired to differentiate between the two former varieties, instead of the words "boiled" and "unboiled," it will be better to speak of "oxide" and "resinate" varnishes respectively.

Objections to Amsel's "water reaction" have already been pointed out (this Journal, 1897, 702); Lippert now quotes further instances which go to prove its utter untrustworthiness. Amsel has also recently stated that when linseed oil is boiled without driers till it attains a syrup-like consistency, it loses its drying power. Products like this certainly do not dry so quickly as ordinary boiled oil, simply because they contain no lead or manganese; but they undoubtedly do dry, or they would not be used by painters, and they yield particularly elastic films.

Weger suggests (this Journal, 1897, 922—923) that the ready solubility of small quantities of certain compounds of manganese, *e.g.*, the acetate and the borate, at comparatively low temperatures (120° C.) is due to the ease with which these bodies are decomposed by the free fatty acid of the oil into manganese linoleate and free acetic or boric acid. If this view be correct, as it probably is, the amount of metal capable of solution under the conditions referred to must be strictly dependent on the acid number of the oil; but it is invariably sufficient to convert the linseed oil into a quick-drying varnish. At higher temperatures (220° to 230°), however, a direct reaction or saponification takes place between the metallic oxides and the fatty glycerides; and the amount of metal dissolved is only limited by the proportion of fatty acid set free.

—F. H. L.

Linseed Oil, Amsel's "Water Reaction" for, &c. W. Lippert. Zeits. angew. Chem. 1897, [20], 655.

See under XXIII., *page* 1045.

PATENTS.

Varnish [Copal], Impts. in the Manufacture of. J. E. and C. S. Bedford, Leeds. Eng. Pat. 19,736, Aug. 27, 1897.

Kauri copal is rather difficult to dissolve either in linseed oil or in turpentine, and the usual practice hitherto has been to fuse the resin in order to increase its solubility. This process has the disadvantage of spoiling its colour, and of causing the formation of some brown products of decomposition. The inventors find that the fatty acids of linseed or castor oil are very efficient solvents; and to prepare a varnish suitable for floorcloth, they heat 200 lb. of the copal in a jacketed pan to 125° C. for four or six hours with 100 lb. of linseed acids, finally adding 700 lb. of turpentine, 150 of boiled oil, 200 of rosin spirit, and 100 of light petroleum.—F. H. L.

Enamel, Patent-Leather Harness; Impts. in. W. Walters and A. Webb, London. Eng. Pat. 20,902, Sept. 11, 1897.

This consists of asphaltum, 1 lb.; caoutchouc, ¼ oz.; mineral naphtha, 1 oz.; and turpentine, 1 gallon. The varnish is applied to old patent-leather harness with a brush, in order to give it a brilliant polish equal to that of the material when new.—F. H. L.

Nitrocellulose, Impts. in Treating for the Purpose of Coating or Impregnating Textile and other Fabrics. D. M. Sutherland, Sunbury-on-Thames, and W. McLaren, London. Eng. Pat. 28,613, Sept. 14 1897.

See under V., *page* 1013.

(C.)—INDIA-RUBBER, &c.

Sulphur Chloride, Analysis of. G. A. Le Roy. Rev. de Chim. Indust. 1897, **8**, [94], 291.

See under XXIII., *page* 1040.

PATENT.

Rubber Compounds and Fabrics, Impts. in the Manufacture of. F. H. Smith and C. Macintosh and Co., Manchester. Eng. Pat. 26,695, Nov. 25, 1896.

India-rubber is incorporated with cellulose in a pulp form, and the mass is then rolled and pressed into any desired shape.

A compound fabric is made by spreading or impregnating cloth with the above material. Viscose (this Journal, 1893, 516) is, according to the patentees, the "form of cellulose" to which their invention is particularly applicable. —H. I.

XIV.—TANNING, LEATHER, GLUE, SIZE.

Tan Liquors, Fermentation Phenomena in. F. Andreasch. Imp. Research Laboratory, Vienna. Der Gerber, **23**, [544], 111, [546], 135.

The Behaviour of the Tannin.—It has been shown, in the researches on the sources of carbon, that neither yeasts nor bacteria ferment the tannin. Some moulds, *e.g.*, Penicillium Glaucum, decompose the tannin, and especially in those materials which contain only a small amount of non-tannin in comparison with their tannin (quebracho and knoppern liquors and extracts).

This is because the acid-producing micro-organisms do not find in these materials sufficient suitable carbon compounds to enable them to compete with the moulds. The latter are able to utilise the carbon of the tannin after all the non-tannin is used up. The most favourable temperature, 25°—35 C., for the rapid fermentation of tannin by Penicillium is seldom met with in practice. The only other changes the tannin undergoes are oxidation phenomena brought about by the oxygen of the air. The most important of these in bark tannins is the formation of phlobaphenes. The precipitation of these bodies is hastened by the acidity of the liquors.

In an experiment to determine this action, sterile air was passed in equal quantities through three flasks, *a*, *b*, *c*, each containing 300 c.c. of pine-bark infusion: *b* received an addition of 2 grms. of acetic acid; *c*, 3 grms. of lactic acid. The tannin before the experiment was 0·2922 grm. per 100 c.c. After the passage of the air it was:—

In *a*	0·2137 grm. per 100 c.c.
„ *b*	0·2000 „ „
„ *c*	0·1987 „ „

All the flasks were free from germs, and the amount of non-tannin was unchanged. Pine, oak, and hemlock barks contain the most easily oxidisable tannins. This precipitation of tannin appears to be analogous to the destruction of the colouring matter of red wine exposed to air and light. The influence of the tannin on the micro-organisms is merely to render their action slower; it never arrests it while any fermentable substance is present; it even assists acetic bacteria by absorbing oxygen, which is an absolute necessity for these organisms.

The size of the cells of bacteria is smaller in tannin solutions than in those free from tannin, but yeasts and moulds are not affected, and no alteration either in the size or form of yeast cells can be detected.

In conclusion, a few of the more important results of the research may be briefly summarised.

1. Putrefactive bacteria from the hides, bat s, &c., accommodate themselves to the acid reaction of tan liquors; they dissolve certain nitrogenous constituents of the hide, and thereby furnish the chief nutriment for the more specific acid-producing bacteria. In liquors which are in use, the production of acid is proportional to the hide substance present, provided sufficient quantity of carbohydrates are present.

2. Acetic acid, which in fresh tan liquors is the chief acid, is always formed by two separate processes. (1) the production of alcohol by yeasts from the glucoses of the non-tannins, and (2) the fermentation of the alcohol by acetic bacteria. In tan liquors it is never formed directly from carbohydrates.

3. Lactic acid is produced by several species of bacteria both from the sugars and other carbohydrates of tan liquors, and from the sugars alone by a yeast. A good supply of nitrogenous nutriment is necessary for its production, the greater part of which is furnished by the hides.

4. Butyric acid occurs in traces only in *sound* tan liquors.

A practical application of the results, is the preparation of sour liquors from materials poor in fermentable matter, by addition of glucose and alcoholic yeasts, or by direct addition of alcohol, which is quickly changed to acetic acid. A proper lactic fermentation is best induced by the sowing of the lactic yeast in the liquors.

The behaviour of the tannins has just been considered, and need not be repeated.

If the acids produced in the liquors by these various fermentations have not the same importance for the tanner as formerly, yet the fermentations and decompositions themselves are of great importance, and a more exact knowledge of them is certainly of interest for him. The author gives the above work as a contribution to a better knowledge of these processes.—J. T. W.

Leather Dressing. Der Gerber, 1897, 109, 121, 147, 159, 171, 175, 207.

In greasing leather with mixtures of dégras and tallow, it is frequently found that the same mixture is applied with but varying success to different batches of hide. Especially is this known to occur when the mixture is applied in a warm state to the skins. This frequently arises from the mixture having been insufficiently stirred during cooling, so that the solid fats crystallise out; or in the properly made mixture having been again heated above its melting point before application, and allowed to cool slowly on the leather. In this case the mixture is no longer homogeneous, but consists of a liquid oily portion and crystalline aggregates of palmitin and stearin. It is therefore obvious that, particularly as regards the tallow, the best article to use is not the kind generally quoted as the best, *i.e.*, the fatty acids of which melt at about 48° C., but the medium and lower qualities. Great care is also required in the selection of the seal or fish oil, the most oxidisable brands of which are by no means the most suitable for the production of dégras, since they are liable to suffer from over-oxidation and then only yield a very poor quality of dégras, containing large proportions of pitchy matter instead of the valuable "moëllon." It is, however, erroneous to ascribe the specific action of dégras exclusively to the "moëllon" (surplus oils removed from softened skins by expression) contained in it. But, on the other hand, the remarkable property of "moëllon" in uniting so firmly with the tanned animal fibre as to be no longer removed from it by any of the solvents of grease, appears to be almost unknown. Oxidised (blown) seal or fish oils, which are extensively offered as dégras substitutes, behave very differently from the real article, which they only resemble in their property of emulsifying with water. On examination under the microscope, it is, however, readily seen that the globules of water in the blown oil emulsion are at least ten times the size of those in the

dégras. The greatest fault of these blown oils is their liability to "spue," and form a resinous coating on the surface of the leather. On the other hand, they possess, nevertheless, the distinct advantage of a greater initial consistency than dégras, and consequently they can be used with a smaller admixture of tallow. Another constituent of all dégras of commerce is wool grease, which, especially in its purified neutral form, is very well suited for the purpose of dressing leather, not by itself, but in admixture with either the seal or fish oils, or with dégras. With particular advantage it is employed to replace, either partly or entirely, the tallow in the grease mixtures used in dressing leather. For practical purposes a distinction is made between light and heavy fish oils, and of the two, the former are the less suitable. Of the heavy fish oils, those are the most valuable for the tanners' use which contain least of palmitin. The colour of these oils is comparatively unimportant. Also mineral greases are employed in the dressing of leather. They act in a particular way, and besides being used to regulate the properties of the other greases, they chiefly serve the purpose of imparting additional weight to the leather. The light mineral oils are generally used in dressing mixtures and dégras to render the harder greases present more liquid. Solid mineral greases are frequently used as tallow substitutes, over which they possess the advantage of not being crystallisable. Rosin oil has acquired an ill repute amongst tanners, although there is no doubt that it can be employed to very great advantage.

—C. O. W.

Catechu. Textile Colorist, 1897, **19**, 298.

There are principally four kinds of catechu, distinguished in commerce according to their origin, *viz.*:—

1. *Bombay catechu*, which is the dried extract from the heart-wood and fruits of *Areca catechu*. It occurs in large irregular lumps of dark brown colour, which have a glossy fracture, and are nearly completely soluble in hot water.

2. *Bengal catechu*, from the unripe pods of *Acacia* and *Mimosa catechu*. It is of a somewhat brighter colour and less soluble than Bombay catechu. Both kinds are imported from India, Java, Pegu, and Singapore.

3. *Gambier catechu*, ordinarily termed "gambier," is the extract from *Uncaria Gambier*. The commercial article occurs in cubes, covered with rush mats, and soft inside. It is imported from Batavia and Singapore, and is earthy and porous, having a dull fracture.

4. *Kino*, which is a product of *Pterocarpus marsupium*, has a dark red-brown colour and very glossy fracture.

All varieties of catechu are only slightly soluble in cold water, but almost completely soluble in hot water. With gelatin or sulphuric acid they give brown precipitates; with salts of iron, green ones. Starch, sand, clay, and blood are common adulterations. Good catechu should not give more than 5 per cent. of ash, nor contain more than 12 per cent. of substance insoluble in boiling alcohol.—I. S.

Sodium Sulphide used in Leather Dressing, Note on the Analysis of the. F. Jean. Ann. de Chim. Analyt. **2**, [18], 341: Analyst, 1897, [260], 306.

See under XXIII., *page* 1041.

Sumac, Adulteration of. W. Eitner. Der Gerber, 1897, **33**, 161.

See under XXIII., *page* 1044.

PATENTS.

Tanning of Hides, Skins, and the like; Impts. in and connected with. J. Y. McQuinn, Runcorn, and E. Caldwell, Warrington. Eng. Pat. 23,742, Oct. 26, 1896.

In the process described, hides and the like are immersed, after depilation and cleansing, in a bath of dilute sulphuric acid for from three to five minutes, according to the strength of the bath, and the thickness and nature of the material to be tanned. For ordinary hides the bath is made up of 2 vols. of rectified sulphuric acid to 8 vols. of water. The hides being now rolled or otherwise partially dried, are placed in a tannage bath composed of or containing an alkaline bichromate, canella, aloes, and sulphuric acid in water, in which they remain for from less than half an hour to over six hours. This completes the tannage. The pits must be constructed of such material as stone or concrete. The object of the invention is economy of labour and the obtaining of results in less time than at present.

—J. G. P.

Tanning of Hides and Skins, Improved Means to be used in the. E. J. Thibaut, London. Eng. Pat. 18,332, Aug. 6, 1897.

Hides and skins are suspended apart from each other, and so maintained within a drum provided with liquid-tight doors for introducing and removing. The drum contains the tanning agent, and is slowly rotated. The suspension and rotation are so related that during each rotation the hides are completely dipped into and withdrawn from the tanning liquid. The drum is mounted on standards, and one of its journals is hollow, and arranged so as to admit the tanning agent into the drum, the other journal being fitted with gear so as to secure relatively slow rotation. Means are provided for running off the tanning agent.—J. G. P.

Tanning of Hides and Skins, Improved Means to be used in the. E. J. Thibaut, London. Eng. Pat. 20,354, Sept. 4, 1897.

This invention differs from that above described (Eng. Pat. 18,332), in that the hides, though suspended within a drum, are not stretched. They are so suspended that, in the course of the rotation of the drum, they roll and rub in contact with each other; and while being raised from out of the tanning liquid and carried over, are separated and straightened out. The drum is here cylindrical, but essentially the same as that described in the preceding abstract.

—J. G. P.

Casein Glue, Liquid; Impts. in. H. Bolder, Charlottenburg. Eng. Pat. 19,661, Aug. 26, 1897.

To prepare this glue, 100 parts of dry casein are treated with a solution of two parts of borax to from 60 to 80 parts of water, the mixture being then heated nearly to boiling, kept at this temperature for about half an hour, and constantly stirred. Instead of borax solution, a solution of ammonia may be used, such that it will give a slight alkaline reaction. Starch may be added.—J. G. P.

Tanning Hides and Skins, An Improved Process for. J. Mossop, jun., and E. B. Garland, both of Cape Town. Eng. Pat. 20,125, Sept. 1, 1897.

This is a process of drum-tanning. For cleansing, after the usual treatment with lime, the hides are subjected, in a rotating drum, to the action of carbonic acid gas under pressure. After cleansing, the hides are placed in a rotating drum, containing a strong tanning liquor, and are kept rotating in the atmosphere of carbonic acid gas, either under pressure or not, until tannage is completed; this being effected in from 2 to 30 hours.—J. G. P.

Tanning of Hides, Skins, and the like, for the Production of Leather; Impts. in the, and in the Apparatus employed therein. E. H. Dewson, Massachusetts, U.S.A. Eng. Pat. 21,601, Sept. 21, 1897.

This is a method of drum-tanning. The drum has a number of compartments, and these are arranged around its axis of rotation. Each compartment is complete in itself, liquor-tight, and independent, with its own charging hole and cover, and carrying its own load of hides and liquor. The hides are always immersed in the liquor, and the drum always substantially in balance.—J. G. P.

XV.—MANURES, Etc.

Potato Scab, Prevention of, by Means of Formalin. Bulletin of the Agricult. Stat. of the Purdue University; through Nature, 1897, **56**, (1456), 502.

Experiments conducted with "seed" potatoes at the Agricultural Station of the Purdue University show that

formalin will practically free infected tubers from scab. The method employed is to soak the tubers for two hours in a solution of formalin of the proximate strength of 1 : 300, made by adding half a pint of "formalin" to 15 galls. of water. This solution is found to be as effective for the purpose as the dangerous and more troublesome solution of mercuric chloride. Seed material of apparently good quality, as well as that much affected with scab, benefited by this treatment. The same formalin solution may be used several times for successive lots of "seed" tubers.

—J. O. B.

Fermentation of Green Grass, Chemical and Bacteriological Investigation of. O. Emmerling. Ber. 1897, **30**, [14], 1869

See under XXIV., *page* 1051.

Phosphates, Valuation of, according to the Amount of Citrate-soluble Phosphoric Acid in. P. Wagner. Chem. Zeit. 1897, **21**, 905.

See under XXIII., *page* 1041.

XVI.—SUGAR, STARCH, GUM, Etc.

Sugar, Diffusion Juice, Conditions for obtaining Dense. B. Mittelmann. Bull. de l'Assoc. des Chim. 1897, **15**, [3], 205—208.

Although all are agreed as to the principle of the production of a diffusion juice of high density, the limit to which the exhaustion of beetroot pulp should be carried, is strongly contested. Karlson maintains that diffusion should be arrested as soon as a product is obtained not capable of furnishing crystallisable sugar, for, assuming the non-sugar in massecuite from such juice to be equally melassigenic with that in molasses, any further exhaustion would give a useless increase of uncrystallisable products and a diminution in the yield of crystalline sugar; the exhaustion should therefore not be pushed beyond 60 or 50, or, as an extreme limit, 40 degrees of purity. When the sugar recovered, however, is obtained free of duty, it will be difficult to resolve to abandon it, although in the form of molasses.

The best temperature for diffusion is always the lowest, as giving a juice of a higher degree of purity. A temperature of 70° C. gives excellent results with rich beetroots, but with those of lower quality, either the temperature must be raised to 75° or 78° C., or, preferably, a longer time of contact must be allowed, by increasing the number of diffusers to 14 or 16. This gives a juice at least one degree purer. It is very advantageous to mash with hot juice, so that the juice leaves at 30° to 35° C. The pulp undergoes a more thorough diffusion, the juice obtained is denser, and the purity is not inferior to that obtained by cold mashing. To secure uniform exhaustion of the pulp, a layer of water 20 to 30 cm. deep is introduced into the diffuser before filling with fresh pulp, and cross pipes at each third of the height are arranged to cause the pulp to distribute itself uniformly.—L. J. de W.

Invert Sugar, Formation of, during Diffusion. H. Pellet. Bull. de l'Assoc. des Chim. 1897, **15**, [3], 208.

When the quantity of reducing sugars in beetroot juice, obtained by rasping and pressing in the cold, is compared with that in diffusion juice, no increase is found during ordinary work, but rather a tendency to a diminution thereof. When a stoppage occurs in the diffusion process, reducing bodies are found in proportion to the length of time work is at a standstill, the principal effect being observed in the first diffusers, which have not been subjected to the higher temperature of the rest of the battery. That Claassen finds an increase of 0·10 per cent. of reducing bodies is attributed to the fact that he used a different method of analysis for the fresh pulp and the diffusion juice.—L. J. de W.

Washing Beetroots. Bull. de l'Assoc. des Chim. 1897, **15**, [3], 197—204.

Absorption of Water or Loss of Sugar in the Hydraulic Conveyors and Washers.—According to arrangement between grower and manufacturer, it is usual to add to the tare, 1 per cent. or 2 per cent., to allow for water absorbed and mechanically retained by the beetroots after washing; but as the operation of taring a sample, usually of 20 kilos., occupies less time than that of conveying and washing the beetroots on the large scale, Pellet has studied the question afresh.

The duration of contact of water and beetroots in the conveyor is relatively short. The speed of the water is 125 to 130 metres per minute, and in many installations the conduits do not exceed 150 to 250 metres in length, and as roots are thrown in throughout the greater part of this length, the average contact is much reduced. Fairly clean beetroots were plunged in water for about two minutes, then drained and weighed. The increase was 0·80 to 0·90 per cent. After wiping completely, the water absorbed was found to be only 0·10 to 0·30 per cent., or 0·60 to 0·70 per cent. of water mechanically retained. Even with α-naphthol, only traces of sugar were found in the water.

Allowing half an hour of contact between the water and the beetroots from their entry into the conveyor to the removal of the washed beets for weighing, it was found that 0·60 to 0·80 per cent. of water was absorbed and 0·20 to 0·25 mechanically retained, and the loss of sugar was 0·020 to 0·035 per cent.

A very sensible difference may thus be found between the richness of the beetroot trimmed and cleaned in the laboratory and that of fresh beetroot slices. The latter will contain 0·6 per cent. of earth, 1 per cent. of water absorbed, 1 per cent. of tops containing 7 per cent. of sugar, leaving 97·4 of beetroot at, say, 13 per cent. of sugar, giving a total of 12·72. From this, 0·03 is to be deducted for loss of sugar in the washers. Thus, instead of 13 per cent. found in the laboratory, we should find only 12·69 per cent.

Mittelmann finds that the sugar lost in the wash water is insignificant, but when beetroots are touched with frost, and warm water must be used to loosen the adhering earth, the injured beetroots may lose 0·40 to 0·60 per cent. of sugar.

The average time occupied by beetroots passing through the washers in a factory observed by Guibal was 45 minutes. He therefore immersed carefully cleaned beetroots in water and examined a portion every 15 minutes for an hour. The rate of absorption of water was greatest during the first half-hour. In 45 minutes the water absorbed was 0·433 kilo. per 100 kilos. of beetroots, the sugar lost 0·101 kilo.—L. J. de W.

Beetroot, Reducing Sugars of. H. Pellet. Bull. de l'Assoc. des Chim. 1897, **15**, [3], 233—236.

On heating to 85° C. for a quarter of an hour, 100 c.c. of a mixture of 50 c.c. of Fehling or Violette's solution and 50 c.c. of a solution of sugar containing no glucose, there is no reduction, but if only a few decigrams of invert sugar be present, the whole of the corresponding copper is reduced. If the mixture contain reducing bodies other than glucose (invert sugar), the liquid filtered after heating to 85° C. and again heated on the water-bath for half an hour to an hour, gives increasing quantities of oxide of copper. It is possible in this way to estimate 0·08 to 0·09 of invert sugar in 100 grms. of molasses, and afterwards 0·70 to 0·80 of reducing bodies which have not at all the characters of invert sugar. Tested thus, the reducing bodies contained in beetroot and cane juice, show very great analogy with invert sugar, so that there is every reason to admit that they are identical with it.—L. J. de W.

Sugar Scums, Filtration and Washing of. Mittelmann. Bull. Assoc. Chim. 1897, **15**, [3], 223—224.

The froth which forms at the commencement of carbonating is nothing but hydro-sucrocarbonate of lime, and instead of using grease or steam as a preventive, the author proposes an excess of carbonic acid, which would immediately decompose this compound.

There are numerous causes which retard the filtration and washing of scums. Cholesterin, isolated by Herzfeld from a sample of scum and originally attributed to the

beetroot, but afterwards found to arise from the grease or oil used to prevent frothing,—even when present in very small quantity, may completely arrest filtration, owing to its property of undergoing a considerable increase in volume. When the juice is overheated during diffusion, pectin is formed, and this unites with iron contained in the lime to produce a flocculent and spongy mass of ferropectin, which obstructs the filter cloths.

Overburnt lime hydrates slowly, and is apt to leave unslaked particles, which, after carbonating, form sucrate of lime; the juice then filters with difficulty. With unripe or frozen beetroots, difficult filtration is avoided by working at a temperature of 70° or 72° C., or even lower, increasing the lime used, and carbonating rapidly with gas of 30 per cent., and heating afterwards to 90° C. Water of condensation often contains ammonium carbonate, and calcareous water contains bicarbonate of lime; these, when used for washing the scum, precipitate carbonate of lime in the pores of the filter cloths, obstructing them. The author prefers water from the triple effect for washing scums, as it precipitates nothing from the juice.—L. J. de W.

Molasses and Peat Fodder in Germany. J. Soc. Arts, 1897, [2343], 1149.

The use of molasses as a food for cattle and sheep has, during the past few years, been largely experimented on by agriculturists in Germany. The chief objection to the use of molasses is the purging effect which it exercises on cattle; but this, it is claimed, has been overcome by the admixture of a dust or mull obtained from moss-turf (peat). The moss-turf—from which the dust is obtained by means of a machine such as is used for making moss litter—is taken from the upper strata of high-lying peat moors, and consists largely of the dried but decomposed fibre of the *Sphagnum cuspidatum* and the *Eriophorum latifolium.* The action of the dust or mull appears to be one counteracting the severe purging caused by the molasses alone. It is recommended that the molasses peat-fodder, which is 50 per cent. cheaper than the best fat-producing food and yet equal in nourishment, should first be given in very small quantities, and the amount gradually increased.—A. S.

Sugar, On the Speed of Reduction of Ferric Alum by. J. H. Long. J. Amer. Chem. Soc. 1897, **19**, 683.

See under XXIV., *page* 1052.

Sugar, Alleged Formation of Humin from, by Oxidation with Potassium Permanganate. H. v. Feilitzen and B. Tollens. Ber. **30**, [17], 2581—2584.

Benni (Dissertation, Giessen, 1896; Zeits. für Naturwissenschaften, **69**), assuming that the formation of humin is due to an oxidation process, has sought to obtain this substance from sugar by means of permanganate. The authors, after investigating the matter, state that the brown precipitate produced by the oxidation of sugar with permanganate, consists mainly of manganese oxide and potash, and that no actual formation of humin takes place.—A. S.

Yeast found in the Sugar Juices of Refineries. Wochenschrift für Brau. 1897, **14**, 602—603.

This yeast (*Saccharomyces Zopfii*) was found by Zopf in beetroot juices together with *Leuconostoc Mesenterioïdes.* It is a small yeast, which forms spores like beer and wine yeasts. It can resist a moist heat of 67° C., and when dry, a temperature of 130°—135° C. It inverts cane sugar, and at high temperatures appears to secrete a peptonising ferment. Only cane sugar and glucose are readily fermented by it. Besides alcohol and carbonic acid, the products of fermentation include an organic acid, which was extracted by ether, but was not sufficient in quantity to be identified.
—A. L. S.

Starch, Products of the Saccharification of, by Diastase. P. Petit. Comptes Rend. 1897, **125**, [6], 355—357.

Starch is treated, at 70° C., with 1 per cent. of diastase, until, in about half an hour, a constant red coloration is given with iodine. The liquid is fermented by means of yeast, and the substance obtained, after purification with alcohol and ether, possesses a constant rotatory and reducing power, and a molecular weight of 428 is assigned to it, and the formula $(C_6H_{10}O_5)_3$. This dextrin is a white non-hygroscopic body, and is not capable of yielding osazones. When treated with boiling dilute hydrochloric acid, it is completely converted into glucose after three hours, but if the reaction be arrested after half an hour, and the product treated with phenylhydrazine and sodium acetate, two osazones are obtained, one being glucosazone and the other corresponding to a member of the cane-sugar group. It appears, therefore, that the dextrin yields on inversion at first glucose and a "diose," and the latter on further boiling furnishes two molecules of glucose.

Further Action of Diastase.—On further treatment at 50°—55° C. with 1 per cent. of diastase, the dextrin yields two substances, and by comparison of their respective rotatory and reducing power with that of the solution containing both, the author calculates that the yield of one substance is twice that of the other. The change occurring is therefore represented by the equation—

$$4(C_6H_{10}O_5)_3 + 4H_2O = 4C_{12}H_{22}O_{11} + (C_6H_{10}O_5)_4$$

—R. B. B.

Starches, Chemistry of the. C. J. Lintner. Chem. Zeit. 1897, **21**, 737—738 and 752—754.

The author has extended his previous researches (this Journal, 1894, 53) on the starches. A short account of the chemical nature of the dextrins is first given.

Isolation of the Dextrins.—The isolation of the dextrins has been greatly facilitated by the application of the cryoscopic and osazone tests, and the purity of a product may now be fairly determined by having recourse to these, together with polarisation, reduction, and iodine-reaction tests. Aqueous alcohol has, after many trials, proved itself the best medium for the isolation and separation of the dextrins. Alcoholic baryta and alcoholic milk of lime possess the disadvantage of tending to decompose them; moreover, when any considerable quantity of material is to be operated upon, as is always the case in such experiments, the use of these reagents is not admissible. The solubility of a dextrin is commonly the greater, the lower the molecular weight of the compound; but there is an unusually great mutual influence between them in this respect when they exist together in solution. Hence, it is necessary to pay great attention to the strength of the alcohol and to the concentration in each individual case. Greater dilution must be employed towards the end of the fractionation. Much depends upon the manner in which this process is conducted. If, finally, strong alcohol be added to the aqueous solution of the dextrin, the result is always far more unsatisfactory than if the hot dextrin solution, mixed with alcohol, be poured, with brisk agitation, into the hot alcohol of known quantity and strength, and the whole be then allowed to cool to the temperature of the room.—W. G. M.

Soluble Starch. A. Wroblewski. Ber. 1897, **30**, [14], 2108—2110.

The authors restrict the term "soluble starch" to the first decomposition product of starch, which gives a pure blue coloration with iodine and does not reduce Fehling's solution. Amylodextrin is a decomposition product of soluble starch; iodine colours it reddish-brown, and it somewhat reduces Fehling's solution.

The best method for preparing soluble starch is as follows:—Triturate 100 grms. of the best rice starch with small quantities of 1 per cent. caustic potash solution and let stand 2—4 hours; repeat this process until the whole has attained a volume of 600—800 c.c. Heat on the water-bath with constant shaking until the jelly-like mass has become perfectly fluid, boil over the naked flame for 20—30 minutes, filter, add acetic acid to feeble acid reaction, precipitate with an equal volume of 95 per cent. alcohol, again dissolve, and precipitate. Finally dissolve in a little water, pour in a thin stream with violent stirring into a large volume of absolute alcohol, wash with absolute alcohol and ether, and dry *in vacuo.* The resulting snow-white substance contains very little ash, gives a pure blue with iodine, does not reduce Fehling's solution, and is soluble in water to

the extent of 4 per cent. The neutralisation by acetic acid is to facilitate washing only; the alkali is not combined with the starch, its action being merely catalytic.

Soluble starch is obtained by the first hydrolysis of starch, effected also by the action of diastase, acids, and water, but with these reagents, hydrolysis proceeds further. By long boiling with dilute potash, a substance is obtained with a very feeble reducing action on Fehling's solution.

—A. C. W.

Soluble Starch. W. Syniewski. Ber. 1897, **30**, [16], 2415—2418.

Compare Wroblewski (previous abstract). The author's method for preparing soluble starch is as follows:—Add 50 grms. of commercial sodium peroxide in small quantities at a time to 500 grms. of well-cooled water; bring into this solution 50 grms. of potato starch rubbed up with 500 grms. of water. In an hour, add alcohol of 95 per cent., dissolve the precipitated mass in cold water, and neutralise the cooled solution with acetic acid: again precipitate, dissolve, and acidify. On repeating this process several times, a substance is obtained containing only traces of ash. From the aqueous solution, on standing, a small yellow flocculent deposit separates, leaving a completely clear liquid. From this, the starch is precipitated by alcohol; it is then ground with alcohol, washed on a filter with anhydrous ether, and dried by warming *in vacuo*. The product was a snow-white amorphous body without taste or smell, giving, on analysis, figures corresponding with the formula $3C_6H_{10}O_5.H_2O$. It is soluble in cold water; solutions stronger than 12·5 per cent. could not be obtained at the ordinary temperature, but it appears to be soluble in hot water in every proportion. It gives a pure blue coloration with iodine, which property is not changed by prolonged heating on the water-bath, and it does not reduce Fehling's solution. At 20°, the specific rotation, $[\alpha]_D$, is 182°·66 for 2·5 per cent. solutions, and increases to 189°·5 at 12·5 per cent. strength. This increase in optical rotation with increasing concentration, it is alleged, accounts for the divergent results obtained by Nägeli, Brown and Morris, &c.

The yield of soluble starch obtained by the author's method is almost quantitative (90 per cent.), and the loss is due to purely mechanical causes.—A. C. W.

Mould [Starch, Dextrin, Maltose, &c.], A New: "Eurotipsis Gayoni." J. Laborde. Ann. Inst. Pasteur, 1897. 1—43.

This mould was first observed by the author on starch paste, where it resembled a growth of *Bacillus Prodigiosus*; however, when examined microscopically, it was seen to be a red mycelium.

This mould will grow on any of the media which commonly are used for growing moulds. It acts on starch, dextrin, and maltose, converting them into glucose. Invert sugar it destroys partly by fermentation and partly by oxidation, the glucose being acted on the more quickly.

Cane sugar and inulin need to be first inverted before the mould can act on them; it can, however, hydrolyse lactose.

Ethyl alcohol (6—8 per cent.) is oxidised by the mould to carbonic acid and water; methyl alcohol can also be decomposed, but propyl, butyl, and amyl alcohol are poisonous to it.—A. L. S.

Starch Products, The Exact Estimation of Total Carbohydrates in Acid Hydrolysed. G. W. Rolfe and W. A. Faxon. J. Amer. Chem. Soc. 1897, **19**, 698.

See under XXIII., *page* 1048.

Carbohydrates, The Identification of. B. Sjollema. Chem. Zeit. 1897, **21**, [74], 739.

See under XXIII., *page* 1044.

Phosphorus, A Plant Constituent containing, which on Decomposition yields Inosite. E. Winterstein. Ber. 1897, **30**, 2299.

See under XXIV., *page* 1051.

XVII.—BREWING, WINES, SPIRITS, Etc.

Wort and Beer, Determination of the Solid Matter of; and the Relation between this and the Specific Gravity. C. N. Rüber. Videnskabsselskabets Skrifter. I. Mathematisk-naturv. Klasse, 1897, No. 5.

(See this Journal, 1894, 756.) The author has already attacked this question, and he came to the conclusion that the results depended on the pressure and temperature at which the drying took place, something similar to dissociation occurring. He, however, has now found that if the temperature be below 80° C., the same constant result will be arrived at, however the drying be effected.

The tables in common use on the Continent for determining the extract of wort and beer are those of Balling and Schultz-Ostermann; Balling's table is based on the strength of cane-sugar solutions, and, of course, is well known not to give the true extract for worts; and as the alteration of volume on dilution is different for malt worts and cane-sugar solutions, the error between these tables varies for different strengths of solution. Schultz-Ostermann's table was obtained by direct determination of the solid matter contained in worts, but the author finds it to be about 5 per cent. too high.

When the solid matter was dried until constant at different temperatures below 80° C. and under various pressures, the same results were obtained, but at 100° decomposition took place and different results were obtained. The drying took two days at 80°; 80 days at ordinary pressure and ordinary temperatures, or 25°; and 10 days under reduced pressure at 55°.

As a result of the experiments on the relation between the specific gravity of worts and beer extracts and the solid matter contained in them, it was found that there was a certain relation between them. This is illustrated by the following example:—A wort of sp. gr. 1·04143 contained 10 per cent. of solid matter; a beer residue of the same sp. gr. contained 9·900 per cent. of solid matter: the ratio between these figures is 1·0100. This ratio the author calls the "quotient." If these two are now concentrated to a sp. gr. of 1·060, and the wort is found to contain 14·262 per cent. of extract, then the beer residue will contain 14·262 ÷ 1·010 = 14·119 per cent. of extract. It thus follows that if a table be prepared for some particular wort or beer residue, then, if the quotients are determined for other varieties of worts or beer residues, the table may be used for determining the extracts of these worts or beer residues.

The author has determined the value of this quotient as affected by the following conditions:—The amount of nitrogen. The composition of the brewing water. The boiling of the wort. The fermentation of the wort. The variation of the kilning temperatures. The different brewing methods (decoction or infusion). The addition of the hops.

The variation of the nitrogen percentages within the usual limits does not have any appreciable effects.

The variation in the composition of the water does, however, have a considerable effect, and the author's results are given in the following tables.

The Munich water contained 26 parts per 100,000 of solid matter, chiefly calcium carbonate. The Copenhagen water contained 34 parts per 100,000, chiefly calcium sulphate. The first artificial water contained 50 parts of calcium carbonate; and second, 50 parts of gypsum per 100,000.

The "quotient" is the extract as found by experiment, divided by the extract as found by the table.

For a fully fermented beer the quotient is 0·990; that is, a beer residue contains 1 per cent. less solid matter than a wort of the same specific gravity.

The kilning temperature, the brewing method (decoction or infusion), or the amount of hops, have no appreciable effect on the quotient.

The author has prepared a series of tables giving the amount of solid matter contained in worts, &c. of different specific gravities; but as this may be derived from the Schultz-Ostermann table by multiplying by 0·9481, it is unnecessary to reprint it here.

Description of Wort, &c.	Quotient.	$[\alpha]_D$	Per Cent. of Albuminoid.
Unboiled infusion wort with distilled water, Munich malt.	1·0035	118·0	..
The same with Copenhagen water.	1·0058	118·2	..
The same with Munich water.....	1·0046	118·5	..
Unboiled infusion wort, Prossnitz malt with distilled water.	0·9993	122·9	..
The same with Copenhagen water.	1·0033	124·2	..
The same with Munich water.....	1·0005	125·7	..
Boiled infusion wort with distilled water, Prossnitz malt.	0·9997	..	4·97
The same with Munich water.....	1·0044	..	4·56
Unboiled infusion wort with distilled water, Hungarian malt.	0·9969	..	4·48
The same boiled..................	0·9945	..	4·20
The same with Copenhagen water.	0·9993	..	3·77
The same with Munich water.....	0·9988	..	3·67
The same with the first artificial water.	1·0026	..	3·68
Boiled infusion wort, Danish malt, with distilled water.	0·9979	..	4·50
The same with the second artificial water.	1·0012	..	5·07

—A. L. S.

Alcoholic Fermentation without Yeast Cells. A. Stavenhagen. Ber. **30**, [16], 2422—2423.

E. Buchner found (this Journal, 1897, 156) that an extract from purified pressed yeast, even after sterilisation by passage through a Berkefeld filter, possessed the property of causing alcoholic fermentation in solutions of cane-sugar, dextrose, levulose, and maltose.

The author, both from his own experience and from the work of Severin-Jolin (Zeits. für Hygiene, **17**, 517), doubts the efficacy of the Berkefeld filter in removing all micro-organisms. On repeating Buchner's experiments with this difference, *viz.*, that a Kitasato filter (Zeits. für Hygiene, **10**, 269), which consists of a porcelain candle similar to the Chamberland filter, was employed instead of a Berkefeld filter, the author obtained an absolutely sterile extract which showed no signs of fermentation when introduced into a sterilised solution of cane-sugar.

Although these negative results may possibly be explained on the assumption put forward by Firotini (Zeits. für Hygiene, **4**, 288) that a porcelain filter does not allow all the dissolved substances to pass through, the author points out that Buchner's results, if true, are directly opposed to Pasteur's theory, and therefore require confirmation, the greatest care being taken at the same time to exclude all secondary actions.—J. S.

Peat, Amount of Pentosans, or Substances yielding Furfural, and other Carbohydrates in. H. von Feilitzen and B. Tollens. Ber. 1897, **30**, 2571—2576.

The amount of carbon, and of pentosans (by Krüger and Tollens' method, Zeits. angew. Chem. 1896, 33, 42, 194), were determined in a number of samples of peat taken at different depths, and also in the vegetation which produces peat. The following results show the percentage amounts in the dry substance, free from ash :—

———	Carbon.	Pentosans.
Calluna vulgaris....................	..	15·36
Sphagnum cuspidatum............	49·80	14·70
Peat litter........................	..	11·41
Speckener peat, 20—100 cm. deep..	51·08	12·75
„ „ 100—200 „ ..	53·52	9·75
„ „ 200—300 „ ..	58·66	5·87
Oc[illegible]ter „ 0 to 20 „ ..	55·47	6·26
„ „ 20— 60 „ ..	55·06	6·40
„ „ 60—100 „ ..	58·25	2·65
„ „ 100—120 „ ..	58·23	2·18
„ „ 180—200 „ ..	57·57	3·31
Worpedorfer „ 0 to 10 „ ..	54·32	8·08
„ „ 10— 38 „ ..	51·92	11·19
„ „ 38— 98 „ ..	52·17	10·80
„ „ 98—117 „ ..	54·28	8·72
„ „ 117—153 „ ..	57·41	4·08
„ „ 153—183 „ ..	57·19	4·23
„ „ 183—211 „ ..	58·82	2·65

Determinations of cellulose, by fusion with potash, gave the following results :—Sphagnum cuspidatum, 21·11 ; Speckener peat at depths of 20—100 and 100—200 cm., 15·20 and 6·87 per cent. respectively.

There is thus a decrease in the percentage of both pentosans and cellulose as the depth increases, but an increase in the percentage of carbon.

With regard to the hydrolysable carbohydrates, Sphagnum was digested with 1 per cent. sulphuric acid at 130°—135°, and the product examined according to the methods proposed by Tollens. The results indicated the presence of galactose, pentosans, and levulose. Dextrose and mannose could not be detected.

In a similar manner, peat-litter, when hydrolysed, was found to contain mannose, galactose, levulose, and pentosans, the pentosazone of which melted at 130°. The residue, after hydrolysis, was shown to contain, most probably, cellulose.—N. H. J. M.

Peat, Fermentation Experiments with. H. von Feilitzen and B. Tollens. Ber. 1897, **30**, 2577—2581.

The process by which it is proposed to obtain alcohol from peat, consists in breaking up the peat with sulphuric acid and fermenting the product with yeast. The yield of alcohol is said to be only about 6 litres from 100 kilos. of dry peat, owing, it is supposed, to the incomplete fermentation of the glucoses.

By digesting peat (300 grms.) with 1 per cent. sulphuric acid (3,000 c.c.), and fermenting the neutralised product with yeast, 3·63 per cent. of alcohol (in the dry and ash-free peat) was obtained. Another sample, which was extracted with ammonia and dilute acid before digestion, and which was subsequently fermented at about 25° C., yielded 4·84 per cent. of alcohol.

Three samples of Speckener peat (see preceding abstract), from different depths, were next hydrolysed, and the glucoses determined by Allihn's method ; similar samples were hydrolysed and fermented. The pentosans had already been determined (*loc. cit.*). The results, showing the possible and actual amounts of alcohol per cent. of dry peat, are as follows :—

———	Speckener Peat.		
	20—100 c.m.	100—200 c.m.	200—300 c.m.
Sugar before fermentation ..	27·87	22·71	11·22
Pentoses....................	14·49	11·08	6·67
Fermentable sugar..........	13·38	11·63	4·55
Alcohol, calculated..........	6·84	5·94	2·33
„ actual...............	6·79	5·46	1·48

Only the hexoses, therefore, yielded alcohol, and the considerable amount of reducing substances, present after fermentation, consists of pentoses. The upper layers of peat yield more fermentable glucoses than the lower, darker-coloured, and more carbonaceous layers.

—N. H. J. M.

Albuminoid Substance, A Structureless, in Bottom-Fermentation Beer Yeast. H. Will. Zeits. für das ges. Brauwesen, **20**, [39], 499—502 ; [40], 512—515 ; [41], 523—525 ; [42], 535—540.

In addition to the results already detailed (this Journal, 1897, 928), it was found that the network formed, in the case of the natural yeast, can be developed by the artificial admixture of relatively large quantities of albumin and peptone with washed yeast ; whereas, on the other hand, additions of gum and vegetable mucilages are much less effective in this direction.

The author also found several forms of network in the film, or yeast ring, in wort cultures. One form is visible direct, in the vicinity of the permanent cells, and gives no albuminoid reaction. A second, from old mucinous mould films, gives all the albuminoid reactions, but has to be prepared to render it visible. Filmy precipitated matters, occurring on the surface of the wort and capable of

assuming the network form, constitute the third class, and give albuminoid reactions. Finally, there is a "crystalline network," consisting of needles of different sizes, and partly of dendritic or fascicular agglomerations of plates. Their nature is as yet undetermined; they are insoluble in hydrochloric acid and concentrated acetic acid, though soluble in nitric acid, highly resistant towards concentrated sulphuric acid, and indifferent towards iodine and aniline dyes.—C. S.

Acetic Acid Bacteria, Contribution to the Physiology and Morphology of the. W. Seifert. Centr. Bl. f. Bakt. und Parasitkd. II. 1897, **3**, [13—14], 337; Zeits. für das ges. Brauw. **20**, [40], 517—518.

Comparative experiments were made on the oxidising action of *Bacterium Pasteurianum* and *B. Kützingianum* (with, in some cases, *B. aceti* Hansen and a species probably identical with *B. xylinum* Brown) on various alcohols and sugars. The nutrient medium generally employed was a decoction of yeast, admixed with the substances under examination; and the temperature of the experiments ranged from 26° to 30° C.

Ethyl- and propyl alcohol were converted by both bacteria into acetic and propionic acid respectively. Butyl- and isobutyl alcohol were also found capable of oxidation, under conditions favouring the development of the organisms; but neither methyl- nor isopropyl alcohol was attacked. On amyl alcohol, *B. Pasteurianum* had no effect, and the action of *B. Kützingianum* remains undetermined.

Ethylene glycol is oxidised to glycollic acid, which restricts the development of the bacteria. Both the last-named organisms exert a much smaller influence on glycerin than does *B. aceti* Brown. Mannite is acted upon only by *B. Kützingianum*, its conversion into levulose being effected less energetically than by *B. aceti* Brown and *B. aceti* Hansen. *B. xylinum* alone oxidises sorbite into sorbose. Glucose is converted into gluconic acid, *B. Kutzingianum* producing the greater effect; the acid restricts both organisms. No change is produced by either in levulose, maltose, propionic acid, or butyric acid, and all are inert towards dulcite. Both readily consume acetic acid.

The author considers it probable that with a more favourable medium, the influence of the organisms might be greater in some of the instances where negative or only slight results were obtained. In any case the conclusion is permissible that the fermentative power of the acetic acid bacteria on monovalent primary alcohols varies inversely with the carbon content of the latter; and also that *B. Pasteurianum* is the weakest of the organisms examined, so far as their effect on the polyvalent alcohols and glucose is concerned.—C. S.

Glycerin in Wine from Grapes affected by Sweet-rot, "Pourriture Noble." J. Laborde. Rev. de Viticulture, 1897, May 8 and Sept. 18; La Bière, **5**, [10], 151—151.

The ratio of glycerin to fermented sugar in wines prepared from grapes which have been allowed, according to the practice current in Sauterne and other districts, to become infected with *Botrytis cinerea*, has been found by the author to range between 4·1 and 11·6 per cent., *i.e.*, considerably higher than in ordinary red and white wines (3·5 per cent.).

The fungus in question causes the grapes to lose moisture, but as its action is gradual and does not come into play on all the grapes in a vineyard at the same time, there result three classes of grapes termed respectively *verts* (unaffected by Botrytis); *pleinpourris* (slightly affected) and *rôtis* (more or less desiccated). That the influence of the Botrytis also induces chemical changes in the fruit, is shown by the subjoined analytical data (1895 grapes):—

Class of Sample.		Must.			Wine.			
		Density.	Acidity per Litre.	Sugar per Litre.	Residual Sugar per Litre.	Alcohol per Cent. by Vol.	Glycerin per Litre.	Ratios of Glycerin to Fermented Sugar per Cent.
			Grms.	Grms.	Grms.	C.c.	Grms.	Grms.
(Grapes) Verts	No. 1......	1,095	2·55	230	0	13·2	7·68	3·3
	No. 2......	1,090	2·70	220	0	12·6	7·16	3·2
„ Pleins pourris	No. 1......	1,115	2·72	263	0	15·5	17·45	6·6
	No. 2......	1,086	2·94	230	0	13·5	11·52	5·0
„ Rotis	No. 1......	1,170	4·31	385	200	10·6	27·50	14·9
	No. 2......	1,157	4·40	339	140	11·2	25·60	12·8

from which it appears that the amount of glycerin increases simultaneously with the progress of the infection. Similar results were obtained with 1896 grapes, and with wines of 1893 vintage. The quantity of alcohol formed, appears to vary inversely with that of the glycerin produced, and though this variation in alcohol content may be in part due—as Gayon has shown—to the retarding influence exerted on the yeast by the presence of high percentages of sugar, still the results obtained are considered to justify the assumption that the development of Botrytis has a similar effect in restricting the formation of alcohol.

—C. S.

Diastase, Chemical Constitution of, and the Occurrence of an Araban in Diastase Preparations. A. Wroblewski. Ber. 1897, 2289.

Diastase was prepared from finely ground malt by first treating it with 68 per cent. alcohol, and then twice with 45 per cent. alcohol. Alcohol was then added to the extract until it attained a strength equivalent to 70 per cent., and the precipitate was purified by solution in 45 per cent. and reprecipitation by 70 per cent. alcohol; and then by re-solution and precipitation with magnesium sulphate. This was dissolved and dialysed until free from sulphuric acid, and finally precipitated by strong alcohol and ether.

The white preparation thus obtained was almost completely soluble in water, gave no coloration with iodine, but all the albuminoid reactions, with the exception of the biuret reaction. It did not reduce Fehling's solution. It agreed in composition and properties with the descriptions of diastase by other writers. When boiled with dilute acids it gave a precipitate which yielded the albuminoid reactions, the filtrate reducing Fehling's solution.

To determine if this preparation was a mixture, Brücke-Kulze's method was employed. The solution of the diastase was treated with mercury-potassium iodide and dilute hydrochloric acid; a voluminous precipitate was thrown down, which was found to contain an albuminoid. The solution gave a precipitate with alcohol, which was found to be a dextrin-like carbohydrate.

The albuminoid was freed from the metals thrown down with it; it was then only partially soluble in water, but was able to transform soluble starch into sugar.

The carbohydrate did not reduce Fehling's solution, and was levo-rotatory. When boiled with dilute acids it yielded arabinose. It thus appears that the substance, previously described as diastase, is a mixture of a carbohydrate with an albuminoid, of which the latter only is the active body. This contains 15·3—16·2 per cent. of nitrogen.—A. L. S.

Barley, The Germination of, with Restricted Moisture. T. Cuthbert Day. Trans. and Proc. Bot. Soc. Edinburgh, 1896, 492—501.

The author's experiments were carried out with three varieties of barley, *viz.*, Hungarians, Scotch Chevalier, and Ordinary Smyrna. The degree of moisture for germination

was estimated by determining the moisture in one portion of the barley, then taking the weight of 100 selected corns and steeping them in distilled water at 57° F. for 24, 48, 72, or 96 hours. At the end of the steeping period the corns were externally dried and weighed. The amount of the soluble matter in the steep water, added to the increase of weight in the corns, gave the total quantity of water absorbed; the absolute amount was found by adding the original moisture present in the sample. The steeped barley was then placed in a small flask immersed in water at 57° F., the apparatus being so arranged that a current of air saturated with aqueous vapour at 57° F., from which carbon dioxide had been removed, could be aspirated. The air in passing through the apparatus could thus take no moisture from the germinating grain, neither could the corns extract any moisture from the air supplied to it.

The aspirated air was dried and passed through weighed potash bulbs. Germination lasted from 12 to 16 days; the corns were then taken out of the flask and weighed, the embryos excised from the endosperms and husks, and the moisture determined in each by drying at 100° in a current of dry air at reduced pressure. A large number of experiments were carried out under the above conditions, and the results shown graphically in curves.

With regard to the quantity of carbon dioxide exhaled, it appears that an increase of moisture always produces a corresponding increase in the amount of this gas. Moreover, when the moisture is considerably increased, it was found that during the first day or two of germination, the quantity of carbon dioxide excreted is little less than for lower degrees of moisture, but the grain soon recovers its partial drowning, and increased activity due to increase of moisture, asserts itself.

The dry weight of the embryo of a corn germinated for 14 days, after a 96 hours' steep, gave a value more than double that obtained when the steep was only 24 hours.

When the number representing the increase in the dry weight of the embryo during germination is divided by the weight of carbon dioxide exhaled, it was found that the product increases in value as the quantity of moisture used in germination is increased. It thus appears that with the larger quantities of moisture, the embryo increases more in weight for the same amount of carbon dioxide exhaled, than it does when less moisture is present.

The percentage of moisture found in the embryo at the end of the germinative period was generally nearly double of that found in the endosperms, the disparity being always greater the smaller the quantity of moisture employed in the experiment. The results of the moisture determinations seem to show that the new forming tissue cannot grow with any degree of success unless it contains above 63 per cent. of water.

Considering the production of carbon dioxide as a measure of growth during germination, it was found that the period of greatest activity, with varying quantities of moisture, generally occurred about the third or fourth day, the period being to some extent delayed with increase of water. After the point of greatest activity is reached there is a gradual fall in vitality to the end of the germinating period.—J. L. B.

Fermentation of Green Grass, Chemical and Bacteriological Investigation of. O. Emmerling. Ber. 1897, **30**, [14], 1869.

See under XXIV., *page* 1051.

Ethyl Alcohol, Highly Diluted [1 *in* 3,000 *to* 1 *in* 10,000]; *Distillation of.* Nicloux and Bauduer. Analyst, 1897, [259], 263; Ann. de Chim. Analyt. **2**, [11], 202.

See under XXIII., *page* 1047.

Wine, Determination of Glycerin in. C. Boettinger. Chem. Zeit. 1897, **21**, [67], 658.

See under XXIII., *page* 1048.

Spirits, Estimation of the Higher Alcohols in. X. Rocques. Analyst, **22**, [259], 263; Ann. de Chim. Analyt. **2**, [8], 141.

See under XXIII., *page* 1048.

PATENTS.

Seasoning or "Curing" Casks, Improved Process of, and Apparatus therefor. J. Gillies. Eng. Pat. 29,899, Dec. 29, 1896.

The casks are cured by steam and heated air or hot products of the combustion of carbonaceous matter, with or without the addition of sulphurous gases. The admission is controlled by a plug-valve so constructed that it may be rotated in its casing to connect the parts with steam and air pipes. An injector is formed on or secured to it, enabling air to be drawn in and mixed with the steam.
—J. L. B.

Ale, Pale; Impts. in or relating to the Manufacture of. A. Fraser, Alloa, Scotland, and Maclay and Co., Limited, Alloa, Scotland. Eng. Pat. 15,200, June 25, 1897.

The patentees claim the use of oat malt as one of the ingredients used in the preparation of pale ale: the combination of oat malt with sugar, hops, and water, and oat malt with barley malt for brewing purposes. It is noted that oats in the process of malting require to be steeped for 45 hours, as compared with 72 hours for barley.
—J. L. B.

Ageing Whiskey and other Alcoholic Liquors, An Improved Process and Apparatus for. D. J. Etly and C. R. Long, Louisville, Kentucky, U.S. Eng. Pat. 18,713, Aug. 12, 1897.

The lower end of a funnel-shaped tube, closed at the bottom and filled with water, is placed within the barrel containing the alcoholic liquid; the temperature being raised and maintained by means of steam. When the heating has been continued sufficiently long, steam is shut off and salt introduced into the tube to form a strong brine. A refrigerating material, such as gaseous ammonia, is forced through pipes contained in the tube until a low temperature is reached. This alternate heating and cooling is continued for varying periods—according to the character of the alcoholic liquid—until the desired flavour and aroma are obtained. An apparatus enabling several barrels of liquid to be treated simultaneously is also described.—J. L. B.

XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

(*A*.)—FOODS.

Food Products, Antiseptics in. F. Jean. Rev. de Chim. Ind. 1897, **8**, [94], 289—293.

Although the use of antiseptics is entirely forbidden in France, the author states that the authorities are often very lax in enforcing the law, and cites an instance in which the vendor of borated butter was acquitted in one court and fined in another. This laxity, together with contradictory judgments, have produced uncertainty among traders as to what is permissible, and have encouraged the sale of antiseptics and preserved articles of food. Thus, hams preserved with borax are largely imported into France from England and America, and, according to Prof. Carles, of Bordeaux, immense quantities of fluorides are being sold under various names in that part of the country. In addition to the more common antiseptics, the author states that fluosilicates and fluoborates are employed, and he has even found fluosilicic acid in butter. With regard to the ill-effects of preserving agents in general, he takes boric acid as the type, and quotes the toxic effects ascribed by Mitscherlich to that substance. According to le Féré, borax is only eliminated from the system very slowly, and has been detected in the urine 40 or 50 days after it had been taken.—C. A. M.

Milk, Preservation of, by Partial Freezing. F. Jean. Rev. Chim. Ind. 1897, **8**, [93], 267—268.

According to G. Quesneville, the pasteurisation of milk is often employed to conceal various forms of adulteration, noticeably the removal of cream, since skimmed milk when heated on the water-bath assumes the appearance of whole

milk. Now that the Casse process has proved successful on a commercial scale, he considers that it should take the place of sterilising by heat, which alters the character of the milk. In the early part of 1896 the Casse Company established two milk depôts at 160—170 kilometres from Copenhagen, for freezing milk, and a third depôt in Copenhagen for defrosting and distributing it. In the three depôts, 30,000 litres of milk can be treated each day at a cost of about ½ centime per litre. In ordinary weather, ¼ to ⅙ of the total milk is frozen; in very hot weather, ⅛ to ⅓. After being frozen, the milk, in blocks of about 12 kilos., is placed in isolated vessels, which next day are filled up with fresh milk and despatched to Copenhagen.

As an experiment, 300 litres thus partially frozen were sent from Copenhagen to Paris, when it was found, after defrosting, that the milk was perfectly normal in character and of excellent quality.—C. A. M.

Milk, Occurrence of Alcohol in. H. Weller. Analyst, 1897, [259], 264; Forsch. Ber. 1897, **4**, 206.

König (Nahr. und Genussmittel, 1893, 249) stated that when cattle are fed upon fodder containing alcohol, the latter does not pass into the milk. The author, however, obtained the following results on examining a milk derived from a number of cows which were fed on distillery slummage containing 5·9 per cent. of alcohol:—Specific gravity of milk, 1·0335; fat, 3·89 per cent.; total solids, 13·307 per cent.; and alcohol (by weight), 0·96 per cent. —A. S.

Coffee, Raw; Amount of Water in. B. Niederstadt. Forsch. Ber. 1897, **4**, 141.

With the object of determining the average percentage of water in raw coffee as put upon the market, the author examined samples of several different kinds. These were dried to constant weight at 105° C., and gave the following results:—Santos I., 13·33; Santos II., 12·20; Santos III., 11·23; Santos IV., 14·50; Bahia, 8·54; Menado, 10·72; Java, 10·75; Columbia, 10·00; Costa Rica, 10·62; Guatemala, 10·18; and Santos V., 9·45; the mean being about 11 per cent.—C. A. M.

Coffee, Alterations produced in, on Roasting. A. Juckenack and A. Hilger. Forsch. Ber. 1897, **4**, 119—135.

The authors have studied the alterations which take place when coffee is roasted in the usual manner, and with the addition of 8 or 9 per cent. of sugar as specified in a German patent. The chief results of their experiments are:—

1. *Caffeine.*—In the ordinary process of roasting, coffee loses about 21 per cent. of its caffeine, and about twice that quantity when roasted with sugar, owing to the higher temperature which is required to caramelise the sugar, so as to glaze the coffee.

2. *Fat.*—From 9 to 10 per cent. is lost during the ordinary process of roasting, and about 20 per cent. in the glazing process. As a rule the different constants of the fat show an increase on roasting by either method.

3. *Organic Matter.*—In the authors' experiments the loss on the average amounted to 11·375 per cent. in the ordinary roasting, whilst in the glazing process it was about 10 per cent., allowance being made for the amount of caramel. This was determined by Stutzer's method. 20 grms. of the glazed beans were shaken in a machine for five minutes with 500 grms. of water at 20° C. The liquid was filtered, and the residue in 250 c.c. of the filtrate, after deducting the ash, was taken as caramel. In this way, on coffee glazed with 9 per cent. of sugar, there was found 1·40 per cent. of caramel, as against 1·25 per cent. on a sample of Java coffee glazed with 8 per cent. of sugar. (This Journal, 1897, 473.)—C. A. M.

Foods, Vegetable; Amounts of Ash and Iron in certain. B. Niederstadt. Forsch. Ber. 1897, **4**, 140.

This paper gives the figures recently obtained by the author in his analyses of various vegetable products used as food. The amount of iron as Fe_2O_3 in 26 samples of American apple cuttings, varied from 0·009 to 0·046, but as these had all been cut with a steel knife, it was possible that part of the iron might have been derived from the knife. The mean percentage of ash in the specimens of malt examined was 2·5, and the iron oxide ranged from 0·25 to 0·91 per cent. In unroasted coffee berries of various origin, Liebig found from 0·019 to 0·011 per cent. of iron oxide whereas the author's analysis of 17 samples gave, as a rule, considerably higher results, the raw berries containing from 0·35 to 0·88 per cent., and the roasted berries from 0·07 to 0·65 per cent.—C. A. M.

Milk, Formalin in; Detection of, by Means of Phloroglucinol. Jorisson. Bull. Soc. Chim. 1897, 167; Analyst, 1897, [260], 282.

See under XXIII., *page* 1015.

Proteid and Gelatinoid Substances, Improved Method of Determining. A. H. Allen and A. B. Searle. Analyst, 1897, [259], 258.

See under XXIII., *page* 1017.

Gases, Method of Collection and Analysing the, in Canned Goods. C. A. Doremus. J. Amer. Chem. Soc. 1897, **19**, 733.

See under XXIII., *page* 1016.

PATENTS.

Preserving Organic Bodies, Impts. in Apparatus for. L. A. M. D. de Belfort, Paris. Eng. Pat. 14,907, June 19, 1897.

See under I., *page* 1005.

Coffee, Impts. in the Treatment of. S. Feitler, Ludwigshafen, Germany. Eng. Pat. 19,815, Aug. 28, 1897.

The coffee is treated, before or after roasting, with gelatin and resinous substances, such as gum, benzoin, shellac, stick lac, &c. The pores of the beans are thus hermetically closed, enabling the aroma to be preserved for a long period.—J. L. B.

(B.)—SANITATION; WATER PURIFICATION.

Disinfection of Houses. F. W. Alexander. Public Health Engineer, **1**, 543—544 and 557—559.

The replies received to a circular letter sent round to the chief sanitary authorities of this country, asking for information upon certain points in the disinfection of houses and clothing, showed that there is a lack of uniformity in the processes now in use, but that disinfection by means of sulphur fumigation is the most usual method.

As the experiments carried out by Drs. Koch, Wolffhügel, Hüppe, and Proskauer proved this mode of disinfecting rooms to be most unsatisfactory and unreliable, the author investigated the relative powers of other disinfectants, *viz.*, corrosive sublimate, formic aldehyde, para-formic aldehyde, and quinosol ("Chinosol"). From the results obtained in these investigations, the following suggestions are made:—

1. That disinfection of rooms by means of sulphur gas should cease, as its efficiency to kill all germs appears to be more than doubtful.

2. That the walls, floors, and ceilings of rooms should be sprayed with a formalin (formic aldehyde) solution, 1 in 80, or with a solution of quinosol, 1 in 1,000.

The benefits accruing from adopting the above method (2) may be summarised:—

1. The disinfectant may be applied immediately to the floors, walls, and ceilings.

2. The disinfectant used is not harmful, either to the men carrying out the work, or to the occupants of the rooms, should it eventually, after becoming dry, float into the air as dust.

3. The disinfectant in solution is of sufficient strength to kill all germs.

4. The necessity of sealing up the rooms for hours would be abolished, and the tenants would practically occupy the rooms almost immediately after the spraying process had been concluded.

5. The spraying process may be carried out in the time taken for sealing up the rooms in the sulphur process.

6. The process is much cheaper than sulphur fumigation. A number of rules are also added for the guidance of the men carrying out the disinfecting work, and relate to the wearing and disinfecting of the overalls worn and the washing out and fumigation of the vans employed for the collection and delivery of articles.—W. P. S.

PATENTS.

Filter for Sewage Effluent and the like, An Improved. C. Fell, Hanley, Staffordshire. Eng. Pat. 22,768, Oct. 14, 1896.

The crude sewage, just before flowing into the precipitation tanks, is made to pass through perforated zinc boxes containing chloride of lime; and the effluent from the tanks is made to pass through similar finely-perforated boxes also containing chloride of lime.—L. A.

Sewage Sludge, Improved Process for Treating. B. Walker, Hawes, Yorkshire. Eng. Pat. 24,833, Nov. 6, 1896.

A small quantity of peat, intimately mixed with sewage sludge, makes the latter much more porous and combustible. Even 0·5 per cent. of peat dust greatly facilitates the filtration and pressing, but a larger quantity, 1 of dry peat to 7 of water in the sludge, is preferable. The pressed cake may be broken up, mixed with more sludge and re-pressed several times, and will burn by itself in a furnace, or even in the open air, if the fire be started with dried cake or other fuel, especially if grease or soap be contained in the sewage.

When iron precipitants are used, the ash consists largely of magnetic oxide of iron, and is an excellent filtering medium. The filter is constructed of furnace clinker, broken and sifted into various sizes, and arranged in layers. The calcined sewage sludge forms the top layer which, when choked, is shovelled off, pressed, and re-calcined. The filter is kept aerated by means of air-pipes communicating with the various layers.—L. A.

Filtering Apparatus [Water], Impts. in and relating to. H. H. Lake, 45, Southampton Buildings, London, W.C. From "La Société Ch. Prevet et Cie.," Paris. Eng. Pat. 25,710, Nov. 14, 1896.

The filter consists of two shells of unsized paper, which are connected at their edges, and enclose a lens of perforated metal, or of porous material such as carbon. The liquid passes through the filter from the exterior to the interior, the lens being formed with, or serving as, a chamber for the reception of the filtered liquid, and being fitted with a delivery nozzle, &c. The filter thus constructed, forms a portable or pocket filter, suitable for use by soldiers, sportsmen, &c., but a number of filtering elements may be combined in a single apparatus, several arrangements of such combinations being described in the specification.
—R. A.

Drains, Improved Means for Testing and Disinfecting. C. T. Kingzett, Chislehurst, Kent. Eng. Pat. 28,090, Dec. 8, 1896.

Any suitable material which emits smell or smoke on simple exposure, or on being wetted, or a disinfectant, is contained in a small metallic cylinder open at one end, and so inserted open end uppermost, into a larger cylinder containing a spring, or having an elastic band stretched over its mouth, that when flushing takes place by means of water, the spring or elastic diaphragm is released, causing the inner case to be forced out and the material liberated.
—L. A.

Garbage, and the Recovery of Ammonia therefrom; Method and Furnace for the Incineration of so-called. J. F. G. de Bonardi, Marquis du Ménil, Paris. Eng. Pat. 29,009, Dec. 17, 1896.

Refuse matter, such as city garbage, after separation of incombustible substances and of powder directly applicable as a manure, is charged through hoppers provided with valves so constructed as to exclude free passage of air, into a distilling chamber constituting the upper part of the furnace. The refuse is carried forward on trays by endless chains to the front of the furnace, where the bottom of the chamber slopes downward, and is provided with valved apertures, through which the charred refuse descends, passing into the incinerating chamber, whence the ash is delivered into a closed ash pit. The incinerating chamber is supplied with air, in such quantity, that whilst ensuring combustion, excess of oxygen is avoided, and the reducing gases resulting are led through a series of chambers in which the greater part of the heat is withdrawn for utilisation, so that they may pass into the distilling chamber at a temperature not exceeding 650° C., the temperature of the incinerating chamber varying between 1,000° and 1,500° C. The gaseous products of distillation are withdrawn by a fan into ammonia-absorbing apparatus.—E. S.

Sewage, Impts. in and relating to Material for Use in the Treatment and Purification of, and other Impure Waters. W. B. Bottomley, London. Eng. Pat. 29,288, Dec. 21, 1896.

Native aluminous and ferruginous calcium phosphate is ground, and treated with sulphuric or hydrochloric acid, in sufficient proportion to form soluble aluminium and iron salts with the phosphate. The mixture is used in precipitating sewage, from 8 to 10 grains per gallon of sewage being recommended in ordinary cases.—E. S.

Sewage and the like, Impts. in or relating to the Filtration of. J. Guthrie, Hawick, Roxburghshire, N.B. Eng. Pat. 18,974, Aug. 17, 1897.

The invention is a mechanical device for automatically controlling the operation of two or more filters working in rotation. As each filter becomes full of liquid, a float rises which, by tilting the inlet trough, diverts the influent to another filter, and at the same time opens a valve which discharges the first filter.—L. A.

(C.)—DISINFECTANTS.

PATENT.

Formic Aldehyde Vapours, Impts. in the Production of. G. B. Ellis, London. From La Société Chimique des Usines du Rhône, formerly Gilliard P. Monnet et Cartier, Lyons, France. Eng. Pat. 27,656, Dec. 4, 1896.

The method claimed consists in mixing a solid polymer of formic aldehyde (for instance, trioxymethylene) with water and then heating the mixture to 130°—135° C. in an autoclave capable of resisting a pressure of 5—6 atmospheres. It is found preferable to mix 100 parts of the solid polymer with 40 parts of sea salt (or other normal salt) and to press this mixture into cakes, for convenience in transport.
—W. P. S.

XIX.—PAPER, PASTEBOARD, Etc.

Observations [and Analytical Methods used] in the case of some of the Chemical Substances in the Trunks of Trees. F. H. Storer. Bull. Bassey Inst. 1897, **2**, 386.

See under XXIII., *page* 1049.

Filter Papers, The Reaction of. L. Magnier de la Source. Rev. Intern. falsific. **10**, 165; Chem. Centr. 1897, **2**, [17], 909.

See under XXIII., *page* 1040.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

Oxygen, Industrial Preparation of. Dutremblay and Lugan. J. Pharm. et de Chim. 1897, **6**, [9], 392—396.

Tessié du Motay's well-known manganate process is carried out best under a slight pressure, it is said, and so that th

entry of air or furnace gases is impossible. The oxygen is forced to the gas-holder by the steam pressure, thus avoiding impurities arising from a lubricated pump. The purifier consists of a horizontal cylinder, divided by screens carrying the purifying mixture over which the air passes. The furnace consists of a generator, combustion chamber, and two heating chambers, each containing ten vertical retorts. These retorts are connected with systems of pipes for leading in air and steam, and for conveying away nitrogen or oxygen. The distribution of the gas, to or from the retorts, is effected by ten counterpoised valves worked by compressed air. A distributor, worked electrically, sends the compressed air to the piston moving each valve. The gas-holder is provided with a condenser. Up to the present, two serious defects have prevented the success of the Tessie du Motay process—the settling of the mixture and the evaporation of the soda. The use of horizontal retorts with a thin layer of manganate in small lumps had avoided the first defect, but this disposition of the material led to the formation of channels. The patents of Lawson (1890), Bowmann (1891), Parkinson (1891), Fanta (1891), Webb (1892), &c., endeavoured to overcome the settling by the use of various admixtures with the manganate. The authors have found the solution of the problem "in mechanical arrangements applicable to vertical retorts."

The compressor is of the compound type, the pistons lubricated by an injection of water, which is extracted from the gas before it reaches the cylinders, by passage through a refrigerator. A furnace containing 20 vertical retorts, 140 mm. in diameter and 2·4 m. high, will make in 24 hours 100 c.m. of oxygen free from carbon, and containing 94—96 per cent. of pure oxygen.—A. C. W.

Cerium. O. Boudouard. Comptes Rend. 1897, **125**, [20], 772—774.

Cerium sulphate, free from thorium, was precipitated by lead acetate and excess of lead removed as sulphide. In the cerium acetate solution on standing, a precipitate formed, which was converted into the sulphate, and the sulphate was analysed by ignition. The atomic weight Ce = 137·85 was deduced from the results of this process. In the same way, the recrystallised sulphate gave Ce = 137·35, and the salt obtained from the mother liquors Ce = 135·1. The mother liquors of the basic acetate were separated into four fractions; these gave figures ranging from 136·05 to 139·1.

Hydrogen peroxide partially precipitates cerium from solutions of its acetate; the precipitated oxide was converted into sulphate and this salt fractionally crystallised. The fractions gave atomic weights between 137·15 and 137·6. The cerium precipitated as oxalate from the residual hydrogen peroxide liquor, gave results from 137·85 to 139·9.

Cerium sulphate was fractionally precipitated by potassium sulphate. From the fractions results ranging between 133·0 and 138·75 were obtained. The author concludes that oxide of cerium is accompanied by small quantities of the oxide of a metal of lower atomic weight, which metal can form a dioxide and an insoluble double sulphate with potassium sulphate.—A. C. W.

Silver, Colloidal. A. Lottermoser and E. von Meyer. J. Prakt. Chem. 1897, **56**, [17—18], 241—247.

Few quantitative observations on the conversion of colloidal into common silver have been made. The use of "soluble silver" as an ointment in septic diseases gave surprising effects; and it was found that the silver passed into the fluids of the body, in spite of the fact that they all contain small quantities of sodium chloride, which ordinarily precipitates silver from the aqueous colloid solution. This behaviour was explained by the discovery that albumin partially or wholly prevents the precipitation of colloidal silver by sodium or hydrogen chlorides.

The conversion of silver from the colloidal to the insoluble form was observed by the following titration method:—5 c.c. of a 0·5 per cent. aqueous solution of colloidal silver was diluted with a known volume of water, and standardised solution of an acid or salt was run in from a burette. At one point a sharp change from the original coffee-brown colour to green could be detected, but the completion of precipitation was proved when a drop of the liquid brought on filter paper appeared colourless, except for the fine particles of silver present. The colloidal solution used, was free from insoluble silver, but contained traces of ammonium citrate. With regard to acids, it was found that the quantity needed to precipitate the colloidal silver was inversely proportional to the co-efficient of affinity of the acid, but that it increased with the degree of dilution of the silver solution, and decreased with a prolongation of the time allowed for the reaction. The following gives the volume (in c.c.) of normal solutions of certain acids, which precipitates the silver from 25 c.c. of 0·1 per cent. colloidal solution:—formic 4·75, monochloracetic 0·93, trichloracetic 0·25, cyanacetic 0·8, oxalic 1·2, malonic 2·41, succinic 16·25, and tartaric 3·75. The volume for $\frac{1}{2}$N. sulphuric acid is 2·13 c.c.

The action of salts on the silver solution varies according to their nature. Small quantities of the alkali salts of acids which form silver salts soluble with difficulty, precipitate the silver, but alkali salts of acids which form readily-soluble silver salts do not throw down all the silver in the insoluble form, even when present in much larger amount. Very small quantities of salts of the heavy and earth metals effect the change. The halogens readily convert colloidal silver into salts, which also behave as colloids; thus when 3 per cent. alcoholic solution of iodine is added to dilute colloidal silver, a milky and yellowish-white liquid results. The whole of the silver iodide, however, is thrown down by a small addition of mineral acid.

Colloidal silver is a very feeble conductor of the electric current, which, however, causes a migration of the particles. Grey, spongy silver accumulates at the cathode; and at the anode there collects a brown mud of silver only, which yields a green aqueous solution. When dried in the exsiccator this modification changes to golden-yellow, and is insoluble in water.—J. A. B.

Sodium Iodide. J. C. Umney. Pharm. J. 1897, **59**, [1424], 312.

See under XXIV., *page* 1051.

Protargol [Albumin containing Silver], Note on. O. Loew. Chem. Zeit. **21**, [84], 876.

Protargol is the name given by Eichengrün to a compound of albumin containing silver, in which the silver (8·3 per cent.) is present, not as a salt, but in molecular combination. The compound, on account of its bactericidal action, has found application in therapeutics. Loew states that he prepared such compounds in 1883, by the continued heating of albumin with ammoniacal silver solution. The silver, which can neither be precipitated by sulphuretted hydrogen nor hydrochloric acid, varies in amount with the quantity of silver nitrate used. Addition of caustic potash causes a marked enrichment in silver, and preparations containing from 32 to 82 per cent. of silver may be obtained. —J. T. C.

Chloroform, Bromoform, and Chloral; Decomposition of, by Aqueous Potash. A. Desgrez. Comptes Rend. 1897, **125**, [20], 780—782.

Alcoholic potash decomposes chloroform to form potassium chloride and formate. Prunier has shown that at temperatures below 30° C., traces of formate are produced, but chiefly carbon oxychloride, and a little hydrogen. The author finds that chloroform is decomposed by aqueous potash in the cold, with the production of carbon monoxide. $CHCl_3 + KOH = KCl + 2HCl + CO$. This is the principal reaction when 10 grms. of chloroform, 400 grms. of water, and 50 grms. of potash are mixed. Light accelerates the reaction. Bromoform is decomposed more slowly by reason of its lower solubility; insoluble iodoform shows no decomposition. Chloral is more rapidly decomposed than chloroform, the heat generated by its first decomposition accelerating the second. Ammonia and the alkaline bicarbonates do not produce this reaction, but André has shown that it takes place with water alone, in sealed tubes at 200°—225°.—A. C. W.

Anesin [Aqueous Solution of Tertiary Trichlorobutyl Alcohol]: A new Anæsthetic. Apoth. Zeit. 1897, **12**, 608.

A new synthetic body has been introduced, which is stated to be an aqueous solution of tertiary trichlorobutyl alcohol, having marked anæsthetic and hypnotic properties. —J. O. B.

Strophanthus Seeds, Adulteration of. P. Siedler. Ber. Pharm. Ges. 1897, **7**, 222.

Strophanthus seeds are often adulterated with those of the African plant *Kicksia Africana*. With the aid of a simple lens they can be distinguished, the *Kicksia* seeds being smooth, spiral, or S-shape, not flat, and with base and tip about equally pointed, while the *Strophanthus* seeds always show traces of hairs, are flatter, rounded at the base and sharp at the tip. The cross section shows the cotyledons much folded in the *Kicksia* seeds, but parallel in the others. Treated with strong sulphuric acid, the cross section of the *Kicksia* seeds turns first brown, then red, whilst that of the *Strophanthus* seeds turns green.—J. T. D.

Pine Oils, American. Semi-Annual Report of Schimmel and Co., Oct. 1897, 47.

Oil of Fir Needles.—Distilled from the leaves of *Pinus sylvestris*, this oil had a sp. gr. of 0·884, and a rotation of $-24° 8'$. This levo-rotation is noteworthy, since it agrees with that of the English distilled oil; the German and Swedish oil is dextrogyrate. The American oil contained bornyl acetate, which, from the acid number, 34·8, is present to the extent of 12·1 per cent. The oil was soluble in 8 vols. and more of 90 per cent. alcohol.

Oil of Hemlock Needles.—Distilled from the leaves of *Abies canadensis*, the specific gravity was 0·911 and the rotation $-25° 22'$, at 16° C. The oil contains 38 per cent. of bornyl acetate, and is soluble in one or more parts of 90 per cent. alcohol.

Oil of Spruce.—Distilled from needles of *Picea nigra*. The specific gravity was 0·930 and the rotation $-23° 50'$, at 18° C. The oil contained 31·8 per cent. of bornyl acetate, and was soluble in $\frac{1}{2}$ vol. of 90 per cent. alcohol; on further addition of $4\frac{1}{2}$ vols. the solution becomes opalescent.—J. O. B.

Larch Needle Oil. Semi-Annual Report of Schimmel and Co., Oct. 1897, 61.

The needles of *Larix europæa* yielded only 0·22 per cent. of oil on distillation. This had the sp. gr. 0·878, and was dextrogyrate $+0° 22'$, at 18° C. It was soluble in 90 per cent. alcohol, and its acid number was 23·3, which, after acetylisation, was increased to 46.

Presuming the ester of larch oil to be bornyl acetate, as is the case with most coniferous essential oils, the first figures would indicate 8·9 per cent. of bornyl acetate, equivalent to 6·5 per cent. of combined borneol, while the latter figures indicate 6·14 per cent. of free borneol; the total borneol being therefore 12·76 per cent.

Submitted to fractional distillation, the following results were obtained:—

From 160° to 165° C., 30 per cent.; from 165° to 170° C., 24 per cent.; from 170° to 180° C., 16 per cent.; from 180° to 190° C., 8 per cent.; from 190° to 200° C., 4 per cent.; from 200° to 230° C., 9 per cent. Residue, 9 per cent.

The oil had a pleasant larch needle fragrance; the small yield and the difficulty in obtaining large quantities of raw material are, however, drawbacks to its considerable commercial use.—J. O. B.

Star Anise, Cultivation of, in China, and Method of Distilling the Oil from. "Decennial Reports, 1882 to 1891, of Trade, &c., of the Ports open to Foreign Commerce in China and Corea." Published by order of Inspector-General of Customs, Shanghai, 1893; through Semi-Annual Report of Schimmel and Co., Oct. 1897, 57.

The Lungshow star anise is considered better than that produced in the Po-sê district, and yields more oil. The trees, originally wild, but now frequently cultivated, grow on hill sides, where the sloping ground prevents water from standing around the roots, yet retains enough moisture as the rain runs down from the higher ground above. The trees are very liable to injury from smoke, and as the common habit in clearing fields is to burn the grass and débris, the neighbourhood of villages is avoided in selecting a place to plant, and the dry grass beneath the trees is cut and carried off yearly to prevent the spread of accidental fires. Young trees bear fruit suitable for commercial purposes after 10 years, and trees 100 years old still bear. The yield of the same tree varies much from year to year, and trees that produce every year are rare. They flower at the beginning of February, and the anise fruit is gathered in August and September. Fresh anise fruit is worth ordinarily 5.50 dols. per picul.

The oil is extracted from fresh anise fruit by distillation. Steam passes through a wooden cylinder containing the anise, placed over the boiler, into an earthenware jar, where it is condensed by cold water placed in a pan on the top. The condensed mixture of water and oil flows through a pipe into a tin-lined box of two compartments, in the partition between which is a hole near the top. The oil floating on the top of the water is drawn off through this hole into the other compartment. The process takes several days.

Fresh anise fruit yields 3 per cent. of oil, and a picul of oil is worth from 180 dols. to 190 dols. For transport it is packed in tins of 32 to 35 catties weight, and sent overland to Hong Kong.—J. O. B.

Cardamom Oil (from Various Species of Cardamoms). Semi-Annual Report of Schimmel and Co., Oct. 1897, 10.

Oil of Malabar Cardamoms.—The fruits of *Elettaria cardamomum* gave 2·14 per cent. of light yellow oil closely resembling in odour the commercial oil derived from Ceylon cardamoms. The specific gravity was 0·943, and optical rotation $+34° 25'$ at 19° C. It was soluble in four or more volumes of 70 per cent. alcohol. The saponification factor (acid number) was 132, indicating a high percentage of ester. The acid constituent of this ester was found to be acetic acid. On fractional distillation, cineol and a small amount of an optically active terpineol were obtained. The latter was strongly dextrogyrate, the figure being $+83° 31'$ at 21° C. It further differed from the terpineol of cajaput, which is optically inactive, in giving a nitrolpiperidine melting at 151°—152° C. instead of 159°—160° C., the melting point of that compound from inactive terpineol.

Oil of Siam Cardamoms.—The fruits of *Amomum cardamomum*, which occasionally appear in the London drug market, where, from their camphoraceous odour, they are known as "camphor seeds," yielded 2·4 per cent. of oil, which was semi-solid at ordinary temperatures, liquefying at 42° C., when it had a specific gravity of 0·905 and a rotation of $+38° 4'$. The odour of camphor or borneol was very marked in this oil. Its saponification factor (acid number) was 18·8. On acetylisation, this was increased to 77·2, corresponding to the presence of 22·5 per cent. of borneol. By centrifugation, the stearoptene was eliminated, and subsequently separated by crystallisation from petroleum spirit into dextrogyrate borneol and dextrogyrate camphor in approximately equal proportions.

Oil of Cameroon Cardamoms.—The fruits from which this oil was distilled were identical with those of Madagascar cardamoms, and are derived from *Amomum danielli*. The yield was 2·33 per cent. The specific gravity was 0·907, and the rotation $-20° 4'$. The odour is similar to that of cineol, which body was detected in the oil by the iodol reaction.

Oil of Grains of Paradise.—The seeds of *Amomum melegueta* gave 0·75 per cent. of oil, which had a specific gravity of 0·894 and a rotation $-3° 58'$, and was soluble in 10 to 11 volumes of 90 per cent. alcohol.—J. O. B.

Harmine and Harmaline.—III. O. Fischer. Ber. 1897, **30**, [16], 2481—2489.

The earlier researches (Ber. **18**, 400; and **22**, 637) on the alkaloids obtained from the seeds of *Peganum harmala* had shown that harmaline, $C_{13}H_{14}N_2O$, is dihydroharmine, and that both alkaloids gave on oxidation the base $C_8H_8N_2$, apoharmine. Harmine and harmaline are optically inactive. The former may be obtained from the latter by oxidation

with nitric acid, or better, potassium permanganate in acid solution. Both alkaloids form addition compounds with methyl iodide, which, decomposed by alkali, yield methyl harmine and methyl harmaline. The tertiary bases so obtained combine with methyl iodide. From the harmine compound, dimethylharmine was obtained.

By reduction of harmaline by sodium in amylic alcohol solution, dihydroharmaline (tetrahydroharmine) is formed. This substance, as also dihydro-apoharmine, had already been shown to be a secondary base. Harminic acid is obtained by the oxidation of harmine or harmaline with chromic acid in acetic acid solution. Methylharminic acid, $C_{10}H_7N_2O_4(CH_3)$, is produced by the oxidation of methylharmine, or by the methylation of harminic acid.

Apoharmine, $C_8H_8N_2$, yields the nitro compound $C_8H_7N_2(NO_2)$ by the action of concentrated nitric acid, and methylapoharmine, $C_8H_7N_2(CH_3)$, on the decomposition of its methyliodide compound with potash.—A. C. W.

Concrete Flower Oils (So-called). Semi-Annual Report of Schimmel and Co., Oct. 1897, 86.

On account of its solubility in strong alcohol, myristic acid has lately been used for the preparation of "concrete flower oils." When submitted to distillation, or exposed to a temperature of 100° C., the following non-volatile residues were left with various samples of "concrete flower oils":—Jasmine, about 50 per cent.; Lilac, about 49 per cent.; Acacia about 69 per cent.; Rose, about 73·8 per cent.; Mignonette about 85 per cent.; Violet, about 86 per cent.—J. O. B.

Rosemary Oil. Semi-Annual Report of Schimmel and Co., Oct. 1897, 52.

In order to determine if the presence of pinene, previously noted in rosemary oil, was due to the addition of turpentine, or if that terpene was a natural constituent of the oil, a collection of rosemary leaves from several Dalmatian islands has been distilled in Leipzig and the oil examined. This oil was found to contain dextrogyrate pinene and camphene, and the same constituents were present in a good Italian sample of the oil. The mere presence of pinene cannot, therefore, alone, be regarded as evidence of adulteration.

Simple fractionation was found to be of little value in determining the quality of the oil, since the same oil gave different results according to the method of distillation, or the form of the retort employed. The variations of atmospheric pressure also greatly interfered with the result. Large additions of turpentine can, however, be detected in this manner, since when more than 20 per cent. of the oil distils below 170° C., it can safely be regarded as adulterated.

Small additions of French oil of turpentine can be detected by the rotation of the first fraction. This should always be dextrogyrate.

A pure rosemary oil should therefore possess the following characters:—Specific gravity should not fall below 0·900. The oil should be dextrogyrate. One volume of the oil should give a bright solution with ½ or more volumes of 90 per cent. alcohol: also with 10 parts of 80 per cent. alcohol.

The first fraction of 10 per cent. obtained by distillation should be dextrogyrate. The following figures were obtained from various specimens of oil distilled in Leipzig:—

—	Yield, per Cent.	Specific Gravity.	Rotation.	Rotation, First 10 per Cent. of Distillate.	Solubility in 80 per Cent. Alcohol.
			° ′	° ′	
1	1·40	0·913	+ 4 16	+ 1 30	1 in 1·2
2	1·70	0·909	+ 3 40	+ 1 22	1 „ 5·5
3	1·50	0·910	+ 5 56	+ 6 26	1 „ 5·0
4	1·73	0·904	+ 6 51	+ 7 49	1 „ 9·0
5	1·75	0·906	+ 8 52	+ 8 24	1 „ 0·7

The oils numbered 1 and 2 were found to contain 5·4 and 5·8 per cent. respectively, of bornyl acetate, with 10 and 18 per cent. of borneol.—J. O. B.

Cassia Oil, Note on Analysis of. J. O. Braithwaite. Pharm. Jour. 1897, **59**, [1428], 597.

Attention is directed to the importance of carefully sampling this oil before submitting it to analysis for the aldehydic value, since it is well known that a wide variation in quality may occur in different bottles in the same consignment. Notwithstanding this, the practice appears to exist of giving analytical certificates on samples drawn from a limited number of bottles, which are erroneously taken to represent the aldehydic value of a large quantity of the oil. An original case of cassia oil, sold upon analysis as assaying from 80 to 85 per cent. of cinnamic aldehyde, was found to give 66, 78, 77, and 77 to 78 per cent. of that body respectively, from the four bottles it contained. The mean aldehydic value of this particular case of oil fell therefore just below 75 per cent.

Purchasers of this oil should stipulate, when buying on the basis of analytical results, that the oil actually purchased should have been examined, or else the whole consignment should be "worked" previous to analysis or should be efficiently sampled.—J. O. B.

Capsicum, Active Principle of. J. Morbitz. Pharm. Zeits. für Russ. 1897; through Pharm. Wochenschr. 1897, **14**, [33], 525.

Capsicum fruits and powder were first freed from fat with petroleum spirit and then extracted with ether. The residue obtained on distilling off the ether was saponified with just sufficient solution of KHO, then again extracted with ether, and the solvent removed. The ethereal residues thus obtained were repeatedly extracted with hot petroleum spirit, from which, on cooling, small crystals separated, which the author regards as the acrid active principle of the fruit, and which he terms capsacutin. The yield is only between 0·05 and 0·07 per cent. on the powdered drug. Capsacutin is neither glucoside, acid, nor alkaloid. It is extremely acrid, the taste being still detected in a dilution of 1 in 11,000,000.—J. O. B.

Toxicodendrol: The Active Principle of Rhus toxicodron and Rhus venenata. F. Pfaff. Jour. Exper. Med., March 1897; through Med. Chron., Aug. 1897, New Ser. **7**, 377.

The author isolated the active principle, toxicodendrol, by extracting the plant with alcohol, distilling off the solvent, and treating the residue with ether, after washing it with water. On evaporating the ether, an oily residue was obtained, which was freed from resin by means of alcohol. By precipitating the alcoholic solution thus obtained, with an alcoholic solution of lead acetate, a compound of lead with the active oil was obtained. This, when decomposed with ammonium sulphide, liberated toxicodendrol as an oily substance possessing intensely irritating properties. It was found in all parts of the plant, the fruits giving 3·6, the leaves 3·3, and the stem 1·6 per cent. The degree of activity varied in the case of different persons; in some cases 1 mgrm. of the oil produced hundreds of vesicles, and even $\frac{1}{1000}$ of a mgrm. proved active in one case. The vesicant action was not immediate, a period of 24 hours to 7 or 9 days elapsing before the eruption appeared.
—J. O. B.

Dioscorine: Toxic Alkaloid from Dioscorea hirsuta. H. W. Schuette. Chem. Centr. 1897, **2**, 131; through Pharm. Zeits. für Russl. 1897, **36**, 379.

The author has extracted this alkaloid, which was first isolated by Boorsma in 1889, by the following process:—

The dried tubercles were extracted by alcohol acidulated with HCl, fatty matter being precipitated by the addition of water, and the clear filtrate so obtained was evaporated to a syrup. This was made alkaline with KOH, and the liberated alkaloid shaken out with $CHCl_3$. On distilling off the solvent, the residue was neutralised with HCl evaporated to dryness, dissolved in alcohol, and the hydro-

chloride purified by repeated crystallisation from that solvent. The base liberated from this salt was obtained as a greenish fluorescent syrup, which slowly crystallised in well-formed yellowish crystals, which melted at 43·5° C. These crystals were very hygroscopic, readily soluble in most solvents, except ether, benzene, and petroleum ether, and were insoluble in CCl_4. Dioscorine was found to be a powerful base, liberating ammonia from its salts; it reacted with litmus, and was slightly volatile. It was precipitated by most of the alkaloidal reagents. With H_2SO_4 and KIO_3 (pure) it gave a reddish-brown colour, quickly turning to purple; with sodium nitroprusside and an alkali it gave a reddish-violet colour. The hydrochloride $C_{13}H_{19}NO_2HCl + 2H_2O$ crystallised in two forms—either in needles or tablets. It lost its water of crystallisation at 37·7° C., and the anhydrous salt melted at 95·5° C. The specific rotation of the aqueous solution was $[\alpha]_D = + 4° 40'$, and the refractive index of a 1·75 per cent. solution $N_D = 1·3376$. Dioscorine platinum hydrochloride, $(C_{13}H_{19}NO_2)_2H_2PtCl_6$, formed orange tablets, and melted at 92°—93° C. The aurochloride $C_{13}H_{19}NO_2HAuCl_4.\frac{1}{4}H_2O$ occurred in yellow arborescent crystals, melting at 77·2° C.

The action of dioscorine on the animal organism was found to resemble that of picrotoxin. In toxicological examination it may be met with in the benzene solution from an alkaline medium, and by Stas-Otto's process is extracted by the alcohol.

Dioscoreine, described by Boorsma as accompanying dioscorine, is stated by the author to be identical with that alkaloid.—J. O. B.

Cosaprine: A New Antipyretic. Z. von Vámossy and B. Fenyvessy. Therap. Monatsh. 1897, **11**, [8]. 428.

Cosaprine is a sulphonic derivative of antifebrine—

$$(C_6H_4)(SO_3Na)(NH.CO.CH_3),$$

and occurs as a greyish-white amorphous powder, readily soluble in water, having a slight saline taste and an acid reaction. As an antipyretic, it is more active than antifebrine, and at the same time the toxicity is very materially reduced.—J. O. B.

Phesine: A New Antipyretic. Z. von Vámossy and B. Fenyvessy. Therap. Monatsh. 1897, **11**, [8], 428.

This newly introduced antipyretic is a sulpho-derivative of phenacetin. The constitution is given as—

$$C_6H_3(O.C_2H_5)(SO_3Na)(NH.CO.CH_3).$$

It is a light reddish-brown, amorphous, odourless powder, with a slight acid and saline taste. It is very readily soluble in water, giving a Bismarck brown solution with a slightly acid reaction.—J. O. B.

Menthol in Peppermint Oil, A Rapid Method of Approximately Estimating. C. Kleber. Pharm. Review, 1897, **18**, 135; through Semi-Annual Report of Schimmel and Co., Oct. 1897.

See under XXIII., *page* 1050.

Clove Oil. E. Erdmann. J. Prakt. Chem. 1897, **56**, 143.

See under XXIII., *page* 1050.

Formaldehyde, Vaporised, for Disinfecting Purposes; Estimation of. P. Strüver. Zeits. Hyg. 1897, **25**, 357.

See under XXIII., *page* 1049.

Mercuric Salts, Determination of Mercury in. L. Vanino and F. Treubert. Ber. **30**, [14], 1999.

See under XXIII., *page* 1043.

PATENTS.

[*Ionone.*] *New Chemical Substance suitable for Perfumery, Confectionery, and the like; Manufacture of.* J. C. W. F. Tiemann, Berlin, Germany. Eng. Pat. 8736 (2nd Edition), May 1, 1893. (See also this Journal, 1894, 272, 417, 903, 972.)

By shaking together for several days a mixture of citral, acetone, and baryta water, extracting with ether, distilling off the solvent, fractionating the residue, collecting the part boiling at 138°—155° C. under 12 mm. pressure, and distilling off in a current of steam any unchanged citral and acetone, a substance is obtained which, after purification by distillation *in vacuo*, consists of "pseudo-ionone," a ketone boiling at 143°—145° under 12 mm. pressure, and having the density 0·904. If pseudo-ionone be heated on an oil-bath with sulphuric acid, glycerin, and water, it is converted into ionone, which may be separated from the reaction mixture by extraction with ether and fractionation of the extracted portion, and further purified by conversion into its phenylhydrazine compound. Ionone, $C_{13}H_{20}O$, boils at 128° under 12 mm. pressure, and has the density 0·935. The patentee claims pseudo-ionone and ionone as new products of manufacture, and the processes for their preparation as described.—A. C. W.

Violets, Artificial Essence or Essential Oil of: A New or Improved Process for the Manufacture of. F. Fritzsche and Co., Hamburg-Ohlenhorst, Germany. Eng. Pat. 26,350, Nov. 21, 1896.

An essential oil, smelling of violets, boiling at 142°—150° C. under 12 mm. pressure, and of density 0·948—0·952, is claimed as a new article of manufacture. This oil differs in physical properties from ionone (see preceding abstract). The patentee claims the process for the manufacture of artificial essence of violets, which consists in heating together lemon-grass oil, alcohol, acetone, and solutions of hypochlorites (bleaching powder not containing free lime), with or without the addition of cobalt nitrate, and the conversion of the oil so obtained, which boils at 155°—175° under 12 mm., into an oil of violet-like odour by heating with sodium bisulphate.—A. C. W.

Menthol, Impts. in the Preparation and Application of. H. Helbing, London. Eng. Pat. 25,778, Nov. 16, 1896.

According to this invention, the menthol is dissolved in ethyl chloride or methyl chloride, with the addition of alcohol, in the proportion of one part of menthol, one part of alcohol, and 18 parts of ethyl chloride. This solution is kept in glass or metal vessels, provided with a small orifice, which is closed with a tightly fitting cap. On removing the cap, the solution of menthol is ejected in the form of a fine spray.—W. P. S.

Coating and Insulating Materials for Medical and Other Purposes, Impts. in the Preparation and Application of. H. Helbing, London, and G. Pertsch, Lyons, France. Eng. Pat. 25,779, Nov. 16, 1896.

This invention relates to the preparation and application of coating solutions (adhesive plaisters and collodions). For this purpose, solutions of various gums, fatty matters, wax, gutta-percha, or nitro-cellulose are dissolved in a large excess of methyl or ethyl chloride (with or without the addition of alcohol or ether). These solutions are placed in glass or metal receivers, having small orifices, which are closed with tightly fitting caps. If necessary, iodoform or other soluble antiseptics may be incorporated in the solutions.—W. P. S.

Acetonalkamines, The Manufacture of Isomeric Unsymmetrical, and Acidyl Derivatives therefrom. Chemische Fabrik auf Actien vormals E. Schering, Berlin, Germany. Eng. Pat. 26,666, Nov. 24, 1896. (See this Journal, 1897, 758, 822.)

The production of isomeric unsymmetrical bases of the acetonalkamine series by separating by crystallisation the mixture of unstable and stable alkamines formed by reduction of the unsymmetrical acetonamine bases, the conversion of these alkamines into the stable form by the action of alkylates, and the production of acidyl derivatives from the stable and unstable forms by replacing the hydroxyl hydrogen by acid radicals (benzoyl, toluyl, mandelic acid, &c.), are claimed, together with the compounds mentioned, as new chemical products.—A. C. W.

Guaiacol and Creosote, Impts. in the Manufacture of Preparations of. G. B. Ellis, London. From La Société Chimique des Usines du Rhône, formerly G. P. Monnet

et Cartier, Lyons, France. Eng. Pat. 27,527, Dec. 3, 1896.

THE substances claimed are the phosphites of guaiacol and creosote, prepared by either of the following methods:—

(1.) Three molecular equivalents of guaiacol or creosote are dissolved in an alcoholic solution of caustic soda or potash, containing a sufficient amount of alkali to form the salts of guaiacol or creosote. To this mixture is slowly added one molecular equivalent of phosphorus trichloride, keeping the solution cool during addition. The mixture is then heated to boiling, and when all the alcohol has evaporated, any uncombined guaiacol or creosote may be removed by washing with aqueous alkali. The residual phosphorous acid ester is then purified by recrystallisation.

(2.) 372 parts of guaiacol are mixed with 137 parts of phosphorus trichloride and heated to 200° C., until no more hydrochloric acid fumes are given off. After cooling, the residue is treated with caustic soda, extracted with ether, and, finally recrystallised from alcohol. The yield by this method is not as large as by the former one.

The guaiacol phosphite obtained forms a white crystalline powder, with a faint odour of creosote. It is readily soluble in alcohol, ether, chloroform, and fatty oils; soluble with difficulty in water, glycerin, benzene, and acetone. Melts at 77·5° C.

Creosote phosphite is a yellow syrupy liquid with a faint odour of creosote.—W. P. S.

[*Saccharine.*] *Benzoic Sulphinide, Impts. in the Manufacture of.* La Société Chimique des Usines du Rhône, Lyons, France. Eng. Pat. 27,655, Dec. 4, 1896.

THE process for the manufacture of saccharine and its stages are claimed, which consist in heating o-benzaldehyde sulphonic acid with phosphorus pentachloride and oxychloride, distilling off the oxychloride, heating the chlorinated compound with ammonia under high pressure after recrystallisation, acidifying the contents of the autoclave and extracting with ether.—A. C. W.

Toluene, Chlorine Derivatives of; Impts. in the Manufacture of. La Société Chimique des Usines du Rhône, Lyons, France. Eng. Pat. 29,717, Dec. 24, 1896.

WHEN *o*- or *p*-toluenesulphonic chloride is distilled alone at the ordinary pressure, sulphur dioxide and *o*- or *p*-chlorotoluene are obtained. The decomposition takes place more easily and at a lower temperature if chlorine be passed through the sulphonic chloride. The patentee claims the process for preparing chlorotoluenes, which consists in passing chlorine through heated o- or *p*-toluene sulphonic chlorides; the manufacture of o-chlorobenzalchloride, $C_6H_4Cl.CHCl_2$, by passing the current of chlorine at 150°—200° until 2 mols. of chlorine are absorbed, distilling the product, and collecting the fraction 225°—235° C.; the manufacture of *p*-chlorobeznalchloride or *p*-chlorobenzochloride by the similar action of chlorine on the *p*-sulphonic chloride, followed by fractionation.—A. C. W.

Orthotoluene Sulphonic Acid and Thio-orthobenzoic Acid [*for Saccharine*], *a Process for the Preparation of, by Oxidation of Thio-orthocresol.* J. Hauff, Stuttgart, Germany. Eng. Pat. 10,810, April 30, 1897.

THE above acids forming the raw materials used for the preparation of the pure benzoyl orthosulphonic imide have not hitherto been obtained on a large scale in a direct manner and pure form. It has been found that on treating thio-orthocresol, prepared according to the methods described by Leuckart (J. Prakt. Chem. 1890 [2], **41**, 186) and Lustig (Gazzetta, **21**, 213), with suitable oxidising agents, preferably potassium permanganate, orthotoluene sulphonic acid is formed in the first place, whilst on further oxidation, the methyl group is transformed into the carboxyl group with formation of thio-orthobenzoic acid.

For the preparation of orthotoluenesulphonic acid, 12 kilos. of thio-orthocresol are mixed with 13 kilos. of caustic soda (40 per cent. NaHO), 32 kilos. of potassium permanganate, and about 100 litres of water. The solution is heated until the red colour has disappeared; it is then filtered, neutralised, and evaporated to dryness. For the manufacture of thio-orthobenzoic acid, the following proportions are used. 12 kilos. of the thiocresol, 14 kilos. of caustic soda (40 per cent.), 64 kilos. of potassium permanganate, and 2,000 litres of water. The treatment of the solution is the same as in the previous case.

It is obvious that this synthesis can be applied to the halogen-sulpho- and nitro-substitution products of the thio-orthocresol substituted in the benzene nucleus, whereby the corresponding substitution products of ortho-toluenesulphonic and thio-orthobenzoic acids are obtained.—H. B.

Iron for Medicinal Purposes, and Solvents therefor; the Manufacture of Improved Preparations of. E. Dolle, Bremen, Germany. Eng. Pat. 15,178, June 21, 1897.

THE iron specific forming the subject of this invention contains iron pyrophosphate in a suspended state, which is obtained by the addition of a solution of sodium pyrophosphate to a solution of diluted ferric chloride, the sodium chloride being removed as it forms. As this preparation of iron is insoluble and is only dissolved in the stomach at the expense of the gastric juice, a solvent is provided consisting of a mixture of sodium pyrophosphate, sodium bicarbonate, and tartaric or citric acid, with sugar or saccharine, and a flavouring material, as citron oil. The iron preparation may be flavoured with suitable extracts. The solvent is, of course, taken into the stomach after taking the iron milk. Molasses and alcohol, when added, serve to hold the iron precipitate in suspension, and to enhance the "keeping" properties of the preparation.
—W. P. S.

Hexamethylenetetramine and its Chloral, Bromal, and Butylchloral Derivatives, Impts. in the Process for the Preparation of. L. Lederer, Munich, Germany. Eng. Pat. 17,693, July 28, 1897.

FORMALDEHYDE and ammonia unite to produce hexamethylenetetramine. The patentee finds, in contradiction to Wohl (Ber. **19**, 1842) that the solution obtained is not decomposed by evaporation under diminished pressure. He therefore claims the process which consists in heating under diminished pressure aqueous solutions of formaldehyde and ammonia or ammonium carbonate, the purification of the product by extraction with alcohol, the process for preparing the chloral, bromal, or butylchloral derivatives, which consists in bringing formaldehyde, ammonia, or ammonium carbonate, and the chlorinated aldehyde into reaction, and the processes for separating these derivatives from their solutions, which consist in evaporation in a partial vacuum or in saturation by a neutral salt.—A. C. W.

XXII.—EXPLOSIVES, MATCHES, Etc.

PATENTS.

Signals, Charges of Detonating Railway Fog; Impts. in the. T. Jenkins, Birmingham. Eng. Pat. 26,819, Nov. 26, 1896.

THE object of this invention is to dispense with the use of percussion caps and anvils or like ignition devices, and also to wholly or partly replace the ordinary powder charges by explosive charges capable of being exploded when struck or crushed.

The explosive mixtures suggested are potassium chlorate, sulphur and fine sand, or chlorate, red phosphorus, sodium bicarbonate and glue, or other chlorate mixtures.

The details of arranging the signals are given in the specification, which is accompanied with drawings.—W. M.

Explosives, Impts. in. G. Kynoch and Co., Ltd., and A. T. Cocking, Witton, Warwickshire. Eng. Pat. 28,889, Dec. 16, 1896.

THE object is to produce a "safety" explosive, and the claim is for the "combination with nitroglycerin of barium nitrate or other similar nitrate, wood meal, and ammonium oxalate, or other similar oxalate, with or without the admixture of sodium carbonate."—W. M.

Sulphuric Acid [Spent Acid in Nitroglycerin Making], Impts. in Purifying and Concentrating, in part applicable to the Manufacture of Nitric Acid. G. Kynoch and Co., Ltd., and A. T. Cocking, Witton, Warwick. Eng. Pat. 28,891, Dec. 16, 1896.

The weak sulphuric acid resulting from the manufacture of nitroglycerin is placed in a strong cast-iron still, set over a fire, and connected to a cooling coil, communicating with a series of Woulff's bottles, the last of which is connected with an exhaust pump. By the aid of heat and the partial vacuum obtained, nitrous fumes are disengaged from the acid and collected in the bottles. The process is continued until the acid is concentrated, when sodium or potassium nitrate may be added, if it be desired to obtain nitric acid, and the process is recommenced, thus avoiding the removal of the sulphuric acid from one vessel to another. —E. S.

Powder Gases at various Points in the Lengths of Gun-barrels, Improved Apparatus for Measuring the Pressures of. J. Polain, Liège, Belgium. Eng. Pat. 12,792, Jan. 2, 1897.

See third abstract below.

XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

Filter Papers, The Reaction of. L. Magnier de la Source. Rev. Intern. falsific. **10**, 165; Chem. Centr. 1897, [2], 909.

The author confirms the observation of Gans (Zeits. für angew. Chem. 1889, 669), that if, in the course of analysis, a boiling solution of potassium bitartrate be filtered through ordinary filter paper, the results of the estimation of the former will be too high. In order to account for this fact, the reactions of several kinds of filter paper were tested, and it was found that some were of an acid and some of a basic character. In the case of the latter, a certain quantity of acid is retained by the filter paper and cannot be removed by washing. A compound of the acid with cellulose is formed and has been separated by the author. In the estimation of acid tartrate of potassium, it is recommended to use a filter paper which has been well washed with boiling water.—A. S.

PATENTS.

Firedamp in the Atmosphere of Mines and other Places ["Gas Battery"]; Improved Method of and Apparatus for Detecting and Indicating the Presence of. E. L. Mayer, London. Eng. Pat. 23,822, Oct. 26, 1896.

"1. The . . . method of detecting and indicating the presence of firedamp in the atmosphere of mines and other places by causing such firedamp to generate an electric current, by means of a gas battery or gas cell [platinum electrodes preferably coated with black or spongy platinum, dipping in electrolyte of dilute sulphuric acid, '4 parts of water to 1 of acid'], and utilising such electric current by means of a galvanometer or equivalent apparatus, capable of being actuated by a weak electric current, to produce a signal, or to control, either directly or indirectly, another electric circuit by which a signal will be produced. . . ." Other six claims relate to apparatus and methods of employment. Eng. Pat. 18,188 of 1895 is referred to (this Journal, 1897, 162).—J. C. R.

Powder Gases at Various Points in the Lengths of Gun Barrels; Improved Apparatus for Measuring the Pressures of. J. Polain, Liège, Belgium. Eng. Pat. 12,792, Jan. 6, 1897.

This specification is accompanied by drawings. The apparatus "consists of a gun barrel having a series of holes bored through the side thereof at right angles to the axis, a sleeve or casing, wholly or partially enclosing the said gun barrel, and having a series of holes formed therethrough, the axes of which are in line with the axes of the holes in gun barrel, and a device applied to each of the said holes in the sleeve or casing and gun barrel adapted to hold a copper cylinder in place, on the head of a piston or plunger subjected to the pressure of the powder gases."

The "device" consists of a perforated plug, a set-screw, and a piston or plunger.—W. M.

Test Papers and Receptacles for same, Impts. in. S. S. Bromhead, 97, Newgate Street, London. From The Chemische Fabrik and E. Dieterich, Helfenberg, Germany. Eng. Pat. 18,096, Aug. 3, 1897.

The test papers are made in ribbon or tape form, either plain or perforated transversely at intervals, and are enclosed in a cylindrical case or box formed with a slot through which the paper can be withdrawn as required.—R. A.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Potassium, Estimation of, by Reduction of the Potassium Platinic Chloride with Sodium Formate. B. Sjollema. Chem. Zeit. 1897, **21**, [74], 739–740.

After describing the inaccuracies and difficulties met with in applying the usual methods for determining potassium, the author gives a method based on that of Corenwinder and Contamine (Bull. de la Soc. Ind. du Nord, 1879). A portion of the solution to be examined, containing 0·5 grm. of substance, is faintly acidified with hydrochloric acid, mixed with platinic chloride solution without previous precipitation of sulphuric acid, and evaporated to a syrup over the water-bath. When the mass is completely cooled, a mixture of 9 parts of a 95 per cent. alcohol and 1 part of ether is poured over it; it is then allowed to stand for some hours, is washed with the same mixture, and transferred to a filter. The double potassium platinic chloride is then dissolved by pouring boiling water over it, and the hot solution is reduced in a porcelain basin with the aid of sodium formate, or, better, the hot chloride solution is gradually poured into the boiling solution of the formate. A glass beaker is to be preferred, however, to a porcelain dish, because, if covered with a watch glass, there is no fear of spirting, and because there are no cracks in the glaze which can retain reduced platinum. With sufficient formate the solution speedily becomes decolorised; it is heated for some time to cause the agglomeration of the platinum and to enable the precipitate to be washed readily. The reduced metal is thrown on to a filter, the beaker being rinsed with dilute acid; the precipitate is finally washed with boiling water, dried, ignited, and weighed. The method is free from most of the usual faults, and is more rapid than those commonly employed, whilst it affords good results. The author prefers to use 90 per cent. alcohol in place of the alcohol and ether recommended by Corenwinder and Contamine.—W. G. M.

Sulphur Chloride, Analysis of. G. A. Le Roy. Rev. de Chim. Indust. 1897, **8**, [94], 294—295.

Sulphur chloride (S_2Cl_2) contains normally 47 per cent. of sulphur, and 52 per cent. of chlorine, but commercial samples often differ widely from this proportion, containing an excess of one or the other element in solution without the physical properties of the substance being sensibly altered. It is, however, essential, in the industries which make use of sulphur chloride (Vulcanisation of rubber; Manufacture of artificial india-rubber; Treatment of ores containing gold, &c.), that its composition should be as normal and constant as possible. The only method of analysing it to be found in the text books is that of Rose, in which the sulphur is estimated by oxidation with fuming nitric acid and precipitation as barium sulphate and the chlorine determined by difference, but the author agrees with Rose in considering this tedious, and liable to error. In place of it he recommends a modification of his volumetric process (Bull. Soc. Indust. Rouen, 1890, 374). From 1 to 2 grms. of the sulphur chloride are pipetted into a weighed quantity of pure benzene, which is again weighed after the addition to obtain the amount of sulphur chloride taken. The mixture is then made up to a definite volume with benzene, and an aliquot portion taken

for the determination. This is shaken with a known volume of soda solution containing 19·37 grms. of Na_2O per litre (1 c.c. = 0·01 grm. sulphur), and the sulphur chloride is gradually decomposed into sulphide, sulphite, hyposulphite, sulphate, and chloride of sodium, with the separation of some free sulphur. This is dissolved by warming the flask on a sand-bath, on which the benzene is subsequently evaporated off. When the sulphur has completely disappeared, an excess of hydrogen peroxide (neutral and free from substances precipitated by silver nitrate) is added, and the liquid warmed, in order to convert the various sulphur compounds of sodium into sulphate. After removing the excess of hydrogen peroxide by boiling, with the addition, if necessary, of a few drops of permanganate, the liquid is cooled, and titrated with normal nitric or sulphuric acid. The amount of soda found free plus that belonging to the sodium chloride subsequently determined, deducted from the original quantity taken, gives the amount of soda converted into sulphate, and this multiplied by 0·01 gives the amount of sulphur. The chlorine is then determined in the neutralised solution by titration with silver nitrate.—C. A. M.

Sodium Sulphide used in Leather-Dressing, Note on the Analysis of the. F. Jean. Ann. de Chim. Analyt. **2**, [18], 341—343. Analyst 1897, [260], 306.

Two samples of sodium sulphide were examined, one—which had produced spots (recognised as due to iron) on doe-skin during the cleansing and plumping processes—giving a green aqueous solution, depositing a sediment of iron sulphide, and the other giving a yellow solution and no deposit of iron sulphide. The method of analysis was as follows:—10 grms. were dissolved in water, and the iron sulphide filtered off. The filtrate was made up to a litre, and the total sulphur determined in 10 c.c. by titrating with $\frac{1}{10}$N iodine solution and starch paste. A second 10 c.c. are mixed with 30 c.c. of water and a quantity of ammonium sulphate solution (6·7 grms. of $(NH_4)_2SO_4$ per litre) equal to the volume of iodine used in the first test and the mixture distilled, the distillate being collected in a receiver containing 2 c.c. of $\frac{1}{10}$N acid. The operation is suspended when the distillate coming over ceases to have an alkaline reaction. The excess of acid in the receiver, after boiling to expel hydrogen sulphide, is titrated with $\frac{1}{10}$N alkali, and each c.c. of acid neutralised by the distilled ammonia, corresponds to 0·0039 grm. of sodium sulphide. The residual liquor in the retort is cooled and titrated with $\frac{1}{10}$N iodine solution (1 c.c. = 0·0079 grm. of sodium thiosulphate). The difference between this and the former iodine titration is calculated as sodium sulphide. If this amount be larger than the amount of sulphide found by the acid titration, the difference represents, in terms of Na_2S, the excess of sulphur present as polysulphide, and may be calculated to sulphur (100 Na_2S = 41·8). The composition of the two samples examined was:—

——	Green Sulphide.	Yellow Sulphide.
Water	63·24	60·32
Sodium sulphide	31·20	28·08
Sulphur in excess	0·53	1·20
Sodium thiosulphate	4·70	10·00
Iron sulphide	0·33	0·40

It was noted in the course of the investigation that (1) when a solution of an alkaline monosulphide is boiled with excess of ammonia, part of the sulphur is volatilised as ammonium sulphide; (2) when sodium thiosulphate is boiled with excess of ammonium sulphate, ammonium sulphide is liberated and sulphur deposited: this decomposition does not take place in presence of alkalis.—A. S.

Sulphuric Acid, Volumetric Determination of Combined. F. Marbontin and M. Molinié. Bull. Soc. Chim. (3), **17**, [22], 950—952 and 953—955.

The authors have determined the corrections to be applied to Windisch's method when, instead of using exactly equivalent solutions of barium chloride and potassium chromate, a slightly stronger solution of the latter is employed. The results obtained by means of this modification agree closely with those obtained by Windisch's method, which has been abandoned, however, on account of the difficulty of accurately titrating arsenious acid with iodine in presence of chromium salts, in favour of the method described in the abstract which follows below:—

To 100 c.c. of the water to be tested, after acidulating with hydrochloric acid and boiling to expel carbonic acid, 30 c.c. of barium chloride solution are added drop by drop. After allowing the precipitate to settle at 40° the acid is neutralised with a few drops of ammonia, the liquid gently warmed, and 30 c.c. of potassium chromate solution added. The volume is made up to 300 c.c., and to 100 c.c. of the clear supernatant liquid, 2 c.c. of sulphuric acid (1 part acid to 3 parts water), and 5 c.c. of arsenious acid solution are added, and the mixture gently warmed and stirred until decolorised. The excess of acid is then neutralised with potassium carbonate and the arsenious acid titrated with iodine. If n represent the volume of iodine solution used for 100 c.c. of diluted water, n' the volume for 100 c.c. of the water to be tested, t the amount of iodine in 1 c.c. of iodine solution, then the quantity of SO_3 per litre, x, $= 10\,(n - n')\,3\,t\,\frac{3 \times 4 \times 127}{4 \times 80}$. A blank experiment is made with distilled water.

The solutions employed contain respectively 4·8 grms. of crystalline barium chloride, 3·9 of crystalline potassium chromate, 4·95 of arsenious acid dissolved in potash and then made slightly acid with sulphuric acid, and 2·54 of iodine per litre. The last is the only one which requires accurate standardisation. The method involves neither filtration nor washing, and impurities in the reagents are said not to affect its accuracy.—E. W. W.

Crucibles, Ground Graphite; Notes on the Assay of. A. F. Crosse. Chem. and Metall. Soc. of South Africa. Proc. at Meeting of Sept. 18, 1897, 11—12.

The common method of roasting the graphite with nitre is unsatisfactory, because of the amount of nitrate required and because a trial assay is necessary. The author prefers to use manganese peroxide, which leaves a residue that is neither reducing nor oxidising in its action. 10 grms. of the finely-crushed crucible are mixed with 35 grms. of the powdered binoxide, and heated to a bright-red heat in a Hessian crucible. The manganese becomes converted into Mn_3O_4, and a solid cake is formed at the bottom of the crucible. The temperature is reduced, and a mixture of 100 grms. of flux (2 of potassium carbonate with 1 of borax and 1 of salt), 50 of litharge, and 2 of flour, and 20 of silica are added. The mass fuses quietly, and when ready, the crucible is cooled and broken up. The lead contains all the gold, and the slag is clean and brown in colour. The average of four such assays should be reported, a single sample being insufficiently representative. Simple roasting of the powdered graphite sometimes suffices for its removal, but the operation is very slow and the results are often lower than those obtained with the aid of manganese peroxide, probably on account of volatilisation in the muffle. Experiments in extraction with aqua regia (even in excess) failed to extract more than two-thirds of the gold. Treatment with bromine water containing liquid bromine led to a solution of the gold in the upper part of the glass vessel used, but reprecipitation occurred lower down, and a colourless solution free from gold was obtained on filtration.—W. G. M.

Iron Ores, Method for Estimating the Reducibility of. J. Wiborgh. Chem. Zeit. 1897. **21**, [75], 750—752.

The reduction apparatus used, consisted of a gas-producer of circular section, 0·25 m. in diameter and 1·2 m. high, in which was suspended centrally an iron tube of 50 mm. diameter; through this the producer gases were passed over the samples to be tested for any required length of time. Wood charcoal was thus burnt with the aid of natural draught, and provision was made for the combustion of the excess gas. The ore to be examined was crushed to pass a sieve of 19 meshes per sq. cm., and was suspended in the reduction tube in baskets of iron wire gauze by means of iron wire. At first 30—40 grms.

of ore were used for each experiment, but subsequently three baskets of kidney shape in section were used, each containing 8—10 grms. of ore, and placed with the others within a larger basket, but separated from one another by thin plates, to prevent any chemical interaction. In this way two different ores might be compared with a standard sample. The temperature was determined by means of metallic alloys and by a thermophone. (This Journal, 1896, 741.) At a distance of 500 mm. from the upper end, the temperature was 400° C., at 900 mm. it was 525°, at 1,200 mm. it was 700°, and at 1,500 mm. 800°—880° C. A bent glass tube was introduced through an opening to remove samples of gas for analysis, which showed that the CO_2 in the gases ranged from 3·2 to 3·6 per cent., and the CO from 30 to 32 per cent. The ore could be gradually or suddenly introduced into the hottest part of the tube, but the former method was preferred, since at 400° C. a separation of carbon was likely to occur, but the same effect could be obtained by retaining the ore at 400° for an hour, and then plunging it direct into the hottest part for another hour. At the end of the experiment, the baskets were cooled in a current of carbonic oxide, to prevent re-oxidation of metallic iron; the ore was then removed and at once tested. But at these high temperatures the ore is so reduced as to contain ultimately, metallic iron and carbon as well as oxide, and therefore requires very careful analysis. The amount of oxygen present after reduction may be taken to indicate the grade of oxidation of the iron. The various tests are thus made :—

(*a*) *Determination of Carbon.*—Särnstrom's apparatus is used, the carbon being burnt by chromic-sulphuric acid to CO_2.

(*b*) *Estimation of Metallic Iron.*—The reduced ore is treated with dilute sulphuric acid, and the evolved hydrogen is collected to serve as a measure of the iron left. From 0·2 to 1·0 grm., according to the degree of reduction, is placed in a test tube and covered with a few c.c. of water; the tube is then closed with a caoutchouc stopper, through which is passed a bent tube and a funnel having a stop cock. The bent tube is connected with the neck of a 200 c.c. bottle, communicating in turn by an india-rubber tube (leading from a tubulure at the bottom) with the bottom of a burette. This bottle contains about 160 c.c. of an alkaline solution to absorb any CO_2 that might be present in the gas, and is immersed throughout in water of constant temperature. When the ore has been placed in the test tube, the apparatus is closed, and the tube is immersed in water at 20° C.; the liquid in the burette is then brought to the same level as that in the bottle, and the reading is taken. The burette is next alternately raised, lowered, and levelled to test the soundness of all the joints. 10 c.c. of dilute (1:8) sulphuric acid is then run from the stop-cock funnel into the test tube, and is allowed to act for an hour at the ordinary temperature, after which the mixture is carefully raised to the boiling-point, the burette being lowered to prevent the pressure becoming excessive. When no more gas is evolved, the tube is cooled first in air and then in a beaker of water at 20° C., and the reading of the burette is taken as before. In this way the difference in volume of gas in the apparatus represents the hydrogen evolved, of which 39·8 c.c. at 0°, or 42·7 c.c. at 20° C. correspond to 0·1 grm. of metallic iron. When much combined carbon is present in iron, the amount of hydrogen corresponding to the iron taken is much reduced, owing to the formation of hydrocarbons, but in these experiments the amount of iron carbide formed is negligeable. A small volume of hydrogen, however, might possibly be produced by the reduction of some of the oxide by its agency.

(*c*.) *Determination of Total Iron* was effected by solution in HCl, evaporation with H_2SO_4, reduction with zinc, and titration with permanganate.

(*d*.) *Determination of Oxidation Grade.*—0·4 grm. is weighed into a test tube provided with a glass stopper having only a minute opening. Here it is treated with 5 c.c. of dilute (1:10) H_2SO_4, with frequent shaking for ½—1 hour until dissolved. When gas evolution has ceased, 5 c.c. of strong (sp. gr. 1·23) acid is added, and the whole is warmed until the iron is completely dissolved, when it is titrated in a beaker with permanganate. With ores difficult of solution, the clear liquid is poured off from any insoluble residue, which is then treated again with strong acid, the two filtrates being mixed before titrating. Thus the total iron in the test is found, but the metallic iron and ferrous oxide are already known, so that the oxidation grade is readily found. Thus, if the total iron be n, the metallic iron r, the ferrous oxide m, the oxidation grade is calculated from the following equation :—

$$\frac{3(n-r)}{2}:(m-r) + 3\left[\frac{(n-r)-(m-r)}{2}\right] = 100:x$$

$$x = \text{oxidation grade}, = 100 \times \left[1 - \frac{(m-r)}{3(n-r)}\right]$$

In this way the oxidation grade of Fe_2O_3 is 100, of Fe_3O_4 is 88·9, and of FeO is 66·7.

(*e*.) *Determination of Reduction Grade.*—As a measure of reducibility (or reduction grade) the quantity of iron reduced is taken, expressed as a percentage of the total iron present in the ore.

From the experiments made it may be concluded that : In proportion as the oxidation grade 77·8 is attained at low temperatures, the ore is easily reducible, *i.e.*, may be reduced by producer gas to metallic iron. This oxidation grade forms a point of equilibrium during the reduction, and metallic iron is not produced until this point is attained. But when reduction has proceeded so far, it progresses rapidly, and metallic iron is formed, while the number representing the oxidation grade gradually falls, without the ferrous oxide forming a second and more marked point of equilibrium as some metallurgists have assumed.—W. G. M.

Ferro-Silicon, Commercial. E. Donath and M. Haissig. Stahl. u. Eisen, 1897, **17**, 670; Chem. Zeit. Rep. 1897, **26**, 205.

Pig-iron, when it contains more than 5 per cent. of silicon, is termed ferro-silicon; commercial samples of this material rarely contain more than 15—16 per cent. of silicon. The analysis of the substance is troublesome, especially in regard to the estimation of the total carbon. Silicon causes the separation of carbon as graphite, but both amorphous and combined carbon are present in the ferro-silicon. Hydrochloric and nitric acids and aqua regia are almost without action upon this material, which, for analytical purposes, must be fused with sodium carbonate and nitre, after which all the constituents, excepting carbon, may be determined. Dilute hydrofluoric acid is a good solvent and leaves the graphitic carbon in such form that it contains 0·36 per cent. of hydrogen and 4·7 per cent. of ash, but gaseous hydrocarbons escape at the same time. A sample containing 14·32 per cent. of Si thus showed 1·43 per cent. of C. Combustion with a boiling mixture of sulphuric and chromic acids, with or without previous treatment in sodium-copper chloride solution, gave numbers agreeing with those obtained by the hydrofluoric method for graphitic carbon. Direct combustion (after finely crushing) with lead chromate, as in elementary analysis, showed 2·79 per cent. of C; and the same result was obtained by the combustion of the residue from chlorine treatment in chromic and sulphuric acids. That the excess of 1·36 per cent. of C thus obtained cannot be present in the form of temper-, carbide-, or hardening-carbon is shown by its behaviour towards various oxidising agents, whilst its solubility in hydrofluoric acid points to its not being present as a carbide of silicon. The authors believe it exists as an unknown iron-silicon-carbide.—W. G. M.

Separation of Metals, Studies on the Electrolytic. A. Waller. Zeits. f. Elektrochem. 1897, **4**, [10], 241—247.

I.—Separation of Cadmium and Zinc.—Neumann by determining the potentials exhibited by these two metals in equivalent normal solutions of their salts has shown that a difference of about 0·345 volt exists. Le Blanc's experimentally determined decomposing values are 2·35 and 2·03 volts for the zinc and cadmium sulphate salts respectively. These confirm Neumann's figures and prove that a separation of the metals is possible by maintenance of the E.M.F.

between these two limits. The following methods are based upon this principle:—

(*a*.) *Deposition from Acid Solutions.*—Heidenreich has recommended the addition of sulphuric and acetic acids, while Smith and Luckow have recommended solutions prepared with acetic acid and sodium acetate. This method yields very correct results, as a comparatively high E.M.F. is requisite to bring any of the zinc down with the cadmium deposit in acid solutions, but it suffers from the disadvantage that the remaining solution must be neutralised before deposition of the zinc is possible.

(*b*.) *Deposition from Solutions of the Double Cyanides.*—This method, proposed by Beilstein and Jawein, has yielded good results in the hands of Freudenberg, but it demands much time and the deposit has a tendency to assume a spongy character.

(*c*.) *Deposition from Solutions of the Double Oxalates.*—This method, first proposed by Classen, has been independently examined by Eliasberg and by the author. Varying proportions of the two metals in the form of oxides were used in these tests (0·08 to 1·2 grms.), and, after conversion into chlorides, 8 grms. of potassium oxalate and 2 grms. of ammonium oxalate were added, and the solution was made up to 120 c.c. for electrolysis. The best deposits of cadmium were obtained at a temperature of 80°—85° C. The E.M.F. at the terminals of the cell must not exceed by more than 0·30 volt the E.M.F. requisite to produce decomposition of the cadmium salt. As the mean resistance of the cell in the author's experiments was = 10 ohms, it follows that a current intensity of 0·03 ampère was the maximum possible, and one of 0·02 ampère was usually employed. The author's results confirmed those of Eliasberg, both the deposits of cadmium and zinc being perfectly satisfactory in character and completeness. The latter metal is deposited from a tartaric acid solution as recommended by Classen after removal of the cadmium. The oxalate method for separating cadmium and zinc is therefore recommended.

II. *Separation of Zinc and Cobalt.*—Neumann has determined the potentials of these two metals when immersed in their salt solutions, and has shown that the difference varies between 0·518 and 0·551 volt. Le Blanc has shown that the decomposing values for the sulphates are 2·35 and 1·92 volts. The differences are sufficiently great to permit the separation to be effected by a method similar in principle to that described for cadmium and zinc. The following methods have been proposed:—

(*a*.) *Deposition from an Ammoniacal Solution.*—This method is recommended by Fresenius and Bergmann. The deposition of the cobalt is satisfactory, but that of the zinc is only possible after the solution has been neutralised and has been made feebly acid.

(*b*.) *Deposition from a Double Oxalate Solution.*—This method is satisfactory for the determination of cobalt alone, but as it is necessary to raise the current intensity in order to separate the last traces of the cobalt, there is great danger of obtaining a deposition of zinc with the cobalt, and the method cannot be recommended.

(*c*.) *Deposition from Solutions containing Tartrates.*—This method has been recommended by Vortmann, but it suffers from the disadvantage that cobaltsesquioxide separates at the anode. The addition of potassium iodide and sodium hydrate, as recommended by Vortmann, does not entirely avoid this evil, but experiments made by Dr. Burckhardt, have shown that the addition of hydroxylamine and sodium hydrate effect what is desired. The author has repeated Vortmann's experiments, and has shown, that if the deposit of oxide upon the anode be weighed, and the weight be included with that of the metal found upon the cathode, the results are satisfactory. The method is therefore recommended.

III. *Separation of Antimony and Tin.*—Neumann has determined the potential of these metals when immersed in acid solutions of their chlorides, and has found a difference of 0·291 volt; but no accurate forecast of the difference in the case of alkaline solutions is possible from these figures. Classen has shown that antimony alone is deposited from the sodium sulpho salt solutions of these metals, and that the tin can be deposited from the remaining solution on conversion into the ammonium sulpho salt. Classen has stated that either strong or weak currents may be used to effect the deposition of the antimony, whereas Ost has stated that tin is deposited with the antimony when the current intensity exceeds 0·30 ampère. The author has calculated the decomposing value of the antimony sulpho salt of sodium from some of Ost's experimental data, and shows that this is about 0·96 volt in the solution at 15° C., and about 0·76 volt at 70° C.

The author has repeated Ost's experiments. In order to obtain good deposits of antimony from the sulpho salt solution, it is necessary to use pure sodium sulphide, prepared according to Classen's method. The weighed portions of the antimony and tin salts were dissolved in 80 c.c. of this solution, from 2 to 3 grms. of pure sodium hydrate in solution were added, and the solution of the mixed salts was diluted to 120 c.c. The electrolysis was carried out at 60° C., with E.M.F.'s varying from 0·65 to 1·60 volts, and with currents ranging from 0·30 to 1·5 ampères in intensity. The results of 16 determinations proved that the decomposition value of the tin sulpho salt of sodium was about 0·75 volt, or 0·40 volt lower than it was assumed to be from Ost's experiments. The author points out that varying amounts of free alkali may be the cause of this difference. In the author's series of experiments it was found that tin was deposited with the antimony at the cathode when the E.M.F. at the terminals exceeded 0·75 volt, and the error caused by this admixture increased with the voltage employed. It is, therefore, necessary, when using this method to effect the separation of antimony and tin, to pay attention, *not to the current strength*, but to the E.M.F. at the cell terminals. This, when using a solution at a temperature of 65° C., must never be allowed to exceed 0·70 volt.—J. B. C. K.

Copper, Quantitative Analysis of Commercial. Hampe. Berg. u. Hüttenm. Zeit. 1897, **56**, 201 and 297.

Estimation of Oxygen.—In reply to Murmann (this Journal, 1897, 359) it is remarked that grease is best removed from copper shavings free from iron, by treatment, for a few moments, with boiling dilute caustic potash; there is then practically no oxidation. It is absolutely necessary to test the bulb-tubes at a high temperature, to ascertain whether their weight remains constant. The cuprous oxide is not at all evenly distributed in copper, and it is necessary to use 80—100 grms. of the metal taken from different parts of the bars, &c. The reduction of varieties of copper containing oxides of other metals besides cuprous oxide, offers no difficulty.

Estimation of Cuprous Oxide.—Thinly rolled, or finely filed copper is treated with cold dilute silver nitrate free from acid. The solution must not contain more than 60 per cent. of silver nitrate, and must be cooled to 0°, when the copper is added, and while the reaction is going on. It is filtered after 24—36 hours. There is no evolution of gas, the process being as follows:—

$$Cu + 2AgNO_3 = Cu(NO_3)_2 + 2Ag, \text{ and}$$

$$3Cu_2O + 6AgNO_3 + 3H_2O = 2Cu_2(OH)_3NO_3 + 2Cu(NO_3)_2 + 6Ag.$$

If the conditions are altered, there is an evolution of gas (nitric oxide):—

$$4Cu + AgNO_3 = 2Cu_2O + Ag + NO, \text{ and}$$

$$4Cu_2O + 5AgNO_3 + 6H_2O = 4Cu_2(OH)_3NO_3 + NO + Ag.$$

Dewey's equation is incorrect, since no nitrous acid could be detected in the solution.

Estimation of Copper.—With regard to the cuprous sulphide method, it is suggested that most of the copper should be precipitated electrolytically, and only the remainder determined as cuprous sulphide.—N. H. J. M.

Mercuric Salts, Determination of Mercury in. L. Vanino and F. Treubert. Ber. **30**, [14], 1999—2001.

If hypophosphorous acid be added to a solution of mercuric chloride in sufficient quantity, the whole of the mercury is rapidly precipitated as calomel. If the acid be in excess, metallic mercury is precipitated; but this can be prevented by the previous addition of hydrogen peroxide.

To 10 c.c. of, say, a 6 per cent. solution of mercuric chloride, an equal bulk of hydrogen peroxide is added, and then hypophosphorous acid till precipitation is complete. The liquid is filtered through a weighed filter and the precipitate washed thoroughly, and dried at 100° C. In the case of mercuric nitrate, a much greater amount of hydrogen peroxide is requisite. This method is accurate, and possesses the great advantage over the precipitation as phosphate, of economy of time to the extent of several hours.—J. T. D.

Colloidal Silver. A. Lottermoser and E. von Meyer. J. Prakt. Chem. 1897, **56**, [17. 18], 2111.

See under XX., *page* 1035.

Phosphoric Acid, Estimation of, by Titration of the Ammonium Phosphomolybdate Precipitate with Standard Alkali. B. W. Kilgore. J. Amer. Chem. Soc. 1897, **19**, 703—711.

The principal objections raised against this method are, that it has not been tried on a sufficient variety of fertilisers and fertiliser materials; and secondly, the difficulty of washing the acid from the precipitates; it has also been noticed that a portion of the precipitate enters into solution. The first objection is disposed of by a series of results obtained by the author with the gravimetric and volumetric methods, which closely agree. A quantity of from 150 to 200 c.c. of water is generally enough to free the precipitates from acid, and it is when large volumes of water are used, that the molybdate precipitate passes through the filter mechanically.

The method was somewhat modified at the last meeting of the Association of Agricultural Chemists in order to avoid the excessive washing above referred to. The substance is, if necessary, incinerated to destroy organic matter, and 2 grms. are dissolved in nitro-hydrochloric acid and the volume made up to 200 c.c. For amounts of phosphate below 5, between 5 and 20, and above 20 per cent., 40, 20, and 10 c.c. respectively of this solution are taken, and 5 to 10 c.c. of strong nitric acid added. The solution is then nearly neutralised with ammonia, heated on the water-bath at 60°—65° C., 50 c.c. of freshly filtered molybdic solution added for each 0·1 grm. of phosphorus pentoxide present, and then digested in a water-bath for 10 to 15 minutes. The clear liquid is quickly decanted into a Hirsch funnel and filtered with or without diminished pressure. The precipitate is washed by decanting twice with about 50 c.c. of nitric acid of 60 per cent., agitating, allowing to settle, and treating once with 50 c.c. of ammonium or potassium nitrate solution. The whole is then thrown on to the filter and washed with 150 to 250 c.c. of water. The filter and its contents are introduced into a beaker, excess of standard alkali and a few drops of phenolphthalein added, and the solution is titrated back with standard nitric acid.—J. L. B.

Phosphates, Valuation of, according to the Amount of Citrate-soluble Phosphoric Acid in them. P. Wagner. Chem. Zeit. 1897, **21**, 905—911.

When a freshly prepared citrate extract is stirred with alkaline citrate and magnesia mixture, the precipitate is quite free from silica, even in the case of highly silicious slags, and it is immaterial whether filtration is proceeded with at once, or after an interval of two hours.

The citrate extract of basic slag changes very slightly in appearance, if at all. When kept for a long time in a covered beaker, the extract does, however, undergo an important change, and solutions which have been kept for three hours, have a tendency to separate silica when alkaline citrate is added. An extract which had been kept for 24 hours, immediately yielded a gelatinous precipitate on addition of alkaline citrate. The separation of silica is much diminished by precipitating the phosphoric acid. When a citrate extract, three hours old, was treated with alkaline citrate, and stirred for half an hour, silica was precipitated. A similar solution, the same age, yielded, with alkaline citrate and magnesia mixture, a precipitate free from silica, and there was no separation of silica in the filtrate when kept for 24 hours.

The phosphoric acid in citrate solutions should therefore, be precipitated as soon as possible, but need not be filtered immediately. Separation of silica from extracts which have been kept, may be avoided by adding the alkaline citrate and magnesia mixture simultaneously, instead of separately.

The citrate-magnesia mixture is prepared by dissolving citric acid (200 grms.) in 20 per cent. ammonia, and diluting to 1 litre with the same ammonia. The solution is then mixed with an equal bulk of magnesia mixture.

To estimate phosphoric acid, 50 c.c. of the extract (which should not have been kept for more than one hour) are treated with 50 c.c. of the citrate-magnesia mixture, stirred for half an hour and filtered at once, or, at any rate, within an hour. The analysis is then finished in the usual manner. —N. H. J. M.

ORGANIC CHEMISTRY.—QUALITATIVE.

Sumac, Adulteration of. W. Eitner. Der Gerber, 1897, 161.

The examination of sumac as to purity is best carried out by means of the microscope. The leaves of *Rhus coriaria*, from which Sicilian sumac is obtained, are covered on both sides, but more particularly the obverse one, with a dense growth of very fine hair, of such a characteristic type as to render the detection of adulteration, even with any of the many other kinds of sumac, an easy matter.—C. O. W.

Carbohydrates, the Identification of. R. Sjollema. Chem. Zeit. 1897, **21**, [74], 739.

It was previously known that, under certain circumstances glucose mixed with solutions of copper salts formed insoluble compounds on the addition of potassium or sodium hydroxide. Glucose gives such a compound with ammoniacal copper sulphate or copper acetate solutions; levulose, on the contrary, gives the precipitate only with the latter. In practice, a 10 per cent. $CuSO_4$ solution is mixed with just sufficient ammonia to re-dissolve the cupric hydroxide, an excess being avoided because of its tendency to re-dissolve the expected sugar precipitate. About 30 c.c. of a 10 per cent. ammonia solution usually suffice for 100 c.c. of the copper liquor. Similarly, a 5 per cent. copper acetate solution is mixed with the exact quantity of ammonia required; in this case the presence of basic or even normal copper acetate necessitates the use of a little acetic acid. Neither of the solutions must smell very perceptibly of ammonia; and either must give a precipitate on dilution or on standing (a condition that is essential to success). The reagent is most satisfactory when it remains clear on the addition of five times, but becomes turbid with ten times its volume of water; and the exact limit of dilution permissible without turbidity, should be known, in order to guard against over-dilution by the sugar solution itself. On the other hand, the use of an excess of the reagent is to be avoided. With very dilute solutions the precipitate may not appear for several minutes or even for an hour, and in this case it is well to make a blank experiment with distilled water in equal volume and in lieu of sugar solution.

With a $\frac{1}{2}$ per cent. solution of glucose, 2 c.c. mixed with $\frac{1}{2}$ c.c. of the ammoniacal copper solution should become turbid at once, and should give a voluminous precipitate within a few minutes. In a mixture of glucose and levulose, the former is recognised by means of the ammoniacal copper sulphate, but the solutions must not be too concentrated. Experiments have not yet been tried to ascertain whether levulose may be detected in the solution from which the glucose precipitate had been obtained and filtered, or whether it would be possible to estimate or separate other carbohydrates by means of copper solutions. Small quantities (even 1 per cent.) of glucose may be detected in cane sugar with the aid of the ammoniacal copper sulphate, and levulose may be similarly detected by the acetate solution. Here, again, concentrated solutions hinder the reaction.

Milk sugar and galactose may give precipitates with copper solutions, the latter showing good results with the acetate, but not with the sulphate solution. Milk sugar gives no result with the sulphate, and not a good one with the acetate. An excess of ammonia is even more fatal to the detection of milk sugar than to that of the hexoses. An acetate and sulphate solution that gave good results with

glucose and levulose, was without effect on milk sugar solutions until 1 per cent. of acetic acid had been added, when it was rendered very suitable, enabling this sugar to be detected even in the presence of much cane sugar. Such a solution is an equally good reagent for glucose and levulose, but it is inconvenient owing to its tendency to become turbid on standing.—W. G. M.

Linseed Oil, Amsel's "Water-Reaction" for. W. Lippert. Zeits. angew. Chem. 1897, 655.

In this article the author gives fuller details of the experiments on which he has based his previous condemnation of Amsel's water reaction. He finds that the aqueous solution of a linseed soap is, and will remain, absolutely clear even in the presence of large amounts (5 or 6 per cent.) of lead, whether the metal has been added to the oil as oxide or as resinate; but that manganese in either form and in far smaller quantities (0·5 per cent. and upwards) causes a distinct opalescence, which appears, if not immediately, at least after a few minutes' standing, and which is proportionate in its intensity to the amount of manganese originally introduced. This turbidity is due to the precipitation of the metallic hydrate; and it does not occur in the case of lead, simply because lead hydrate is soluble in the excess of caustic alkali which is always present after saponification.

Turning to the possibility of detecting rosin, rosin oil, and mineral oil by means of this test, the author finds that a 2·5 per cent. litharge-varnish mixed with as much as 50 per cent. of rosin still gives a clear "water reaction;" and that, as might be expected from Coreil's work (this Journal 1892, 550), rosin oil can only be recognised with certainty when it forms some 15 per cent. of the total varnish. It is curious to note that, in presence of 20 per cent. of either pale or dark rosin oil, the soap solution is at first milky, but becomes clear after five minutes, and finally turns cloudy again in a few hours' time. Mineral oil also only causes a distinct turbidity when it amounts to about 30 per cent. of the oil under examination.—F. H. L.

Cotton-Seed Oil, A Characteristic Reaction of. G. Halphen. J. Pharm. et de Chim. 1897, **6**, [9], 390—392.

Cotton-seed oil, warmed with carbon bisulphide, gives a reddish coloration after some time. To adapt the reaction to analytical purposes, equal volumes of the oil under examination, of amyl alcohol, and of carbon bisulphide containing 1 per cent. of sulphur, are placed in a test tube heated on a boiling brine bath. If in 10—15 minutes a red or orange coloration appear, the presence of cotton-seed oil is proved. Should there be no coloration, 1 c.c. of the same carbon bisulphide is added and the tube again warmed, and if the result be negative this is repeated. The coloration is not equally intense with all varieties of cotton-seed oil, but appears to depend to some extent on the process of refining to which the oil has been subjected. The reaction serves for the detection of 5 per cent. of the oil.—A. C. W.

Formalin in Milk, Detection of, by means of Phloroglucinol. M. Jorissen. Bull. Soc. Chim. 1897, 167; Analyst, 1897, [260], 282.

Ten c.c. of the milk are shaken with several drops of a 10 per cent. aqueous solution of phloroglucinol, and then a few drops of caustic soda or potash are added. A red coloration is produced with milk containing as little as 1 part of formalin in 20,000, whilst normal milk gives no reaction.—A. S.

Oil of Star Anise, Adulterated with Petroleum. J. C. Umney. Chemist and Druggist, 1897, **51**, (913), 623.

In a recent importation of about 600 lb. of star anise oil, five cases were found to contain oil adulterated with petroleum. These had different specific gravities, ranging from 0·894 to 0·939, and the melting point, after congelation, varied from 5·7° C. to 11·5° C., the figures for the specific gravity of the genuine oil being not below 0·980, and the melting point not below 15° C. The petroleum was separated by charring the oil with strong sulphuric acid, when the undecomposed residue had a specific gravity of 0·835 at 15° C. The probable amount of petroleum present in the oils would appear to vary from 37 to 56 per cent. In view of the advance in price of the oil and the active demand prevalent, importers should be on their guard against this form of adulteration.—J. O. B.

Sesamé Oil, Some Substances in; and their Relation to the Characteristic Colour Reaction of the Oil. V. Villavecchia and G. Fabris. Ann. del Lab. Chim. centr. delle Gabelle, **3**, 13—26; Chem. Centr. 1897, [11], 772. (See also this Journal 1894, 69.)

The authors confirm the supposition that Baudouin's reaction for sesamé oil with hydrochloric acid and sugar is due to the furfurol yielded by the latter; and they state that an intense red coloration is produced with sesamé oil, by employing a 1 per cent. solution of furfurol in 95 per cent. alcohol and hydrochloric acid. The substance in sesamé oil which is responsible for this colour reaction was isolated in the following manner:—A quantity of the oil was saponified with alcoholic potash, the alcohol evaporated off, and the soap dissolved in warm water and precipitated with barium chloride. The baryta soap was dried, exhausted with boiling alcohol, the alcoholic solution evaporated to dryness, and from the residue, the following three substances were isolated:—(1.) Sesamin, $(C_{11}H_{12}O_3)_2$, which yields on treatment with nitric acid, two crystallisable compounds melting at 235° C. and 145 C. (2.) Higher alcohol $(C_{25}H_{44}O + H_2O)$ crystallises from alcohol; melting point, 137·5° C.; does not give the furfurol reaction, but forms an acetyl compound (melting point, 130°—131°) insoluble in water, easily soluble in benzene. (3.) A thick odourless oil; easily soluble in alcohol, ether, chloroform, and acetic acid; slightly soluble in alkalis; insoluble in water and mineral acids. This compound is actually the cause of the furfurol action.—A. S.

ORGANIC CHEMISTRY.—QUANTITATIVE.

Nitrogen Determination, Kjeldahl's; A Source of Error in. B. Sjollema. Chem. Zeit. 1897, **21**, 740—741.

To prevent bumping in the distillation of the ammonia from the alkaline solution in the Kjeldahl process, it is generally recommended that some fragments of zinc should be placed in the solution. This is very advantageous; and in applying Jodlbauer's modification it is especially so, as it is ordinarily by no means certain that the ammonia will have been completely distilled over by the time that the bumping of the heavy saline solution commences. The author finds that there is a distinct amount of caustic soda or potash carried over mechanically during the distillation when zinc is employed, and hydrogen is, therefore, evolved if the ordinary bulb-tube (Fig. 1) be employed. This may

Fig. 1.

Fig. 2.

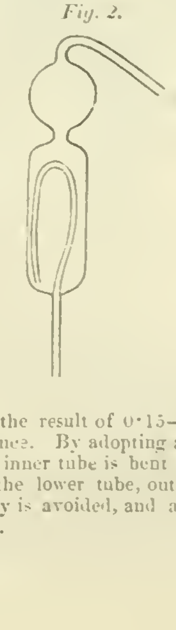
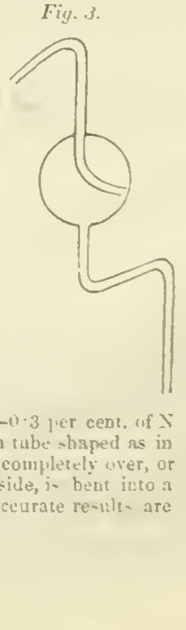


Fig. 3.



cause a variation in the result of 0·15—0·3 per cent. of N on 1 grm. of substance. By adopting a tube shaped as in Fig. 2, in which the inner tube is bent completely over, or as in Fig. 3, where the lower tube, outside, is bent into a Z shape, the difficulty is avoided, and accurate results are obtained.—W. G. M.

Fehling's Solution, False Results obtained with. M. Z. Jovitschitsch. Ber. **30**, [16], 2431—2432.

The author's attention was called to the fact that Fehling's solution is frequently reduced by sulphuric, nitric, and hydrochloric acids, as well as by grape sugar, phenylhydrazine, hydroxylamine, &c., even although the reaction of the solution is so alkaline that litmus paper is coloured distinctly blue. The acids used were found to be chemically pure. If just sufficient of one of these mineral acids is added to 2—3 c.c. of freshly prepared Fehling's solution, so that red litmus paper is still coloured distinctly blue, then, on gentle warming, cuprous oxide is precipitated. The reduction may also take place in the cold, as the following experiment shows:—

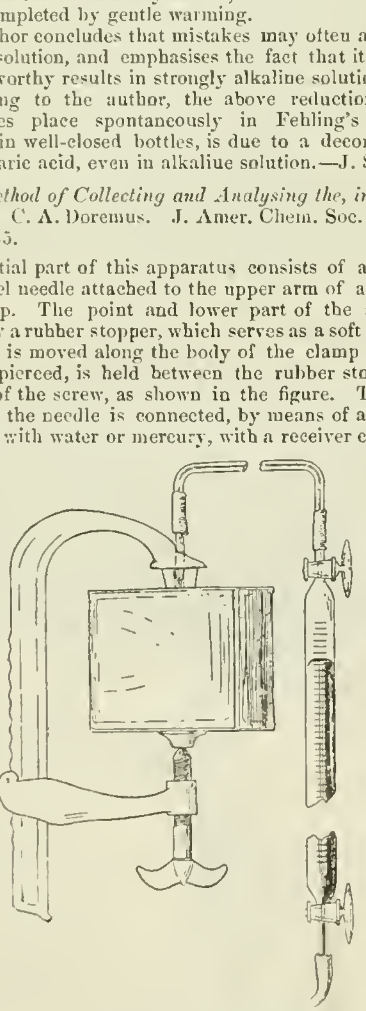
A few drops of one of these mineral acids (not too strong) are added to 1—2 c.c. of strongly alkaline Fehling's solution until only a faint blue colour remains, *i.e.*, until the solution becomes only faintly alkaline. On now adding a few more drops of the original Fehling's solution, an immediate turbidity sets in, and the reduction may then be completed by gentle warming.

The author concludes that mistakes may often arise with Fehling's solution, and emphasises the fact that it can only give trustworthy results in strongly alkaline solution.

According to the author, the above reduction, which often takes place spontaneously in Fehling's solution preserved in well-closed bottles, is due to a decomposition of the tartaric acid, even in alkaline solution.—J. S.

Gases, Method of Collecting and Analysing the, in Canned Goods. C. A. Doremus. J. Amer. Chem. Soc. 1897, **19**, 733—735.

The essential part of this apparatus consists of a bevelled hollow steel needle attached to the upper arm of an adjustable clamp. The point and lower part of the shaft are covered by a rubber stopper, which serves as a soft pad. The lower arm is moved along the body of the clamp until the can to be pierced, is held between the rubber stopper and the head of the screw, as shown in the figure. The upper portion of the needle is connected, by means of a capillary tube filled with water or mercury, with a receiver containing



either of these liquids. When the can is fixed within the apparatus, a few turns of the screw clamps it tightly between the rubber pad at the top and the screw below. The rubber yields to the pressure, making a tight joint around the needle. When the latter pierces the tin, the gases escape gently into the eudiometer.—J. L. B.

Calcium Carbide and Acetylene, Analysis of Commercial, and the Purification of the latter. G. Lunge and E. Cedercreutz. Zeits. angew. Chem. 1897, 651.

Owing to the irregularity in composition of all commercial calcium carbide and the circumstance that it cannot be ground to powder in air without suffering some decomposition, in order to determine the amount of acetylene the carbide will evolve on treatment with water, it is necessary to operate on 50 (or better 100) grms. at a time. This involves the use of a gas-holder having a capacity of at least 40 litres; and the liquid employed as the "seal" should be a saturated solution of common salt, which, in very exact work, must be previously treated with acetylene to prevent any deficiency in the gas measured. If this is done, in reducing the volume of the gas to normal temperature and pressure, it is unnecessary for technical purposes to make any allowance for the vapour tension of the liquid; but if plain water has been used in the tank of the gas-holder, this correction must be carried out. The acetylene is most conveniently generated in an apparatus designed in such a manner as to cause the carbide to fall into the liquid (salt solution) by degrees; but the reverse process may be employed if desired.

The most serious impurity in commercial acetylene is phosphuretted hydrogen, which, like ammonia, appears to favour the formation of the explosive copper acetylide, and which also, if present in sufficient quantity, may render the gas spontaneously inflammable. The phosphuretted hydrogen is best determined by passing the gas as it is given off from the carbide through a solution of sodium hypochlorite, precipitating the phosphoric acid with magnesia mixture. If care be taken that all reagents are free from sulphuric acid, any sulphuretted hydrogen in the acetylene may be simultaneously estimated by acidifying the filtrate from the magnesia precipitate and then adding barium chloride. It should be observed, however, that the amount of sulphuretted hydrogen in different specimens of acetylene yielded by the same carbide, varies considerably according to the conditions obtaining at the time of preparation; and, moreover, the whole of the sulphur in the gas does not exist as hydrogen sulphide. Examined for phosphorus in this manner, two different samples of carbide evolved, as the mean of several tests, 0·1435 grm. (94·1 c.c.) and 0·338 grm. (222 c.c.) of phosphuretted hydrogen per kilo. respectively.

With suitable modifications the process becomes available for the purification of commercial acetylene. The hypochlorite is used in its original form of bleaching-powder, and it is preferably moistened with only sufficient water to make it cohere into balls. In this case the gas must be well cooled before it passes into the purifier, in order that it may not carry too much moisture with it; or it may be dried and freed from ammonia by treatment with sulphuric acid of sp. gr. 1·6 to 1·7, which does not act on the hydrocarbon itself. After the chloride of lime purifier, a vessel containing lime should be employed, so that any solid or gaseous chlorine compounds may be retained.—F. H. L.

Oils and Fats, Animal and Vegetable; Purity of Commercial. D. Holde. Chem. Rev. Fett- u. Harz-Ind. **4**, [20], 271—276.

In determining the iodine number it has been found advisable at the Charlottenburg Versuchsanstalt to allow the iodine solution to act on the liquid fats for 24 hours, with exclusion of light, the results being more uniform than can be obtained by the ordinary two hours' reaction. By this means the differences between the values given by the Hübl and the Waller solution are in most cases reduced to about three units. In the case of Japanese wood oil, however, the difference amounts to 17 units, and consequently it is considered that the use of Waller's reagent should be restricted to such oils as are found, by sufficiently comprehensive experience, to give concordant results with both methods.

Out of 25 samples of fats and oils examined, four were found to be adulterated, three of them seriously.

A so-called "linseed-oil varnish," found in practical use to be deficient in drying properties, proved to be merely raw linseed oil, requiring four days to dry properly. A "spermaceti oil" consisted almost entirely of porpoise oil, and a specimen of "fuller's grease" was found to contain 88 per cent. of unsaponifiable bituminous oil.—C. S.

Unsaponifiable Oil in Greases with a Lime Base, Determination of. H. Bailey. Chem. News, 1897, **76**, 174.

The author failed to obtain satisfactory results with the ordinary method—complete saponification with caustic alkali and extracting the unsaponified oil from the dried soap with a suitable solvent—of determining unsaponifiable oil in greases with a lime base, as prolonged drying was necessary to free the soap from water, and it was difficult to completely extract the unsaponified oil from the pulpy mass.

The following method is recommended as giving accurate results and requiring less time than the ordinary process:—10 grms. of the sample are boiled, with constant stirring, with dilute hydrochloric acid, until the grease is completely decomposed and free from lumps. The oils and fatty acids are filtered off, and the residue is thoroughly washed with boiling water. The filter paper is now broken, the liquid oil washed through by a jet of boiling water, and a concentrated solution of 2 grms. of caustic potash added, together with a little alcohol, after which the solution is heated, with constant stirring, until saponification is complete, preferably with the addition of a little more alcohol to replace that lost by evaporation. Most of the alcohol is evaporated off, about 100 c.c. of water added, and the mixture warmed until clear. Then, after cooling, 30 c.c. of methylated ether or petroleum spirit are added, and the mixture well shaken and allowed to stand. The soap solution is drawn off, and the ethereal layer washed two or three times with water, the washings being added to the soap solution. The soap solution is then shaken with 20 c.c. of ether to remove any traces of oil, and the washed ethereal solution transferred, together with the first ethereal solution, to a small, tared flask with wide mouth. The ether is evaporated off and the residue of oil dried in an air-bath at a temperature of a little over 100° C., until of constant weight.—A. S.

Proteid and Gelatinoid Substances, Improved Method of Determining. A. H. Allen and A. B. Searle. Analyst, 1897, [259], 258–263. (See also Analyst, 1897, 228.)

A quantity of the solution containing about 1 grm. of the albuminoid matter, is made up to about 100 c.c. with water, and acidified with hydrochloric acid. Bromine is then added in considerable excess, and the liquid stirred vigorously for some time. The whole is allowed to stand for half an hour, or until the precipitate has settled, and the liquid is then decanted through an asbestos filter (a cylindrical funnel containing a little glass wool covered with a pad of pulped asbestos). The precipitate adhering to the beaker is washed several times with cold water, and the washings poured through the filter. When most of the free bromine has been washed out of the precipitate, the liquid may not run clear, and it is therefore advisable to keep the washings separate from the filtrate, and, if necessary, to wash with sodium sulphate solution or with bromine water. The contents of the filter-tube are returned to the beaker in which the precipitation was effected, and the nitrogen in the precipitate estimated by the Gunning-Kjeldahl process. It was found that the precipitates produced by bromine in solutions of the proteids are insoluble in water and in dilute hydrochloric acid; but that the extractive matters of meat are only precipitated in concentrated solutions, the precipitates formed being in great part dissolved on dilution, especially in presence of a little hydrochloric acid. The following table gives the results obtained by this method with various proteid and gelatinoid bodies:—

Substance.	Nitrogen per Cent.		Nitrogen multiplied by Factor.		Factor.
	Total in Original Substance.	Precipitated by Bromine.	Total in Original Substance.	Precipitated by Bromine.	
Commercial gelatin	14·10	14·00	77·50	77·0	5·5
Gelatin-peptone	14·10	13·90	77·50	76·5	
Commercial scale albumin	8·80	8·72	55·80	55·2	6·33
Syntonin from scale albumin	9·86	9·76 9·69 9·60	62·41	61·78 61·34 60·77	
Digested scale albumin	8·89	8·81	56·30	55·80	
Fresh white of egg	1·89	1·88	11·96	11·90	
Syntonin from white of egg	1·89	1·89	11·96	11·96	
Peptone from white of egg	0·70	0·69	4·43	4·37	
Beef extractives	0·33	0·004	2·11	0·03	

It is suggested that, by saturating the liquid with zinc sulphate and treating the filtrate with bromine, the real peptone formed in digestion experiments might probably be determined with accuracy. It was found, on applying this method, that Liebig Company's meat extract does not contain any considerable quantities of real peptones.—A. S.

Beeswax, Examination of. K. Dieterich. Chem. Rev. Fett- u. Harz-Ind. **4**, [10], 259—263.

In comparing the hot and cold methods for determining the acid number, the ester number, and saponification value, and the ratio number, the author obtained the following *average* figures in a number of samples of pure yellow wax:—

——	Acid Number.	Ester Number.	Saponification Value.	Ratio.
Hot method	20·07	76·30	95·90	1 : 3·8
Cold "	19·37	70·07	90·80	1 : 3·6

and concludes that the lower values yielded by the cold method are the more reliable of the two, since the lower temperature employed, precludes the possibility of error arising from the formation of decomposition products. The cold method is said to be equally efficient with the hot method in the detection of adulteration, and the results of the examination of several adulterated samples are supplied in the paper.

In a note, Henriques states that he has never noticed the above-mentioned differences yielded by the two methods when applied to beeswax, and only found decompositions occur in the case of bodies (*e.g.*, wool-fat, Chinese insect wax) containing a large proportion of unsaturated alcohols.
—C. S.

Ethyl Alcohol, Highly Diluted [1 *in* 3,000 *to* 1 *in* 10,000]; *Distillation of.* Nicloux and Bauduer. Analyst, 1897, [259], 263: Ann. de Chim. Analyt. **2**, [11], 202—204.

It has been found that on distilling dilute solutions of alcohol, the percentage of alcohol coming over is in inverse ratio to the concentration of the solution. Thus, with mixtures of alcohol and water from 1 in 500 up to 1 in 1,000, the whole of the alcohol is not over when one-third of the solution has been distilled off, but when the concentration is below 1 in 3,000, the whole of the alcohol is obtained in the distillate, when the latter amounts to one-fourth of the original volume of the solution. The alcohol may be estimated by the Nicloux bichromate method by adding a solution of potassium bichromate (19 grms. per litre) and 4–5 c.c. of pure concentrated sulphuric acid, and then comparing the colour with standard samples containing known quantities of alcohol.—A. S.

Spirits, Estimation of the Higher Alcohols in. X. Rocques. Analyst, 1897, **22**, [259], 263; Ann. de Chim. Analyt. **2**, [8], 141—146.

The author has improved the ordinary method of heating the alcohol with sulphuric acid and determining the amount of coloration produced, in that he first fixes the aldehydes by means of an amine. 100 c.c. of the sample, previously distilled and adjusted so as to contain 50 per cent. of alcohol, are boiled gently for an hour with 2 grms. of *m*-phenylenediamine chloride and a few granules of pumice in a flask fitted with a reflux condenser. The mixture is cooled and then distilled, care being taken that no spray passes over during distillation. The operation should be completed in 40 minutes, and the condenser must be long enough (40 ins.) to cool the distillate to the ordinary temperature. 75 c.c. of distillate are collected; this volume contains the whole of the alcohol, and is consequently of 66·7 per cent. strength. 10 c.c. of the distillate are taken, 10 c.c. of pure, colourless sulphuric acid monohydrate added, the mixture shaken, heated for an hour in a brine-bath boiling at 120° C., and compared with a standard solution of 0·667 grm. of isobutylic alcohol per litre, of 66·7 per cent. alcohol (corresponding to 0·5 grm. per litre of 50 per cent. alcohol), prepared and treated in a similar manner. The comparison is made on the cooled liquids in a Dubosc colorimeter, the amount of higher alcohols (per litre of 50 per cent. alcohol) being found and expressed as isobutylic alcohol by means of the formula $\frac{500 \times H}{H_1}$, where H = the stratum of the standard liquid, and H_1 that of the sample. The depth of colour is not exactly proportional to the quantity of the higher alcohols present, the variation between the apparent and actual amounts being as follows:—

Apparent.	Actual.	Apparent.	Actual.	Apparent.	Actual.
1·125	1·000	0·760	0·700	0·379	0·400
1·009	0·900	0·640	0·600	0·255	0·300
0·886	0·800	0·500	0·500	0·150	0·200
..	..	..	..	0·060	0·100
..	..	..	..	0·019	0·050

The delicacy of the reaction is nearly twice as great with the above method, employing 66·7 per cent. alcohol, as in the ordinary method with 50 per cent. alcohol. Several readings should be made in the colorimetric estimation—two with a 10 mm. and two with a 20 mm. stratum—and the mean of the results taken. If the amount of higher alcohols be more than 1 grm. per litre of 50 per cent. alcohol, the sample should be suitably diluted with pure 66·7 per cent. alcohol after, or with 50 per cent. before, being distilled. The method is stated to give constant though not absolute results.—A. S.

Alcohols, Analysis of. E. Barbet. Bull. Assoc. Chim. 1897, **15**, [3], 250—252.

Phenol and sulphuric acid in presence of aldehydes, give a violet coloration; thymol and sulphuric acid give a greenish coloration and coloured compounds with higher alcohols. In view of the fact that quantitative methods based on these reactions have been proposed, the author has experimented, and finds that the phenols act only on the aldehydes, and that this action is so variable, according to the nature of the aldehyde, that no quantitative estimation can be based on it. A mixture of 1 per cent. of pure amyl alcohol and pure isobutyl alcohol in ethyl alcohol equally pure, although of bad flavour, gave no indication with either thymol, naphthol, or hydroquinone.—L. J. de W.

Wine, Determination of Glycerin in. C. Boettinger. Chem. Zeit. 1897, **21**, [67], 658—659.

The method proposed depends upon the conversion of the glycerin into its acetyl derivative, which can then be weighed. The operation is carried out as follows:—About 1 grm. of hydrogen potassium sulphate is put into a small stoppered bottle, and this, with contents, is carefully weighed; the substance to be treated is next introduced, and the whole again weighed; pure acetic anhydride (1—1·5 c.c.) is then added, the bottle is closed, gently shaken, and heated in a water-oven for two hours. The product is treated, when cold, with a few drops of alcohol, and extracted with ether, the ethereal extract being afterwards evaporated in a weighed beaker, and the residue dried at 105°. The author does not regard this method as finally settling the difficult question of the determination of glycerin in wine, but he believes it to be a step in the right direction, and suggests that possibly the formation of some other ethereal derivative of glycerin may prove still more simple.
—A. K. M.

Gutta-Percha. J. A. Montpellier. Rev. de Chim. Indust. 1897, **8**, [94], 300—302.

The author here concludes his account (this Journal, 1897, 925) of gutta-percha, and gives the following scheme for its analysis:—

Estimation of Gutta and Resins.—From 0·5 to 1 grm. of the finely divided sample is placed in a weighed filter-paper, supported by a perforated platinum cone, which is suspended (its point in the liquid) in a flask connected with a Soxhlet apparatus. Here it is extracted with boiling alcohol for five or six hours, after which the cone is transferred to the Soxhlet extractor, and the extraction continued for the same period. The resins (fluavile and albane) dissolve in the boiling alcohol, whilst the gutta and impurities remain in the filter-paper, and are dried at 100° C. in a current of carbon dioxide, and weighed. The impurities are determined in a separate estimation and deducted.

Separation of Albane and Fluavile.—This can only be done incompletely, but an approximate determination of the amount of each may be made by extracting 5 grms. of the sample with alcohol, as above, and evaporating the alcoholic extract until the albane begins to separate out. On cooling, the fluavile remains in solution, and the albane, which has crystallised out, is collected on a weighed filter, dried at a low temperature, and weighed.

Estimation of Water.—One grm. of the gutta-percha is dried at 100°—110° in a current of carbon dioxide for six or seven hours.

Estimation of Impurities.—A weighed quantity (not exceeding 1 grm.) is extracted with boiling chloroform in the same way as in the determination of the gutta with alcohol. This dissolves both gutta and resins, and the impurities left behind are dried in a current of carbon dioxide and weighed.

Ash.—This is determined in the usual manner. Its amount never exceeds 0·5 per cent.

With regard to the water in gutta-percha, the author finds that although at first it lowers the insulating power of the latter, this is only temporary, and the greater the amount of water, the sooner the gutta-percha oxidises on exposure to the air.—C. A. M.

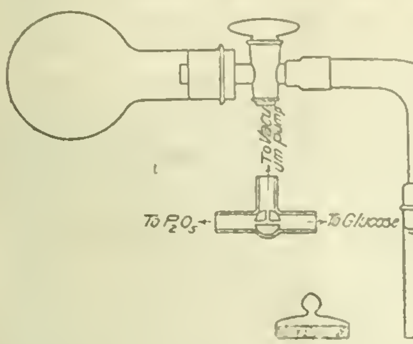
Starch Products, The Exact Estimation of Total Carbohydrates in Acid-hydrolysed. G. W. Rolfe and W. A. Faxon. J. Amer. Chem. Soc. 1897, **19**, 698—703.

An attempt has previously been made by one of the authors (this Journal, 1897, 167) to ascertain the factor to be employed for deducing the solid matter from the specific gravity of any starch product hydrolysed by means of acid. The authors have now endeavoured to confirm these calculated factors by the actual determination of the total solids in a number of representative acid-hydrolysed starch products. For this purpose an apparatus has been devised, which permits of the major bulk of water being removed before coming into contact with the drying agent, and avoiding evaporation on a water-bath.

The tared beaker containing the solution to be evaporated is slipped on the end of the adapter, an air-tight joint being made with "bill-tie" tubing. By the three-way stop cock, communication can be made with the vacuum pump, and with a 250 c.c. flask containing phosphorus pentoxide; or either can be shut off.

The method of drying is as follows:—The cock is opened to the pump only, and the air exhausted to 680—690 mm.,

the beaker being then lowered into an oil-bath and heated to about 100° C. until most of the water is evaporated.



Communication is made with the pentoxide flask, and the residue dried to constant weight at 120° C.

The results were tabulated and plotted. They form a straight line, which is very slightly higher than the line calculated by using the revised carbohydrate values of Brown, Morris, and Millar (this Journal, 1897, 165). Within the specific gravities 1·035 and 1·045, the authors can calculate the absolute specific gravity influence of any acid hydrolysed starch solution by the equation $\Sigma = 0·004023 - 0·000001329(195 - [\alpha]_D)$ when the specific rotatory power (obtained by the factor 0·00386) is known. For commercial glucoses, the factor 0·00393 is sufficiently exact for most determinations.—J. L. B.

Formaldehyde, Vaporised, for Disinfecting Purposes; Estimation of. P. Strüver. Zeits. Hyg. 1897, **25**, 357.

The gases evolved by the lamp, or aliquot portions of the air of the chamber, in which formaldehyde is being volatilised, are passed through two washing flasks containing water, and a third vessel filled with ammonia to act as a check. At the end of the test the latter is treated with silver nitrate, in order to see if any formaldehyde has escaped the water, and, if not, excess of standard ammonia is added to the contents of the first two flasks, and the liquid allowed to rest for 24 hours. It is then titrated with decinormal oxalic acid, or evaporated to dryness and the hexamethylene-amine weighed. The reaction is as follows:—$6CH_2O + 4NH_3 = (CH_2)_6N_4 + 6H_2O$.—F. H. L.

Observations [and Analytical Methods used] in the case of some of the Chemical Substances in the Trunks of Trees. F. H. Storer. Bull. Bussey Inst. 1897, **2**, 386.

The author, after a brief discussion of the broad physiological problem, passes to the record of experimental results, with critical notes upon the analytical methods employed. The main value of the paper is as a contribution to the methods of the agricultural laboratory.

The following are the more important experimental numbers obtained:—

Percentage of Pentosans (= Furfural × 1·84) *present in the Wood of Grey Birch* (Betula populifolia) *at different Seasons.*

——	Inner Wood.	Outer Wood.	Bark.
May	39·2	36·1	30·8
July	(1) 30·5 (2) 30·6	34·7	21·1
October	29·8	30·0	22·7

Percentage of Starch in same Wood and at the same Periods.

May	4·9	5·4	7·7
July	3·8	3·9	7·5
October	3·8	3·5	4·2

The above determinations were made by the "malt..." process recommended by Wiley (Agricultural Analysis, v. **3**, 198), and include any (small) proportion of ready formed sugars present in the wood. Methods based upon acid hydrolysis followed by estimation of cupric reduction were critically investigated, the numbers obtained showing the hemicelluloses and pentosans to be considerably attacked. In the following tables the cupric reduction is calculated to pentosan in the case of the woods and to hexosans for the substances poor in pentosans.

Hydrolysis by the Acid of 2·5 per Cent. HCl, 3 hours' Digestion at 90°—100°. (CuO Reduction calculated as stated.)

——	Inner Wood.	Outer Wood.	Bark.
Grey birch (May)	22·0	25·2	22·9
„ (July)	22·7	22·2	21·5
„ (Oct.)	21·9	(1) 24·5 (2) 23·8	28·9
Alder (Jan.)	25·1	25·6	26·1
Sugar maple (Oct.)	19·6	20·5	..
White pine	12·2		
Apricot stones	23·5		
Peach „	20·8		
Ivory nut	32·3		
Date stones	25·0		

The same series of specimens were then treated under less severe conditions, *viz.*, boiled with an acid of 1 per cent. HCl strength for one hour, and the soluble products similarly estimated. The proportions converted were approximately one-half of those of the above table.

It will be seen, by comparing the numbers in the above table for grey birch with the preceding table giving total pentosans in the same wood, that the hydrolysis of the pentosans, even with the stronger acid, is still only partial. A number of determinations of furfural in the residues from the hydrolytic treatments, confirmed these figures, showing a considerable proportion of "furfuroids" remaining, *i.e.*, resisting the hydrolysis. The subjoined table comprises a typical selection of results:—

Percentage of Furfural calculated to Original Dry Substance.

——	Original.	From Residues of Action of HCl, 2·5 per Cent.	From Residues of Action of HCl, 1 per Cent.
Grey birch-bark, cut in May	16·7	6·5	..
„ „ „ July	11·4	6·.	..
„ outer wood (July)	18·8	8·6	12·9
„ inner wood „	16·6	8·0	..
Sugar maple, outer wood (Oct.)	18·2	4·9	..
„ inner wood	20·9	6·4	..
Alder, outer wood	10·9	..	6·7
Date stones	9·6	4·3	3·1
Apricot „	18·4	6·9	12·8
Peach „	15·0	7·6	8·7

The author next endeavoured to estimate the proportion of wood-gum obtainable from a selection of the specimens above investigated, using the process of solution in cold alkaline lye, and precipitation by acids in presence of alcohol. The following percentages were obtained:—

——	Inner Wood.	Outer Wood.	Bark.
Grey birch (May)	14·3	14·4	..
„ (Oct.)	13·1	13·5	6·8
Sugar maple	..	..	4·5
Alder (Jan.)	19·4	15·0	11·8

But little value is attached to these numbers by the author as the substances so isolated are undoubtedly mixtures.

In conclusion, the paper contains the results of determination of "crude fibre" (Weende method) and cellulose by the method of "fusion" with alkaline hydrate (Lange's method). The imperfections of both methods are discussed and admitted.—C. F. C.

Menthol in Peppermint Oil, A Rapid Method of Approximately Estimating. C. Kleber. Pharm. Review, 1897, **15**, 135; through Semi-Annual Rep. of Schimmel and Co., Oct. 1897, 45.

About 5 grms. of the oil, accurately weighed, are mixed in a flask, furnished with an accurately fitting condenser, with about 5 c.c., carefully measured, of acetic anhydride, and boiled for 30 minutes. In the meanwhile, an equal quantity of the same acetic anhydride is titrated with normal soda solution and phenolphthalein. After cooling the boiled liquid, water is added, and the condenser carefully washed; the whole aqueous solution is then titrated with normal soda. The difference in the number of c.c.'s in the two titrations, multiplied by 0·156, gives the amount of menthol in the oil taken. The disadvantages of this method are the relatively large volume of normal solution used in the titration, and the fact that the required number has to be found by difference in two experiments, which necessarily makes the method less exact; it has the advantage that all the operations can be performed in 50 minutes. —J. O. B.

Clove Oil. E. Erdmann. J. Prakt. Chem. 1897, **56**, 143—156.

On treating clove oil with warm or cold potash, an oily residue is left, which contains oxygen. The boiling point of this substance, freed from eugenol, varies as much as that of clove oil itself, but its specific gravity is lower. The addition of concentrated potash causes a partial crystalline solidification. If the treatment of the residue with potash be repeated, the specific gravity falls further, and approaches that of pure caryophyllene, but, even after continued shaking with concentrated aqueous potash, the oil still contains oxygen, though the substance containing it is removed after a short treatment with alcoholic potash. Clove-stalk oil on treatment with cold aqueous potash leaves only sesquiterpenes. The author found the oxygen-containing substance to be eugenol, and proved that for the most part it was contained in the higher boiling fractions of the clove oil.

The terpene obtained on saponifying clove oil with alcoholic potash, diluting with water, and extracting the phenol with ether, consists of pure caryophyllene, but the great variation in the boiling point of the oily residue from the clove oil, referred to above, is due, not to caryophyllene, but to the aceteugenol.

In Thom's method of valuing clove oil (Verhand. der Gesell. deutsch. Naturf. u. Aerzte, 1891, 184) by determining the amount of eugenol, the latter is converted into benzoyl eugenol, and estimated as such. The author points out that this method is based on the assumption that only free eugenol is present, and shows that none, or only a trifling amount, of the aceteugenol is determined, and that higher results are obtained by previously saponifying the oil completely. After the free eugenol has been determined in the usual way, 5 grms. of the clove oil are saponified by heating over the naked flame for three minutes with 11 grms. of soda-lye (29 per cent. NaOH), after which 10 c.c. of water are added, and the estimation finished as in Thom's method. (Alcoholic potash must not be used in the saponification, or esters will be formed.) In this way, the following results were obtained: —

——	Thom's Method.	Total Eugenol.
Clove oil A.	83·90	85·68
„ „ B.	82·97 82·77	84·84
„ „ C.	80·20	81·90

The difference in these figures corresponds to a percentage of aceteugenol in the oil of from 2·1 to 2·6 per cent.

Specimens of clove stalk oil were found to contain no aceteugenol, and this appears to be a point of difference between the two oils.

On saponifying clove oil with alcoholic potash and titrating the free acid, the result shows a greater amount than corresponds to the aceteugenol as determined above. This difference and the fact that in Thom's method a certain amount of saponification takes place, shows that other compounds must be present. Of these the author detected salicylic acid, but in much smaller quantity than the acetic acid. He considers it probable that the hydroxyl group of the salicylic acid is replaced by acetyl, and that the acetylsalicylic acid forms an ester with the eugenol.

These constituents have but little influence on the odour of clove oil, which is derived from substances in the fractions of lower boiling point. Acet-salicylic-aldehyde was not detected, but furfural was extracted and also identified by the xylidine reaction, the vapour from the oil giving an intense red when brought into contact with filter paper moistened with a mixture of *m*-xylidine and glacial acetic acid, or with aniline acetate. (See Schimmel, this Journal, 1897, 557.) A still more sensitive reaction for furfural is given by a solution of 5 grms. of β-naphthylamine in 20 c.c. of glacial acetic acid, the test being applied by placing several drops on filter paper and adding a drop of the liquid to be tested. Strong furfural solutions give an intense purple colour. Dilute aqueous solutions (1 : 10,000) give the reaction strongly, and it is still recognisable in solutions of 1 in 100,000. By means of this reaction furfural can be detected in cigar smoke. Both clove oil and clove-stalk oil give an intense purple colour.

Characteristic colours are also given by other aldehydes. Citral turns a reddish yellow; salicylic aldehyde and anisaldehyde, a yellow; formaldehyde, propylaldehyde, and valeric aldehyde, extremely faint yellow; acetaldehyde and crotonaldehyde, a somewhat stronger brownish yellow. A yellow colour is also obtained with eugenol.

With regard to the presence of other bodies in clove oil, it is probable that eugenol is not the only phenol in the higher boiling fractions, for the variation in the boiling point of crude eugenol shows that it is not a simple substance, and on distilling crude eugenol with steam, the author obtained a residue of a phenol-like character. —C. A. M.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Phosphorescent Strontium Sulphides, Stability of. J. R. Mourelo. Comptes Rend. **125**, 462—464. (See also this Journal, 1897, 642.)

By exposing samples of phosphorescent strontium sulphide, prepared by various methods, to the combined influence of air and sunlight at 45° C. for three hours, the author found that all undergo more or less decomposition with liberation of hydrogen sulphide and partial oxidation. Impurities, and especially those of an alkaline nature, increase the intensity of the phosphorescence and also, by retarding oxidation, greatly affect the stability of the sulphide. The phosphorescent monosulphide, like the sulphides of the alkalis and alkaline earths, is unstable owing to its tendency to form polysulphides, sulphate, and hydrosulphides. Such decomposition diminishes the phosphorescence only in proportion to the amount of sulphide destroyed and the production of non-phosphorescent compounds.—E. W. W.

Strontium Sulphide, Production of, by the Action of Sulphuretted Hydrogen on Strontium Oxide or Carbonate. J. R. Mourelo. Comptes Rend. 1897, **125**, [20], 775—777.

When sulphuretted hydrogen is passed over strontia or strontium carbonate heated to bright redness in a porcelain tube, strontium sulphide, SrS, is left as a white granular mass, without smell, but decomposed by moisture. If the temperature be not high enough, water condenses in the

porcelain tube, the inverse reaction takes place, and strontium hydrosulphide and hydrate are obtained as secondary products. The sulphide produced according to this method is not phosphorescent, but becomes so on oxidation at a high temperature.—A. C. W.

Sulphuric Acid, Action of Hydrogen on. Berthelot. Comptes Rend. 1897, **125**, [20], 743—746.

Sulphuric acid is constantly employed for drying hydrogen gas, several atomic weight determinations depend on the results of reducing oxides in hydrogen thus dried. If 0·5 grm. of sulphuric acid (boiled), be heated in a sealed tube at 250° C. for six hours with 11 c.c. of hydrogen, all the gas is absorbed. $H_2SO_4 + H_2 = SO_2 + 2H_2O$. A slow current of hydrogen may be passed through the acid for an hour at the ordinary temperature, without the production of an appreciable quantity of sulphur dioxide, but if the contact be prolonged, as for example, in a sealed tube for two months, 75 per cent. of the hydrogen is absorbed. Light has no noticeable effect on the reaction.

If oxygen be also present, the same volume of hydrogen is absorbed, and also some oxygen, but the oxidation of the sulphur dioxide by oxygen appears to proceed more slowly than the reduction of the acid by hydrogen. This is the case at the ordinary temperature or at 250° C. The sulphur dioxide acted upon, is that dissolved in the acid, rather than the gaseous part, for a mixture of hydrogen, oxygen, and sulphur dioxide heated to 100° C for 24 hours suffered no diminution in volume, though at 250° C., some of the oxygen had combined. Hydrated nitric acid, $HNO_3 + 2H_2O$, is not attacked by hydrogen at the ordinary temperature or at 100° C.—A. C. W.

Sodium Iodide. J. C. Umney. Pharm. Journ. 1897, **59**, [1424], 312.

The requirements of the official test for sodium iodide in the British Pharmacopœia require that the substance shall contain 99 per cent. of anhydrous salt, while the directions for its preparation are considered by the author to favour the formation of both the anhydrous salt, crystallising out above 20° C. and the hydrated crystals containing 2 mols. H_2O, which form below that temperature. The German and United States Pharmacopœias recognise this difficulty by requiring the official salt to contain at least 95 per cent. NaI.

A recent examination of commercial samples shows that none approach the official requirements; the amount of NaI present in them ranging from 81 to 91 per cent. It is suggested that, if it be impossible to obtain the anhydrous salt on the commercial scale, a definite statement to that effect should be made, with a view to modifying the requirements of the official test.—J. O. B.

Evolution of Oxygen during Processes of Reduction. K. Frenzel, S. Fritz, and V. Meyer. Ber. **30**, [16], 2515—2519.

In former communications (Ber. **29**, 2549 and 2828; also **30**, 1935) it was shown that hydrogen and carbon monoxide were slowly but completely absorbed by neutral or alkaline solutions of potassium permanganate. On agitating the gas, however, with acidified permanganate solution, the oxidation was also found to be complete, but there was at the same time a considerable quantity of free oxygen evolved.

It has been suggested by Erdmann (Annalen, **294**, 68) that the liberation of oxygen was due to the transitory formation of hydrogen peroxide.

The authors have therefore tried the action of air, hydrogen, carbon monoxide, and carbon dioxide on the following oxidising agents in the dry state, and when necessary at a high temperature:—Potassium permanganate, silver oxide, potassium peroxide, lead peroxide, and barium peroxide. In this way the possible intermediate formation of hydrogen peroxide is excluded. In nearly all cases free oxygen was obtained although the oxidising agent was not heated up to the temperature at which it decomposes.

The apparatus used and the results obtained are given in tabular form.

Potassium peroxide which does not evolve oxygen at the melting point of glass, gives off oxygen when treated with carbon monoxide, carbon dioxide and hydrogen, at the temperature of boiling sulphur.

The formation of the oxygen is explained by the following equations:—

$$K_2O_4 + CO = K_2CO_3 + O_2$$
$$K_2O_4 + CO_2 = K_2CO_3 + O_3$$
$$K_2O_4 + H_2 = 2KOH + O_2$$

Silver peroxide and potassium permanganate when heated below their decomposition temperatures also liberate oxygen.

During the reaction a rise of temperature, and occasionally ignition, take place, and the heat developed in this case is sufficient to evolve oxygen by heating the substance above its ordinary temperature of decomposition.—J. S.

Mercury, Direct Action of Sulphuric Acid upon, at the Ordinary Temperature. Berthelot. Comptes Rend. 1897, **125**, [20], 749—750.

Sulphuric acid brought to its maximum concentration point by boiling, attacks mercury at the ordinary temperature, but without any visible escape of gas. The acid contains mercuric sulphate, and gives up sulphurous acid to a stream of carbon dioxide. A trace of black mercuric sulphide is also formed. The reaction is not to be attributed to foreign metals. The mercury used was very pure, and the phenomenon was repeated when three successive quantities of acid were used with the same sample of mercury. A small percentage of water prevents the reaction.
—A. C. W.

Phosphorus, A Plant Constituent containing, which on Decomposition yields Inosite. E. Winterstein. Ber. 1897, **30**, 2299—2302.

E. Schulze and the author (Zeits. f. Physiol. Chem. **22**, 90) have described a constituent of plant seeds containing phosphorus, which was extracted by a 10 per cent. solution of sodium chloride; the compound could be regarded as a calcium-magnesium salt of combined phosphoric acid. The author has investigated the nature of the organic compound in combination with the phosphoric acid, and for that purpose he treated the powdered seeds of *Sinapis agra* (previously freed from fat) with dilute acetic acid. The extract was carefully purified and isolated as a white amorphous mass. To study its decomposition, the calcium was removed by oxalic acid and the magnesium salt decomposed with fuming hydrogen chloride in a sealed tube at 130°—140° C. From this product, inosite was separated.—J. L. B.

Fermentation of Green Grass, Chemical and Bacteriological Investigation of. O. Emmerling. Ber. 1897, **30**, [14], 1869—1870.

The moist grass was pressed into an earthenware jar the tightly fitting lid of which carried a thermometer and tube to convey the gases evolved. The composition of the grass calculated on the dry substance, is given in the following table:—

——	Fresh Grass.	After Four Weeks.
	Per Cent.	Per Cent.
Wood fibre	26·40	31·36
Ether extract	1·86	3·24
Protein	11·80	9·13
Ash	7·62	8·14
N-free extract	52·32	48·13

The temperature rose after 24 hours, through 26° C., remained constant for some time, and then slowly fell to the temperature of the room. A small but steady evolution of gas took place, this contained 64 per cent. of carbonic acid and 36 per cent. of nitrogen. No methane was found.

The figures given, show that the nitrogen free extract (carbohydrates chiefly), and the protein, decreased, whilst the cellulose, contrary to general opinion, was not attacked. The increase in ether extract is caused by the formation of

organic acids. A small quantity of quinone was isolated from the fermented grass, this was probably formed by the oxidising action of the oxygen enclosed with the grass. In a second experiment, where air was replaced by carbonic acid, no quinone was detected, and much less fermentation took place.

Bacteriological investigation revealed the presence of traces of moulds, chiefly mucor; among bacteria, besides hay bacilli mostly as spores, granulobacteria, several cocci, and in abundance *β-mycoides.* This last is known to energetically decompose albumin, the author also finds that it produces appreciable quantities of lactic acid from glucose, and forms acids from arabinose and xylose, it hydrolyses starch by means of an enzyme. Other lactic acid bacteria could not be found, thus the albumin decomposition and acid formation, must at least in part be attributed to *β-mycoides.*—A. C. W.

Sugar, On the Speed of Reduction of Ferric Alum by. J. H. Long. J. Amer. Chem. Soc. 1897, **19**, 683—698.

The author has previously found (this Journal, 1896, 550 and 663) that certain salts invert cane sugar with simultaneous reduction or other change. When a solution consisting of ferric alum and cane sugar was exposed to the light for six months, the former substance was completely reduced. Attempts were made to measure the rate of reduction of the ferric salt by the change of colour during the reaction, without, however, satisfactory results. If a direct reduction of the ferric salt is brought about by the sugar, the change is one that should easily be followed by a series of electrical conductivity measurements, and experimental evidence is brought forward to demonstrate the correctness of this supposition. The resistances, moreover, obtained in ohms corresponded very closely with those of solutions prepared with sugar, ferrous sulphate, ammonium sulphate, and sulphuric acid, in the proportions which would result from the reduction of the original amount of ferric alum, the resistance of the sugar being not greatly changed by inversion or partial oxidation.

The results of the experiments indicate that the speed of reduction increases regularly from beginning to end, the cause of this acceleration being accounted for by the production of sulphuric acid during the reaction. The speed of the reduction varies also with the amount of sugar in solution.—J. L. B.

New Books.

Manuel d'Analyse Chimique. Appliquée à L'Examen des Produits Industriels et Commerciaux. Par Emil Fleurent, Dès Sc., Professeur remplaçant du Cours de Chimie Industrielle au Conservatoire Nat. des Arts et Métiers. Georges Carre et C. Naud, 3, rue Racine, Paris. 1898. Price 12 frs. H. Grevel and Co., 33, King Street, Covent Garden, W C.

This 8vo volume, bound in cloth, and containing 582 pages of subject-matter, is devoted to the subject of Chemical Analysis applied to technical products. The work is illustrated with 101 beautifully executed engravings, and for index, a carefully classified table of contents is given at the end of the book. The following subjects are treated of:—Introductory Observations.—I. Laboratory Organisation. II. The Reagents: Preparation and Use. III. Taking Samples. IV. Interpretation of the Analytical Results. Part I. Mineral Substances.—Qualitative. I. Bases. II. Acids, &c. Chap. II. Quantitative Analysis of Mineral Substances. Quantitative Analysis of Metallic Products and their Derivatives. Methods of Analysing certain Alloys. Methods of Analysing certain Inorganic Colours. Limes, Mortars, and Cements. Manure Analysis. Part II. Organic Substances. Chap. I.—I. Elementary Organic Analysis. II. Kjeldahl's Method. III. Dumas' Method of Determining Nitrogen. Chap. II.—Study of Soils. III. Complete Analysis of a Manure. IV. Analyses of Vegetable and Animal Products. V. Analytical Study of Fermented Liquors. VI. Analysis of Textiles. VII. Volumetric Determination of Acetone in Denatured Alcohols. VIII. Flash Point of Petroleums, &c.

Subject-Matter Index of Technical and Scientific Periodicals. Compiled by the Order of the (German) Imperial Patent Office. Year 1896. Carl Heymann's Verlag, Mauerstrasse 44, Berlin. Price M. 18. Williams and Norgate, 14, Henrietta Street, Covent Garden, London, W.C. Gustav E. Stechert, 9, East 16th Street, New York.

This important work, of quarto size, is written in German, English, and French. It commences with an Index of cited periodicals, and of the abbreviations of titles afterwards to be used, which fills eight columns. There are 506 pages of subject-matter, *i.e.*, index matter, and these are followed by a specially detailed alphabetical index of the subjects treated of therein. But the index matter itself is also in a certain sense treated alphabetically. The general or principal subjects are alphabetically arranged, whilst the subsidiary titles or subjects known by authors' or patentees' names, follow in due order under these. When, however, a Chemical Substance (such, *e.g.*, as Acetylene) is indexed, its name appears at the head of its class, and then a special classification follows, with headings suited to a Chemical Substance. Thus: "(1) *Properties,*" and then grouped below, the references to the names of investigators with literary sources appended; "(2) *Production or Preparation*"; "(3) *Applications,*" and "(4) *Explosions and Sundries.*" Thus, a person desiring information on Acetylene generally (for he might find it under "*Calcium Carbide*" as well as under "*Acetylene*"), may turn it up first in the alphabetical index at the end. If he be specially anxious for information on the narrower issue of "Explosions in the use of Acetylene," he will find all such reference information in sub-group 4, under "Acetylene," and with the authors' or investigators' names alphabetically arranged. This example will serve generally.

Die Chemisch-Trockene Reinigung von Kleidungsstücken, u.s.w., mittelst Benzin, Tetrachlorkohlenstoff, u.s.w. Ein Hilfs- und Lehrbuch für Chemische Waschanstalten und Kleiderfärbereien. Von L. Abel. Gustav Weigel, Leipzig. Price 1*s.* 8*d.* H. Grevel and Co., 33, King Street, Covent Garden, London, W.C.

8vo volume in pamphlet form, containing 58 pages of subject matter illustrated with numerous woodcuts representing apparatus and machinery used in the processes of "Drycleaning of Articles of Clothing." The text of the work is subdivided into the following sections:—I. The Sorting and Arrangement of the Goods. II. The Reservoir. III. The Cleansing Agents: Petroleum Benzine, Benzol, Toluol, and Xylol. IV. Carbon Tetrachloride. V. The Washing Machines, with single and double covers. VI. The Wet Cleansing. VII. Cleansing of Glacé-, Kid, and Leather Gloves. VIII. Processes for Cleaning and Restoring ordinary Carpets. IX. English Process for ditto.

Trade Report.

EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

Venezuela.

Bd. of Trade J., Dec. 1897, 704.

Regulations for the Importation of Butter and other Fats.

The *Journal Officiel* of the 4th November publishes a notice stating that the Government of Venezuela has recently decreed that the Customs authorities are to disregard all certificates of analysis as to the nature of imported lard or butter which are made by foreign chemists. Importers of the above products must furnish a certificate made out by a Venezuelan chemist, and affixed to the manifest. In places where there is no chemist, a similar certificate must be procured from some competent person appointed by the Customs officials.

The above decree is the result of the importation of various kinds of fat or margarine, subject to a duty of 1·25 francs per kilo., under the name of pure fat and

butter (0·25 per kilo.). It applies especially to fats and butters of American origin, which occupy a preponderating place in the Venezuelan market.

Petroleum in the Dutch East Indies.

Bd. of Trade J., Dec. 1897, 726.

The French Consul at Batavia reports that the production of petroleum in the Dutch East Indies increased from 3,770,200 litres in 1890 to 17,680,400 litres in 1894, to 39,604,600 in 1895, and to 64,104,300 in 1896.

Thus, it is seen the production in 1896 is 18 times as large as in 1890, and since 1894 it may be said that it has almost doubled each year.

Not content with checking the current of foreign importation and with causing Russian and American importations to withdraw in a large measure, the owners of the deposits in the Malay Archipelago, and especially those of Sumatra, have commenced to export to the neighbouring territory. The following figures show the exports in question :—

Year.	Litres.
1890	56,366
1891	46,492
1892	3,912,555
1893	11,037,132
1894	26,587,884
1895	36,330,411

Never before the year 1892 did the foreign sales reach the importance which they attained in that year, with a figure of almost four million litres, and in a period of four years it has increased in a proportion of nine to one. Further information, which the newspapers of Batavia give, is to the effect that new sources, richer still than those of Sumatra, have recently been discovered in Balangan, in the district of Amoentai, on the Island of Borneo.

The Wood-pulp and Cellulose Industry of Norway.

Foreign Office Annual Series, 20,13.

In a report to the Foreign Office by Mr. Dundas, H.M. Consul-General at Christiania, it is stated that wood-pulp and cellulose is one of the most important export industries of Norway, and though of comparatively recent development, it has advanced with marvellous rapidity. For Norwegian wood-grinding factories 1896 was about an average year, and for cellulose the demand all through the year was in excess of the supply. The total exportation of this article in 1896 was 314,2?0 tons (of which 34,8?3 tons were Swedish production), of the estimated value of 854,560*l.*, against 276,047 tons (of which 29,822 tons were of Swedish production) and 748,222*l.* in 1895.

The bulk of the production of this article goes to Great Britain, the next most important customers being France, Belgium, Denmark, and Spain.

Some idea of the share taken by Great Britain in this trade may be formed when it is mentioned that during 1896 she imported pulp to the value of 616,210*l.* out of the total estimated value given above. The following ports were the chief centres for the export of wood-pulp and cellulose in 1896 :—

Port.	Quantity.
	Tons.
Christiania	64,014
Skien	31,662
Fredrikshald	12,107
Drammen	130,543
Arendal	2,773
Larvik	5,142

The number of factories engaged in the production of wood-pulp at the end of 1896 was 62, and two more were in course of construction.

GENERAL TRADE NOTES.

The American Beet-Sugar Industry.

Scient. Amer., Nov 20, 1897, 323.

This autumn, there will be in operation nine beet sugar plants whose daily capacities in tons of beets per day of 24 hours each are approximately as follows :—

Watsonville, Cal.	1,[illegible]
Chino, Cal.	[illegible]
Alvarado, Cal.	[illegible]
Los Alamitos, Cal.	[illegible]
Lehi, Utah	[illegible]
Grand Island, Neb.	[illegible]
Norfolk, Neb.	[illegible]
Eddy, N.M.	200
Rome, N.Y.	200
Total	[illegible]

Last year the seven of these plants that were then in operation produced about 40,000 tons of sugar. What the output of this season will be can only be estimated roughly at this date, but it ought to be between 45,000 and 50,000 tons.

Danger of Explosion and Fire in the Manufacture and Storage of Aluminium Bronze Colours.

Zeits. Oeffentl. Chem.; through Sc. Amer., Dec 4, 1897, 18293.

The author observed repeatedly that aluminium bronze colours oxidised in the settling vessels to aluminium hydrate with evolution of hydrogen. The decomposition by water only took place if the temperature of the rooms in which were the settling vessels rose above 25° C. The evolution of hydrogen begins slowly, but in consequence of the heat of the reaction, becomes very vigorous. As there may be present in a settling vessel 6 kilos. or more of the bronze colour capable of evolving at least 7 cb. m. of hydrogen, there is the possibility of a dangerous explosion by the formation of considerable quantities of inflammable gas in the rooms. By the admission of moisture and heat to packed aluminium bronze, evolution of hydrogen may take place, and if the packing material be inflammable, may lead to fire. Spontaneous combustion of aluminium bronze colours was also caused by friction or blows, due probably to small quantities of fat adhering to the powder. These phenomena were not observed with bronze colours made from copper-zinc or copper-tin alloys.

The Present Production of Petroleum.

Dingler's Polyt. J. 1897, **306**, [6], 143; *through Oesterr. Zeits. f. Berg- u. Hüttenw.*

The present production of petroleum is estimated at more than 181,000,000 hectolitres, of which the United States furnish 101,000,000; Russia, 73,000,000; Austro-Hungary, 2,390,000; Canada, 1,530,000; India, 570,000; and Java, 560,000 hectolitres; the remainder comes from Peru, Roumania, Germany, Japan, Italy, &c. In the great Appalachian coal fields, which furnish 59,000,000 hectolitres out of the total production of the United States, 20—25 springs were recently opened, two of which yield 270 hectolitres daily; a considerable increase in production has also occurred in South California and Wyoming. The greatest increase of production, however, is anticipated from Peru, where the principal occurrence is situated in the district of Paira and extends over more than 180 square myriometers. Of 49 springs which have been sunk since 1892, 44 have proved profitable, some of them yielding 130—140 hectolitres per day.

In Russia, 269 new springs were opened in 1895, at an average depth of 165 metres; 1,371 springs have been bored during the last six years. Of the year's production of 73,000,000 hectolitres, 23,000,000 hectolitres were obtained by simple running off, and the remainder by means of windlass and buckets.—A. S.

Fraudulent Practice in Olive-Oil Trade.

Chem. Zeit. 1897, **21**, 749.

The head of a firm in the olive-oil trade has recently been condemned before the Hamburger Landgericht, and received a very heavy sentence, consisting of imprisonment, loss of civic rights, and a fine, for repeated and extensive frauds. The fraud consisted in having sold for many years as pure, olive oil adulterated with from one third to one-half of cotton-seed oil, and oleïn mixed with mineral oil in similar proportion, the former for use in the textile industries, the latter for the treatment of wool. The mixtures were effected in free ports and passed through the Customs office as pure olive oil (or oleïn respectively), which, when denatured, is exempt from duty. During the tariff war with Spain, Spanish oil was imported as Italian, in order to escape the extra duty. In order to persuade inland purchasers to the belief that it was practically pure olive oil, and to account for its low price, the said firm had also a house in Trieste, through which the purchases were made.—W. G. M.

Quicksilver Production in the United States.

Eng. and Mining J., Nov. 20, 1897, 601.

The quicksilver receipts at San Francisco this year have unusually light; for the ten months ending October 31st they were 14,372 flasks (of 76 lb. each), against 22,709 flasks for the corresponding time last year. This does not imply a corresponding fall in production, however, since it is understood that shipments direct from the mines, not consigned to or through San Francisco, have been larger this year than in 1896. It is well known, however, that there is a falling off in the production of quicksilver this year, as we pointed out previously in giving the statistics of the first six months. The Sulphur Banks Mine in Lake County has closed down, it is said permanently, and the Altoona Mine in Trinity County has been idle on account of the flooding of the workings. An attempt to unwater tnem is now being made, but considerable doubt is expressed as to the success of this.

Outside of California there has been no quicksilver production in the United States this year. The Oregon discoveries, which were reported some time ago, have not been developed, and nothing has been heard from the supposed deposits in Texas. Nor have the new mines in British Columbia proved yet to be of any special consequence. In Mexico, however, there have been some new discoveries of quicksilver, which appear to be of much importance, and we are likely to see a large increase in the production of that country on account of them. In view of the large amount of quicksilver which is used in the metallurgical practice in Mexico, proportionately much more than in the United States, it is a matter of a good deal of interest to the citizens of that republic to obtain the necessary supply from domestic sources; heretofore, notwithstanding the exploitation of three important mines, it has been necessary to import quicksilver from the United States.

The demand for quicksilver in the United States is good, and the price at present is higher than it has been for nearly two years past.

Calcining Ores before Treatment with Cyanide.

Eng. and Mining J., April 12, 1897, 669.

Hitherto, all the ores of the Mercur district in Utah, where the cyanide treatment had the first marked success in the United States, were leached raw. For a considerable period it has been known that in many instances a portion of the gold was lost, but owners were apparently satisfied, so long as operations paid a profit. The model De La Mar plant, now being built, is arranged to calcine all ore and roast the arsenical products. Experiments recently made on clayey La Cigale ore, on the West Dip portion of Mercur camp, show that, by calcining, the ore leaches better in 24 hours than in five days without calcining; and calcining furnaces are to be immediately put in. Like results were had on Sacramento, Sunshine, and other ores. Even the Mercur management now admits that this innovation can be adopted to advantage, though the ores of this mine are more cherty and freer from slimy material than any of their neighbours. In all likelihood, within a year, every cyanide mill of that camp will be equipped with calcining furnaces. By this means also, it is believed that most of the mill tailing dumps can be made to afford a profit, and they will probably be some day reworked. (See also this Journal, 1897, 803.)

English Coal and its Competition with Foreign Coal.

Archiv. für Post und Telegraphie, 1897, 414; *Proc. Inst. Civil Eng.* 1897, **130**, [4], 54.

It is stated that anxiety is being caused in German commercial circles, owing to the fact that the export of coke and coal from England to Germany is increasing more rapidly than the gross English exports of these materials. The gross export in 1895 was 33,112,000 tons, an increase of 38,000 tons over that of 1894, whilst the export to Germany in 1895 was 4,144,000 tons, an increase of 25,000 tons over that of 1894. For the nine months, January to October 1896, the increase in the gross export was only 4 per cent., whilst the increase in the export to Germany during that period was 14 per cent.

The annual consumption of coal and coke per head of population in Great Britain increased 669 lb. between the years 1875 and 1895; the corresponding increase in Germany amounted to 1,980 lb. The production of coal the colonies and foreign possessions of Great Britain is increasing, the total in 1894 amounting to 11,368,000 tons. India produced only 1,320,000 tons in 1883, but in 1895 a total of 4,396,000 tons; the small output of coal in South Africa is also increasing.

It is pointed out that Japan is becoming a serious competitor for the coal trade of East Asia, her output already amounting to 1,400,000 tons. It is stated that the Germans have began to work the coal fields in Amoy, and the Chinese the Nankin fields. The bulk of the Russian output of 8,667,000 tons, is produced by the Don fields.

England supplies almost all the coal used in Italy, 50 per cent. of that used in Spain, and 11—12 per cent. of that used in France; but English coal cannot now be delivered and sold at a profit in the Black Sea. The Scandinavian peninsula obtains 90 per cent. of its coal from Great Britain. Coal can be delivered to the German coast more cheaply from Great Britain than from the German coal fields, and the output of the German pits does not keep pace with the home demand. The author considers that the only way of enabling Germany to compete with England in the coal trade is to lower German freights.

—A. S.

BOARD OF TRADE RETURNS.

Summary of Imports.

Articles.	Month ending 30th November	
	1896.	1897.
	£	£
Metals	1,581,522	1,650,649
Chemicals and dyestuffs	374,377	372,609
Oils	807,822	645,080
Raw materials for non-textile industries.	3,914,085	4,073,889
Tota value of all imports	42,492,369	41,161,877

Summary of Exports.

Articles.	Month ending 30th November	
	1896.	1897.
	£	£
Metals (other than machinery)	2,720,569	3,036,552
Chemicals and medicines	591,998	753,840
Miscellaneous articles	2,682,539	2,848,314
Total value of all exports	18,570,877	19,773,594

Imports of Metals for Month ending 30th November.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
Copper:—			£	£
Ore .... Tons	4,844	6,029	49,861	37,318
Regulus .... „	7,623	5,857	180,512	140,467
Unwrought ..... „	4,621	4,526	227,430	222,[illegible]
Iron:—				
Ore.............. „	277,420	170,277	188,450	339,147
Bolt, bar, &c. ... „	5,293	5,956	43,310	50,081
Steel, unwrought.. „	1,320	1,296	9,270	11,743
Lead, pig and sheet „	12,422	14,167	139,305	182,572
Pyrites .... „	42,493	51,453	71,573	82,278
Quicksilver .... Lb.	16,026	80,250	1,393	7,330
Silver ore........ Value £	..	..	130,232	117,995
Tin .... Cwt.	72,356	41,339	211,802	133,302
Zinc .... Tons	8,289	5,923	139,591	100,796
Other articles ...Value £	..	..	188,993	224,929
Total value of metals	..	..	1,581,522	1,650,649

Imports of Oils for Month ending 30th November.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Cocoa-nut......... Cwt.	27,816	8,887	32,623	12,757
Olive.............. Tuns	919	583	31,185	20,831
Palm .... Cwt.	86,183	76,812	100,556	80,526
Petroleum .... Gall.	18,914,067	16,680,094	381,767	283,434
Seed .... Tons	3,657	3,190	77,840	66,764
Train, &c. .... Tuns	2,167	1,220	39,230	19,434
Turpentine .... Cwt.	41,823	56,816	40,428	63,457
Other articles .. Value £	..	..	104,193	97,877
Total value of oils...	..	..	807,822	645,080

Imports of Raw Material for Non-Textile Industries for Month ending 30th November.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Bark, Peruvian .. Cwt.	3,568	1,115	6,361	2,652
Bristles........... Lb.	398,951	322,300	49,784	38,166
Caoutchouc ...... Cwt.	37,421	34,341	495,490	355,427
Gum:—				
Arabic......... „	4,392	3,695	11,153	8,587
Lac, &c. .... „	10,467	11,267	52,565	35,793
Gutta-percha .... „	3,162	1,780	52,330	15,288
Hides, raw:—				
Dry............ „	27,889	14,495	62,749	116,875
Wet........... „	77,636	60,060	165,603	121,613
Ivory............ „	825	708	33,723	32,565
Manure:—				
Guano......... Tons	687	2,468	2,005	14,120
Bones .... „	2,722	3,178	9,122	12,268
Nitrate of soda... „	8,106	4,460	64,157	32,852
Phosphate of lime „	16,196	20,740	24,620	32,368
Paraffin.......... Cwt.	77,104	83,340	76,481	72,122
Linen rags....... Tons	1,565	2,176	16,637	20,819
Esparto.......... „	13,364	12,729	51,134	54,775
Pulp of wood .... „	25,851	39,403	145,483	193,260
Rosin............ Cwt.	150,905	69,632	40,897	13,688
Tallow and stearin „	147,976	153,330	153,410	152,737
Tar ....Barrels	10,219	13,088	8,074	11,192
Wood:—				
Hewn .... Loads	155,049	204,762	308,491	433,657
Sawn .... „	502,982	556,464	1,083,776	1,285,461
Staves.......... „	10,247	10,222	38,463	39,869
Mahogany ..... Tons	3,860	7,050	35,409	63,153
Other articles....Value £	..	..	945,808	914,511
Total value	..	..	3,914,085	4,073,889

Besides the above, drugs to the value of 98,620*l*. were imported, as against 85,821*l*. in November 1896.

Imports of Chemicals and Dyestuffs for Month ending 30th November.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Alkali............. Cwt.	11,652	16,401	[illegible]	[illegible]
Bark (tanners', &c.) „	22,190	15,011	[illegible]	5,001
Brimstone .... „	56,180	[illegible]	[illegible]	[illegible]
Chemicals...... Value £	..	..	[illegible]	[illegible]
Cochineal .... Cwt.	[illegible]	[illegible]	[illegible]	[illegible]
Cutch and gambier Tons	3,284	[illegible]	[illegible]	[illegible]
Dyes:—				
Alizarin ....Value£	..	..	24,365	[illegible]
Anilin and other „	..	..	10,744	[illegible]
Indigo .... Cwt.	712	771	[illegible]	[illegible]
Nitrate of potash . „	18,780	[illegible]	[illegible]	[illegible]
Valonia .... Tons	[illegible]	[illegible]	8,025	[illegible]
Other articles... Value £	..	..	[illegible]	71,777
Total value of chemicals	..	..	374,677	[illegible]

Exports of Metals (other than Machinery) for Month ending 30th November.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Brass............ Cwt.	10,769	10,454	44,850	46,994
Copper:—				
Unwrought.... „	25,306	42,099	64,055	107,834
Wrought ...... „	22,292	26,525	70,092	82,812
Mixed metal... „	21,021	22,277	49,205	52,425
Hardware...... Value £	..	..	178,320	[illegible]
Implements ..... „	..	..	116,083	128,543
Iron and steel ... Tons	298,972	313,965	1,997,506	2,179,513
Lead .... „	2,549	2,427	33,481	37,449
Plated wares ... Value £	..	..	43,177	49,725
Telegraph wires . „	..	..	[illegible]	34,341
Tin .... Cwt.	12,453	8,767	39,018	28,910
Zinc .... „	12,150	16,717	9,262	12,551
Other articles .. Value £	..	..	65,138	93,295
Total value	..	..	2,72[illegible]	3,096,552

Exports of Miscellaneous Articles for Month ending 30th November.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Gunpowder...... Lb.	[illegible]	[illegible]	[illegible]	[illegible]
Military stores.. Value £	..	..	97,255	[illegible]
Candles .... Lb.	[illegible]	[illegible]	[illegible]	[illegible]
Caoutchouc ..... Value £	..	..	105,537	[illegible]
Cement.......... Tons	[illegible]	[illegible]	[illegible]	[illegible]
Products of coal Value £	..	..	[illegible]	[illegible]
Earthenware ... „	..	..	[illegible]	[illegible]
Stoneware ...... „	..	..	[illegible]	[illegible]
Glass:—				
Plate........... Sq. Ft.	[illegible]	[illegible]	6,724	7,288
Flint........... Cwt.	[illegible]	[illegible]	[illegible]	[illegible]
Bottles......... „	[illegible]	[illegible]	[illegible]	[illegible]
Other kinds.... „	[illegible]	[illegible]	[illegible]	[illegible]
Leather:—				
Unwrought . . „	[illegible]	[illegible]	[illegible]	[illegible]
Wrought ..... Value £	..	..	[illegible]	[illegible]
Seed oil......... Tons	[illegible]	[illegible]	[illegible]	[illegible]
Floorcloth ..... Sq. Yds.	[illegible]	[illegible]	[illegible]	[illegible]
Painters' materials Val. £	..	..	[illegible]	142,141
Paper .... Cwt.	[illegible]	75,341	127,978	[illegible]
Rags............ Tons	[illegible]	[illegible]	[illegible]	[illegible]
Soap .... Cwt.	41,228	[illegible]	[illegible]	[illegible]
Total value	..	..	[illegible]	2,848,314

Exports of Drugs and Chemicals for Month ending 30th November.

Articles.	Quantities.		Values.	
	1896.	1897.	1896.	1897.
			£	£
Alkali............ Cwt.	414,780	472,170	107,161	121,837
Bleaching materials „	111,174	131,938	37,428	41,116
Chemical manures Tons	17,837	25,306	101,406	150,898
Medicines....... Value £	..	..	85,379	102,924
Other articles... „	..	..	260,624	337,065
Total value	..	..	591,998	753,840

Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—PLANT, APPARATUS, and MACHINERY.

Applications.

26,635. J. Weck. Improvement in sterilising apparatus. Nov. 15.

26,664. F. W. Lord. Improvements in apparatus for cooling and drying air to be used in storage chambers containing perishable produce. Complete Specification. Nov. 15.

27,061. J. E. Thornton and J. P. Lea. Improvements in generators, and apparatus in connection therewith. Nov. 19.

27,373. C. E. Müller and R. F. Hurran. A new or improved flask for bacteriological cultivations. Nov. 22.

27,458. J. McLaren and H. McLaren. Apparatus for separating oil and water or other liquids of different specific gravities. Nov. 23.

27,711. J. H. Carpenter. Improved apparatus for purifying air and gases, also for heating and cooling of the same. Nov. 25.

28,006. G. C. Foucher.—From C. A. Garrioli fils, France. Improvements in vaporising or atomising apparatus. Nov. 27.

28,113. J. Bock. Process and apparatus for obtaining large, well-formed crystals from solution. Nov. 29.

28,368. J. Leich and S. Delporte. An improved apparatus for compressing and mixing gas and air, more particularly for lighting purposes; also applicable for air compressing. Complete Specification. Dec. 1.

28,747. J. McNeil and J. Lewkowitsch. Improvements in evaporating or concentrating apparatus. Dec. 6.

28,908. H. H. Lake.—From The Manhattan Concentrator Company, United States. Improvements in machines for separating substances of different specific gravities. Dec. 7.

29,178. J. G. Lorrain.—From C. S. Ruckstahl, United States. A new or improved thermometer. Dec. 9.

29,286. T. H. P. Heriot. Apparatus for separating suspended solids from liquids, for separating soluble from insoluble solid matters, and for partially separating insoluble solid matters of different specific gravities. Dec. 10.

29,326. J. Godfrey and H. J. Hayes. Improvements in the construction of mechanical calcining furnaces. Dec. 11.

Complete Specifications Accepted.*

1896.

29,757. R. H. Yeomans. Furnaces for evaporating pans and other purposes. Dec. 15.

29,884. T. G. Webb. Improvements in and connected with apparatus for concentrating sulphuric acid and other liquids. Dec. 8.

1897.

5588. W. L. Wise.—From F. J. Brischar. Closing device for bottles and the like designed to contain gas or other fluid under high pressure. Nov. 24.

5589. W. L. Wise.—From F. J. Brischar. Closing device for bottles and the like, designed to contain gas or other fluid under high pressure. Nov. 24.

18,816. L. Fremy. Apparatus for sterilising liquids and for use as a receptacle for containing the same. Nov. 24.

21,989. W. D. Cliff and the Leeds Fireclay Company, Ltd. Improvements in inclined retorts. Nov. 24.

24,698. M. Deville. Centrifugal apparatus for sorting or separating ores. Dec. 15.

II.—FUEL, GAS, and LIGHT.

Applications.

23,887a. B. J. Atterbury. Improvements in apparatus for the production of acetylene gas. Nov. 20. Date claimed under Patents Rule 19, Oct. 16, 1897.

26,566. J. Gore. Improved burner for acetylene and like gases. Nov. 15.

26,567. F. G. Bartlett. An improvement relating to incandescent gas-lighting burners. Nov. 15.

26,588. W. S. Sargeant. Improved method of employing liquid fuel. Nov. 15.

26,591. A. Wartenweiler. New or improved acetylene gas lantern. Nov. 15.

26,594. E. Helvig. A new apparatus for producing acetylene gas. Nov. 15.

26,633. G. W. Chalmers. Improvements in or connected with incandescent gas burners wherein fragile mantles are used. Nov. 15.

26,745. G. Brogden. Improvements in incandescent mantle supports. Nov. 16.

26,776. J. Kremer. Improvements in acetylene gas generators. Nov. 16.

26,842. E. S. Bond. Improvements in means for selectively using acetylene or the like gas with ordinary lamp apparatus. Nov. 17.

26,843. E. S. Bond. Improvements in means for storing and using calcium carbide or the like for acetylene and the like gas-generating apparatus. Nov. 17.

27,016. A. C. Fraser. Improvements in methods of and apparatus for burning acetylene and other rich gases. Nov. 18. Date applied for April 19, 1897, being date of Application in United States.

27,029. R. von Maiti. Improvements in acetylene lamps. Nov. 18.

27,065. J. Benjamin. An improved gasometer for generating acetylene gas. Nov. 19.

27,240. A. Forcher. Improved acetylene gas generator for illuminating railway carriages. Complete Specification. Nov. 20.

27,288. J. Reibel. Improvements in apparatus for generating acetylene gas. Nov. 20. Date applied for May 14, 1897, being date of application in France.

27,300. J. Reibel. Improvements in devices for feeding the carburet in acetylene gas generating apparatus. Nov. 20.

27,317. S. McNamee. Improvements in preparing peat for sanitary fuel and other purposes. Nov. 22.

27,360. J. Hopwood and C. Cummins. An improved fuel. Nov. 22.

27,379. B. Talbot and W. B. Hughes. Certain improvements in the construction of gas producers. Complete Specification. Nov. 22.

27,405. R. Farrington, M. H. Hawes, and W. H. Hand. Improvements in or relating to incandescent lighting. Nov. 22.

27,416. A. A. Dickson. Improved process and apparatus for forming cold peat into blocks for fuel. Nov. 23.

27,428. H. S. Blunt. Improvements in and connected with electric furnaces for the production of calcium carbide and other substances. Nov. 23.

27,478. E. Fleming and F. Smith. Improved means for mixing air and gas for heating and lighting purposes. Nov. 23.

27,536. O. Falbe and E. Borchardt. Improvements in or relating to burners for acetylene gas. Nov. 23.

27,593. J. Jameson. Improvements in the production of light and radiant heat by combustion of gas or inflammable vapour. Nov. 24.

27,639. W. J. H. Lazarus. Improvements in and connected with the production of acetylene gas. Nov. 24.

27,650. A. J. Boult.—From E. J. Armand-Machin, France. Improvements in or relating to the construction of acetylene gas generators. Nov. 24.

27,652. M. Bernstein, Proprietor of the Firm of J. F. Wallmann and Co. Process for the manufacture of automatic igniters for illuminating gas. Complete Specification. Nov. 24.

27,703. G. Fincken and J. Rollins. An improved saturating appliance for lime-lighting. Nov. 25.

27,767. J. H. Schülke. Improved acetylene gas generator. Complete Specification. Nov. 25.

27,869. J. Bowing. Improvements in the manufacture of coke, and in apparatus therefor. Nov. 26.

27,876. The Liver Acetylene Gas Company, Ltd., and E. Evans. Improvements in apparatus for generating acetylene or other gas generated by the addition of a liquid to a solid. Nov. 26.

27,886. J. Skar. Process for the manufacture of fuel blocks. Nov. 26. Date applied for, May 4, 1897, being date of application in Norway. Complete Specification.

28,001. H. Winckler.—From H. Freudenthal, Germany. Improved means and ingredients for effecting an increase in the lighting properties of gas and oil flames. Nov. 27.

28,003. J. S. Billwiller. Method of and means or apparatus for purifying the products of combustion of acetylene gas flames. Nov. 27.

28,012. H. H. Lake.—From W. A. Shearer, South African Republic. Improved apparatus for the production of acetylene gas from calcium carbide. Nov. 27.

28,031. E. A. Wood. Improvements relating to incandescent gas-burners. Nov. 29.

28,094. W. Tyree. An improved acetylene gas generator. Complete Specification. Nov. 29.

28,102. M. Wagner. Improvements in apparatus for generating acetylene gas. Complete Specification. Nov. 29.

28,106. A. J. Evans. An improved gas producer. Nov. 29.

28,226. H. L. Hartenstein. *See* Class VII.

28,258. L. J. Keck. Improved lamp for generating and burning acetylene gas. Nov. 30.

28,264. Sir H. Grubb, Kt. Improvements in apparatus for the generation of acetylene gas. Nov. 30.

28,368. J. Leich and S. Delporte. *See* Class I.

28,399. E. G. Gearing. Improvements in or relating to acetylene generating and storing apparatus. Dec. 1.

28,439. V. Sockeel, A. Lanby, C. H. Drisse, and Sneck. Novel apparatus for the production of acetylene gas, also called "Lumière Soleil." Dec. 2.

28,448. G. Tripel. A new or improved apparatus for use in burning liquid fuel. Dec. 2.

28,477. T. H. Gittins and J. Marshall. Improvements in the manufacture of fuel. Dec. 2.

28,496. H. Gardner. Improvements in the manufacture of artificial fuel and fire lighters. Dec. 2.

28,504. W. E. Kochs. Improvements in coke ovens and in machinery in connection therewith. Complete Specification. Dec. 3.

28,526. J. R. Wigham. A method of increasing the illuminating power of gas made from coal or other material. Dec. 4.

28,698. R. Kirk. Improvements in and relating to lamps for generating and using acetylene gas. Dec. 4.

28,706. J. Moeller. An improvement in incandescence mantles. Dec. 4.

28,778. J. B. Melhuish, M. Guthrie, and J. H. Hughes. A new gas. Complete Specification. Dec. 6.

28,798. B. Richard. Improvements in and relating to apparatus for generating and purifying acetylene. Complete Specification. Dec. 6.

28,824. H. Bryant. Improvements in apparatus for making illuminating gas. Dec. 6.

28,836. J. B. Melhuish. An apparatus for the generation of ethine gas from a carbide of a metal of the alkaline earths. Dec. 7.

28,909. G. A. Bronder. Improvements in machinery for discharging gas retorts. Complete Specification. Dec. 7.

29,051. J. O. O'Brien.—From E. M. Boule, France. Improvements in apparatus for generating acetylene gas. Complete Specification. Dec. 8.

29,096. J. A. Drake and J. S. Marsland. Improvements relating to inclined gas retorts. Dec. 9.

29,103. R. Farrington. Improvements in and relating to a preparation for cleaning and strengthening incandescent mantles. Dec. 9.

29,114. C. J. Shears. A process for treating incandescent mantles for illuminating purposes. Dec. 9.

29,201. E. V. Prevost, O. Raus, and A. Cerkel. Improvements in apparatus for the production of acetylene. Dec. 9.

29,234. W. H. Russell and G. E. Russell. Improvements in and relating to the manufacture of gas. Complete Specification. Dec. 10.

29,277. E. Evans. Improvements in holding or storing carbide of calcium and other like chemical, and in regulating the evolution of acetylene or other gas therefrom. Dec. 10.

29,304. M. H. Hawes, R. Farrington, and W. H. Hand. Improvements in or relating to gas and oil lighting. Dec. 10.

29,393. A. M. G. Sébillot. Apparatus for generating acetylene gas. Dec. 11.

29,405. V. Maréchal and A. Moreau. Improvements relating to acetylene generators. Complete Specification. Filed Dec. 11. Date applied for June 11, 1897, being date of application in France.

29,421. D. Kempe. Improvements in apparatus for burning vaporised petroleum and the like for heating and illuminating purposes. Complete Specification. Dec. 11.

Complete Specifications Accepted.

1896.

19,741. J. Steiger. Manufacture of artificial fuel. Dec. 15.

27,194. A. J. de Pimental Hargreaves. Manufacture of acetylene gas, and apparatus therefor. Dec. 8.

27,744. A. Kiesewalter. Manufacture of a carbide which, by its decomposition by water, produces acetylene gas. Dec. 15.

28,265. E. Barker. The incandescent fire ball. Dec. 8.

28,588. L. Mond. Production of a mixture of air and water-vapour for gas producers. Dec. 1.

28,721. R. G. Leventhorpe. Production of hydrogen gas, and apparatus therefor. Dec. 15.

1897.

658. L. Tèdé. Apparatus for the production of acetylene. Nov. 24.

1254. R. J. Moss. Apparatus for generating and purifying acetylene gas. Nov. 24.

1370. F. G. Bartlett. Improvements relating to incandescent gas lighting. Dec. 8.

1784. W. P. Thompson.—From A. Gobron. Portable apparatus for generating and burning acetylene gas. Nov. 24.

1929. T. Thorp and T. G. Marsh. Apparatus for the production of acetylene from calcium carbide. Dec. 1.

1952. J. C. Mewburn.—From A. M. Scott. Apparatus for generating gas by the reaction of solid substances with water or other liquid, especially applicable to the generation of acetylene gas by contact of water with metallic carbides. Dec. 1.

2129. W. L. Wise.—From A. Piatti and Co. Manufacture of gas. Nov. 24.

6132. W. Climie and A. Watson, jun. Gas producers. Nov. 24.

9294. A. H. Barthez. Improved portable and automatic apparatus for the production of acetylene gas for lighting purposes. Dec. 1.

15,813. E. Andreoli. Manufacture or production of ozone, and apparatus therefor. Dec. 1.

16,890. R. H. Whiteley and W. Ellis. Apparatus for enriching coal gas. Dec. 8.

20,574. G. Lebrun and F. Cornaille. A burner for acetylene and other gases rich in carbon. Nov. 24.

22,625. W. L. Voelker. Manufacturing incandescible materials. Dec. 8.

22,626. W. L. Voelker. Improvements in incandescible materials and mantles, and the process of making same. Dec. 8.

23,802. L. S. Buffington.—Acetylene gas generators. Nov. 24.

24,602. G. Mahnke and C. Kraft. Improved means of protection of incandescent gaslight mantles. Dec. 1.

24,815. H. Ottenbruch. Mantles for incandescence gas lighting. Dec. 1.

24,822. A. Syssoyeff and L. Braly. Improvements in auto-luminous compositions. Dec. 8.

25,632. R. E. Perraga. Method and apparatus for generating formaldehyde gas. Dec. 8.

26,311. J. Janz. Manufacture of fabrics for incandescence mantles for illuminating purposes. Dec. 15.

26,325. A. J. Boult. — From F. Alexandre, France. Apparatus for the manufacture and use of acetylene gas. Dec. 15.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Application.

28,653. A. Gordon. Improvements in and relating to retorts for the distillation of shale and other substances. Dec. 4.

Complete Specification Accepted.

1897.

2515. H. J. Dawes. — Distillation and breaking up of liquid hydrocarbons and similar substances. Dec. 1.

IV.—COLOURING MATTERS and DYES.

Applications.

26,563. W. H. Claus, A. Rée, and L. Marchlewski. Production of new azo colouring matters. Nov. 15.

26,675. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Improvements in the manufacture of derivatives of the naphthalene series. Nov. 15.

27,051. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of new colouring matters. Nov. 18.

28,107. S. Pitt.—From L. Cassella and Co., Germany. Improvements in the manufacture of azo dyestuffs and in the production of fast dyeings on animal fibre. Nov. 29.

28,604. H. H. Lake.—From Farbwerk Mülheim vorm. A. Leonhardt and Co., Germany. Improvements in the manufacture of colouring matters. Dec. 3.

28,707. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of *m*-oxybenzaldehyde *o*-sulphonic acid. Dec. 4.

Complete Specifications Accepted.

1897.

1592. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of rhodamine dialkylamides. Nov. 24.

1962. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Manufacture of blue mordant-dyeing colouring matters. Dec. 8.

2655. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Manufacture of colouring matters of the rhodamine series. Dec. 8.

2874. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Manufacture and production of new disazo dyes on cotton. Dec. 8.

3452. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Production of colouring matters. Dec. 8.

4703. W. G. Heyes.—From The Chemical Works, Sandoz. Production of substantive disazo colouring matters. Dec. 15.

22,677. A. Ashworth and J. Bürger. Production of colouring matters. Nov. 24.

23,816. A. Ashworth and J. Bürger. Production of colouring matters. Nov. 24.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

Applications.

27,020. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. A new process for mercerising vegetable fibre. Nov. 18.

27,651. K. Klietsch. Improvements in or relating to the manufacture of linoleum, kamptulicon, and similar materials. Complete Specification. Nov. 24.

27,943. J. Archer and J. Arthur, jun. Improvements in or relating to the manufacture of linoleum and like floor coverings. Nov. 27.

28,380. H. B. Edwards.—From O. M. Thowless, United States. A method of or process for manufacturing raw silk. Dec. 1.

28,491. C. Wetherwax. Improvements in the treatment of vegetable fibres. Dec. 2.

28,631. H. Pauly. An improved process for the manufacture of artificial silk. Dec. 4.

29,072. J. Y. Johnson. — From W. H. Drury, United States. Improvements in the manufacture of yarns, and apparatus therefor. Dec. 8.

Complete Specifications Accepted.

1896.

27,483. H. C. Longsdon. Improved yarn-drying apparatus. Nov. 24.

1897.

90. H. W. Godfrey. Manufacture of floorcloth and the like. Nov. 24.

3222. J. Millar. Improvements in textile fabrics or cloths. Dec. 15.

5350. M. Beck. Apparatus for finishing, stretching, mordanting, and drying textile fabrics. Dec. 8.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Applications.

26,667. P. A. Newton.—From The Alexander Smith and Sons Carpet Company, United States. Improvements in methods of and apparatus for printing or colouring yarn and similar material. Nov. 15.

27,270. C. Deleseluse. Improvements in bleaching cotton, linen, and other vegetable textile materials. Nov. 20.

27,690. W. Beaumont. Improvements in apparatus for dyeing, bleaching, and otherwise treating cops of yarn. Complete Specification. Nov. 25.

27,855. E. Fletcher. Improvements in hank-dyeing machines. Nov. 26.

27,937. T. Ingham. An improvement in dyeing. Nov. 27.

28,090. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. A new process for producing black shades on cotton. Nov. 29.

28,115. C. Spindler and C. Göhring. Improvements in the treatment of silk for dyeing and bleaching. Complete Specification. Nov. 29.

29,317. H. Lang, C. Rütter, and M. Rutter. An improved process of dyeing. Dec. 11.

29,359. J. Birtwisle. Improvements in and relating to the dyeing and like treatment of cops and similarly wound yarns. Dec. 11.

Complete Specifications Accepted.

1896.

29,645. W. Hadfield, J. J. Sumner, and H. Hadfield. Apparatus for use in the continuous bleaching and washing of woven goods in the piece, applicable also in the washing and clearing of printed or dyed goods. Dec. 1.

1897.

1629. T. Ingham. Improved dyeing process. Nov. 24.

2620. W. Watson and E. Bentz. Production of coloured discharge patterns on indigo-dyed cotton fabrics in calico printing. Dec. 8.

VII.—ACIDS, ALKALIS, AND SALTS.

Applications.

27,019. L. H. Frentz. Manufacture of conglomerates containing alkaline oxidising bodies for bleaching linen and other objects. Nov. 18.

27,221. A. R. Scott. Improvements in obtaining chlorine. Nov. 20.

28,226. H. L. Hartenstein. An improved process and apparatus for carburetting calcium. Complete Specification. Nov. 30.

28,688. W. H. Graham. Metallic carbides, and processes and apparatus for their manufacture; applicable also to other purposes. Dec. 4.

29,008. J. Bastick and W. A. Thornton. Improvements in or relating to the manufacture of calcium carbide. Dec. 8.

29,009. J. Bastick and W. A. Thornton. Improvements in or relating to electric furnaces for the production of calcium carbide and other substances. Dec. 8.

29,384. F. J. Bergmann. Process for the production of calcium carbide in blast furnaces. Dec. 11.

Complete Specifications Accepted.

1896.

28,294. E. A. Goddin and R. Lees. Process and apparatus for obtaining common salt and other like salts from their solutions. Dec. 8.

29,066. E. J. Hunt and E. F. Watson. Manufacture of chlorine. Dec. 8.

29,884. T. G. Webb. *See* Class I.

1897.

1218. H. H. Lake.—From J. S. Levi and Co. Process for obtaining barium carbonate from native sulphate of barium. Nov. 24.

1762. G. D. Howard.—From H. Howard. Methods of and apparatus for concentrating sulphuric acid or the like. Dec. 8.

1227. T. Ryan, jun., A. Dyson, and The United Alkali Co., Ltd. Manufacture of caustic soda or caustic potash from carbonate of soda or carbonate of potash. Nov. 24.

4747. A. Knop. Manufacture of nitrites. Dec. 15.

19,512. J. W. Keneval, C. A. Spofford, and J. H. Mead. Means for manufacturing carbides. Dec. 1.

21,073. J. Reich. Method of transforming silicofluorides and borofluorides into corresponding fluorides or fluorine compounds. Dec. 1.

22,136. F. Lutze. Process for the preparation of carbolic acid. Dec. 15.

24,524. A. MacNab.—From W. A. Willock, India. Manufacture of bay salt. Dec. 8.

VIII.—GLASS, POTTERY, AND ENAMELS.

Applications.

26,571. W. Lindsay and D. Anderson. Improvements in and relating to apparatus for the manufacture of glass articles, and appliances for shaping and forming same. Nov. 15.

26,901. A. Houghton. Improvements in the process of and apparatus for drawing glass tubing. Complete Specification. Nov. 17.

27,248. W. Haley. A new power press for pressing glass or metals while in a liquid state. Nov. 20.

27,786. T. Pfister. Improvements in cloisonné and mosaic work. Nov. 26.

27,796. J. H. Lea. Improved kilns and ovens for use in firing tiles and other pottery-ware. Nov. 26.

27,841. W. Owen. Improvements in kilns for calcining or fritting materials used in the production of potters' glazes. Nov. 26.

28,120. J. T. Newell. Improvements in or relating to furnaces for glass or ceramic ware. Nov. 29.

28,315. L. Grote. Improved method of and apparatus for manufacturing water-glass. Dec. 1.

28,466. C. Keller and A. Keller. Manufacture of ceramic, earthenware, porcelain, stoneware, glass, crystal, and such like ware, with metallic decorations, and furnaces or ovens therefor. Dec. 2.

28,885. C. Kempton, sen. An improved furnace for making glass and manipulating glassware. Dec. 7.

Complete Specifications Accepted.

1896.

28,789. C. F. Ravenhill. Glaze for clay tobacco pipes. Dec. 8.

1897.

1871. A. E. Doucede. Method and means for connecting tubes and pipes of stoneware, earthenware, and glass. Dec. 1.

24,317. B. Swinden. Method of and composition for polishing glass. Dec. 1.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Applications.

27,104. H. Eichler. New or improved material for flooring. Complete Specification. Nov. 19.

27,561. W. Johnson and H. Walker. Improvements in or relating to kilns for burning cements, limes, and other like substances. Nov. 24.

27,677. A. E. Scharffetter.—From C. Franz, Austria-Hungary. Improvements in the manufacture of bricks for building purposes. Nov. 25.

27,682. W. P. Ingham. Improvements in or connected with fireproof walls or partitions. Nov. 25.

27,825. J. C. Sellars. Improvements in the manufacture of cement. Complete Specification. Nov. 26.

27,939. F. M. Lyte, O. J. Steinhart, and W. H. Wickham. Improvements in the preparation of uninflammable wood. Nov. 27.

28,276. H. Spatz and H. Quistorp. Process for the production of asphalte powder for making street asphalte plaster. Nov. 30.

28,310. P. Tickle. Through-burning of clay and terra cotta goods. Dec. 1.

28,656. W. H. Northcutt. Combination paving blocks and system of paving. Complete Specification. Dec. 4.

28,842. H. W. Gruner. Improved composition for the manufacture of artificial stone, cement, and the like. Dec. 7.

28,938. H. V. Simpson. Improvements in treating wood for the purpose of rendering it non-inflammable or for preserving it. Dec. 7.

28,974. T. Potter. Improvements in fireproof floors, ceilings, and partitions. Dec. 8.

29,020. F. S. Smith. Improvements in the construction of bricks and other moulded building materials. Dec. 8.

29,137. A. R. Crawford and W. Crawford. Improvements in the manufacture of briquettes for heating purposes. Dec. 9.

29,249. The British Marble and Slate Syndicate, Ltd., and W. F. Baker. Improved method and means for utilising slate refuse. Dec. 10.

29,263. C. Hannay. Improvements in firebricks, slabs, or tiles. Dec. 10.

29,420. E. Vleck. An improved method of manufacturing plates, slabs, or the like for building purposes. Complete Specification. Dec. 11.

Complete Specifications Accepted.

1896.

26,792. H. Brockbues. Manufacturing a material for covering walls and floors. Dec. 1.

29,109. W. H. Coward. Manufacture of composition and other compressed blocks for paving. Nov. 24.

29,212. Petrifite, Ltd.—From J. Steiger. Manufacture of cement. Nov. 24.

1897.

1881. La Société Metallurgique de Champigneulles et Neuves-Maisons. Manufacture of cement. Dec. 15.

X.—METALLURGY, MINING, Etc.

Applications.

26,706. H. R. Angel. Improvements in the reduction of ores containing antimony. Nov. 16.

26,891. J. C. Richardson. Improvements in or relating to methods (wet) of treating gold, silver, and other ores. Nov. 17.

27,015. J. E. Dowson. An improved process relating to the reduction of ores, and improvements in furnaces therefor. Nov. 18.

27,081. W. B. Johnson.—From The Carnegie Steel Company, Ltd., United States. Improvements in apparatus or appliances for casting metals. Nov. 19.

27,160. B. H. Thwaite and H. V. Holden. Improvements in decarburising cast iron or steel, and apparatus for that purpose. Nov. 19.

27,248. W. Haley. *See* Class VIII.

27,546. T. Levoz. New construction of converter apparatus for producing steel and wrought iron of superior quality. Complete Specification. Nov. 23.

27,550. C. P. Shrewsbury. Improvements in and connected with the extraction of gold. Nov. 23.

27,555. G. D. Burton. A new or improved process of and apparatus for treating ores. Nov. 23.

27,614. J. W. Clarke. Improvements in apparatus for concentrating and amalgamating ores. Nov. 24.

27,702. J. Parry and G. H. Llewellyn. Improvements in the manufacture of crude metal suitable for the more rapid production of steel by the Siemens or open-hearth process. Nov. 25.

27,717. W. Buckley. Improvements in or applicable to ingot and similar moulds, and to the manufacture of ingots. Complete Specification. Nov. 25.

27,752. R. A. Hadfield. Improved manufacture of steel. Nov. 25.

27,753. R. A. Hadfield. Improved manufacture of steel. Nov. 25.

27,881. F. D. James and M. B. Fox. An improved metal, and method of producing it. Nov. 26.

28,440. E. Govett. Machine and apparatus for the extraction of gold, silver, and other metals from their ores by amalgamation with lead. Dec. 2.

28,441. H. C. Jenkins. The treatment of refractory gold ores. Dec. 2.

28,568. C. M. White.—From King, United States. A new or improved process of annealing iron castings. Complete Specification. Dec. 3.

28,723. J. de F. Maunder. Improvements in the treatment of ores. Dec. 4.

28,745. E. Smith and A. T. Smith. Improvements in the method and apparatus for annealing wire. Dec. 6.

28,896. J. Rudolphs and J. Landin. An improved method or process of extracting metals from pulverised or finely divided ores or compounds. Complete Specification. Dec. 7.

29,162. E. E. Atkins. Annealing metals. Dec. 9.

Complete Specifications Accepted.

1896.

26,297. J. Campbell. Method of and apparatus for treating refractory gold and silver, and other metal-bearing ores and materials. Dec. 1.

27,346. J. J. Shedlock. Extracting metals from their ores, and apparatus therefor. Nov. 24.

28,361. M. M. Marcus. Making metal alloys. Dec. 8.

28,880. C. de E. Llatas. Manufacture of steel. Dec. 15.

1897.

516. A. S. F. Robinson. Method and machinery or apparatus for extracting gold or other valuable metal from crushed ore or material containing it. Dec. 15.

880. A. Etard. Treatment of ores or other matters containing gold, silver, or copper, for the obtainment of the metal therefrom. Dec. 15.

1778. C. Wiehmann. Extracting gold from ores. Dec. 15.

3303. F. W. Streathfeild. Extracting metals from their ores, or in obtaining solutions of same. Dec. 1.

18,640. J. Landin. Wet process for extracting gold from gold ores or gold-ore waste. Dec. 15.

19,764. A. E. Thomas. Thomas's universal converter for cleansing molten iron from its impurities and converting the same into refined iron or malleable iron or steel as may be required to produce. Dec. 1.

24,121. W. P. Thompson.—From E. C. Ketchum. Process for treating mixed sulphide ores. Nov. 24.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

Applications.

26,656. P. F. Ribbe. Improvements in and relating to electric accumulator plates. Complete Specification. Nov. 15.

26,680. A. Tribelhorn. Improvements in electric accumulators or storage batteries. Nov. 15.

26,880. G. W. B. Crees, W. L. Durban, and J. Gore. Improvements in and relating to electrical furnaces specially applicable for the production of calcium carbide. Nov. 17.

26,881. G. W. B. Crees, W. L. Durban, and J. Gore. Improvements in and relating to electrical furnaces specially applicable for the production of calcium carbide. Nov. 17.

26,919. O. C. Strecker and H. H. Strecker. Improvements in or relating to the application of electrodes in electrolytic processes. Complete Specification. Nov. 17.

26,921. O. C. Strecker and H. H. Strecker. Improved process for preparing insoluble or hardly soluble salts from metallic anodes by electrolysis of aqueous solutions containing two salts. Complete Specification. Nov. 17.

26,923. O. C. Strecker and H. H. Strecker. Improvements in or relating to the production of protoxides and oxides or of hydro-protoxides and hydroxides from metallic anodes by electrolysis of alkaline salt solutions. Complete Specification. Nov. 17.

27,129. A. L. C. Nodon, L. A. Bretonneau, and P. d'Alton Shée. A new or improved electrolytic method of and apparatus for saponification. Complete Specification. Nov. 19.

27,165. J. W. Townsend. Improvements in electrical storage batteries or accumulators. Nov. 19.

27,508. O. March. Improvements in and connected with the extraction of gold by electrolysis. Nov. 23.

27,551. J. W. Swan and A. C. Hyde. Improvements in means to be employed in the electrolysis of salts. Nov. 23.

27,683. W. H. Lane. Improvements in and connected with electrical storage batteries. Nov. 25.

27,706. J. E. Hartley and H. E. Hartley. Improvements in barrelling electro-metallic depositing apparatus. Nov. 25.

27,707. J. E. Hartley and H. E. Hartley. Improvements in rotative electro - metallic depositing apparatus. Nov. 25.

27,712. A. Hirsch. Improvements in electrodes for use in connection with apparatus for welding and soldering by means of electricity. Nov. 25.

27,940. M. Orban. Improvements in electric batteries. Nov. 27.

27,973. C. Clark. Improvements in charging secondary batteries or accumulators. Nov. 27.

27,982. W. Rowbotham. Improvements in primary batteries. Nov. 27.

28,212. H. Riecken. Improvements in the electrolytical treatment of ores and slimes for the recovery of precious metals therefrom, and apparatus therefor. Nov. 30.

28,242. P. J. R. Dujardin. Improvements in or relating to negative plates for secondary batteries. Nov. 30.

28,460. C. Payen. An improved electrical accumulator. Dec. 2. Date applied for, May 1, 1897, being date of application in Belgium.

28,795. H. J. Brown, O. R. H. Schmidt, and A. Mackenzie. *See* Class XIII.

29,011. J. Bastick and W. A. Thornton. Improvements in or relating to electric furnaces for the production of calcium carbide and other substances. Dec. 8.

29,048. F. E. Elmore and A. S. Elmore.—From J. O. S. Elmore, India. Improvements in effecting electrolytic deposit of iron. Dec. 8.

29,180. P. Brandt. An improved electrical accumulator. Complete Specification. Dec. 9.

29,273. H. H. Lake.—From C. Francke, Germany. Improvements in plates or electrodes for electric accumulators. Complete Specification. Dec. 10.

Complete Specifications Accepted.

1897

490. W. Wright. Electric deposition of metal on pipes, tubes, and other articles. Nov. 24.

615. E. Bohn. Means for electrically obtaining ozone, and employing the same for curative and other purposes. Dec. 15.

1204. H. W. Hancock and A. H. Dykes. Method of preparing positive plates for secondary batteries. Dec. 8.

1627. A. E. Cook. Apparatus for the electro-deposition of metals. Dec. 8.

2709. W. E. Heys.—From E. Dumoulin. Improvements in the electro-deposition of copper and other metals. Dec. 8.

3346. C. Payen. Secondary batteries. Dec. 1.

14,708. G. H. Nash. Apparatus for coating metal articles by electro-deposition. Dec. 15.

19,638. R. Kennedy. Improvements in electrical storage batteries and plates therefor. Nov. 24.

19,846. L. Guelzow. Manufacture of plates for electric accumulators. Nov. 24.

21,704. S. W. Hart. Secondary electric batteries or accumulators. Nov. 24.

21,705. S. W. Hart. Secondary electric batteries or accumulators. Nov. 24.

22,203. A. J. Boult.—From F. H. Soden, United States. Apparatus for electrically treating ore. Dec. 15.

24,150. H. S. Lloyd. Electrodes for storage batteries. Nov. 24.

25,386. H. K. Hess. Improvements in chemical electric generators. Dec. 15.

XII.—FATS, OILS, AND SOAP.

Applications.

27,262. J. W. Owen. Improved detergents, and method of manufacturing the same. Nov. 20.

28,206. A. Euston. Improvements in the treatment of oleaginous seeds for the extraction of oil therefrom. Complete Specification. Nov. 30.

Complete Specifications Accepted.

1896

28,682. J. L. Garle and C. C. Frye. Process for the decolorisation of palm oil. Nov. 24.

1897.

751. J. T. Potts. Manufacture of soap. Nov. 24.

1305. T. C. Bryan and B. J. Sharpe. Washing solution. Dec. 8.

3700. J. David. Treatment of oleic acid. Dec. 1.

5256. H. H. Lake.—From F. W. Klever. A method of dissolving iodine in mineral oils. Dec. 1.

23,110. T. Arthur. A solution for washing and cleansing glass, and painted and varnished surfaces. Nov. 24.

23,197. J. Monterrubio. Improved system for manufacturing soaps by the direct employment of oleaginous seeds. Nov. 24.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

26,800. C. J. Grist. Improvements in the manufacture of paints, anti-corrosive compositions, and compositions for like uses. Nov. 16.

26,867. J. S. Macarthur. Improved anti-fouling composition. Nov. 17.

28,484. H. Locsner. Improved manufacture of waterproof and weather-proof paint. Dec. 2.

28,795. H. J. Brown, O. R. H. Schmidt, and A. Mackenzie. A new or improved compound or material for electrical insulating purposes. Dec. 6.

29,092. M. Zingler. Improvements in the treatment of decayed or putrid india-rubber. Dec. 9.

29,291. J. Kerner. The manufacture and production of an opaque pigment or body colour. Dec. 10.

Complete Specifications Accepted.

1897.

3576. J. C. Mewburn.—From J. C. Taylor. Manufacture of anti-fouling compositions or paints. Dec. 15.

9447. J. W. Blake and S. J. Blake. Compositions or paints for preserving structures. Nov. 24.

11,168. E. Garnier and S. J. Prescott. Improvements in india-rubber compounds and in gutta percha compounds. Dec. 15.

19,179. H. C. Woltereck. Manufacturing white-lead by electrolysis. Dec. 15.

21,718. R. Strobentz and J. Fried. Manufacturing black pigments from carbonaceous material. Dec. 8.

23,957. F. J. Bergmann. Production of graphite. Dec. 1.

24,504. A. Stoop. Improved process for the manufacture of paints and pigments. Dec. 15.

26,107. H. Markus and G. B. Behrens, of the firm of D. Lee and Co. Manufacture of india-rubber and gutta-percha. Dec. 15.

XIV.—TANNING, LEATHER, GLUE, and SIZE.

Applications.

26,677. W. F. Reid and E. J. V. Earle. Improvements in the manufacture of enamelled leather. Nov. 15.

26,760. H. Holmes. Improvements in the process of depilating hides or skins. Complete Specification. Nov. 16.

29,292. W. E. Heys.—From E. Dumoulin, France. Improvements in the method of and means for rendering animal, membranous, gelatinous, albuminous, fibrinous, and analogous substances, and articles composed of or containing such substances, insoluble. Dec. 10.

Complete Specifications Accepted.

1896.

28,067. E. A. Muskett and J. B. Scammell. Treatment of raw hides. Dec. 8.

1897.

1587. T. D. Bowman and J. Bowden. Manufacture of imitation leather. Dec. 8.

3180. S. Millar and C. E. Millar. Treating hides and skins. Dec. 8.

24,080. A. J. Boult. From G. Brigalant, France. Treatment of leather waste, and apparatus employed therein. Dec. 1.

XVI.—SUGARS, STARCHES, GUMS, Etc.

Applications.

26,985. J. Peeters. Process for the production of new vegetable extracts and new vegetable peptones of commercial utility by the transformation of all kinds of yeasts and these new products of commercial utility themselves. Complete Specification. Nov. 18.

27,050. A. Classen. Manufacture of soluble modifications of albuminous substances, and processes therefor. Nov. 18.

27,161. A. Classen. See Class XX.

27,534. W. P. Thompson.—From L. Bussy, H. Philippe, and P. Bussy, France. An improved impermeable water-resisting paste-gum or coating. Nov. 23.

28,589. J. J. Hignette. Improved process for the purification of saccharine juices, and apparatus therefor. Dec. 3.

29,292 W. E. Heys.—From E. Dumoulin, France. See Class XIV.

Complete Specifications Accepted.

1897.

34. E. L. Newsome. Appliance for supplying glucose, syrup, or other material to preserving pans when boiling. Nov. 24.

1338. A. Verley. Manufacture of sugar. Nov. 24.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

26,985. J. Peeters. See Class XVI.

27,376. W. Jago. An improvement in the manufacture and treatment of malt preparations. Nov. 22.

27,858. E. B. Ludlow. An improved appliance for aërating, rousing, and mixing fermenting and other liquids, or for lifting the same. Nov. 26.

28,579. C. Schmitz. An improved process and apparatus for producing beer-wort from malt flour. Complete Specification. Dec. 3.

29,089. G. F. Zimmer. Improvements in and connected with gelatinising and drying apparatus used in the manufacture of flaked maize malt and similar substances. Dec. 9.

29,157. H. M. Kettelwell. An improved method and apparatus for the prevention and extinction of fires in breweries, distilleries, and other like premises. Dec. 9.

Complete Specifications Accepted.

1896.

26,655. C. Zimmer. Process for brewing. Dec. 1.

1897.

2287. E. E. M. Payne, T. O. Kent, and E. E. Pullman. Compositions or mixtures to be used for the preservation of malt liquors, such as ale, beer, stout, porter, and other like saccharine liquids, and methods of using and applying such compositions or mixtures. Dec. 8.

13,722. J. Goodfellow. Manufacture of nutritious products from yeast. Dec. 1.

16,232. H. Hempel. Process for producing a carbonaceous agent for enriching spirit. Nov. 24.

21,878. E. Zdarek. Manufacture of ethyl alcohol. Nov. 24.

XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

Applications.

A.—Foods.

26,574. L. Checkemian. Improved food preparations, maghertaze, mazoon, or tog hourt, a most delicious, nourishing and appetising food. Nov. 15.

26,628. A. Schmidt. Process for the production of food for cattle. Complete Specification. Nov. 15.

26,658. F. H. M. Röver. Improvements in or relating to the preservation of eggs. Complete Specification. Nov. 15.

26,754. W. G. Day. Improvements in methods of preserving perishable substances. Complete Specification. Nov. 16.

27,439. P. Ghinozzi. New process for preserving food and all kinds of organic substances. Nov. 23.

27,617. W. Lauwer. New or improved process for preserving meat and other alimentary substances. Nov. 24.

28,359. M. Delavigne, C. Helmerich, and A. Kluglein. An improved process and apparatus for the preservation of eggs. Complete Specification. Dec. 1.

28,379. H. H. Lake.—From A. Moyet, France. Improvements in apparatus for sterilising meat in the raw state. Dec. 1.

29,189. A. Bernstein. The manufacture of a substitute for eggs. Dec. 9.

B.—Sanitation.

26,625. H. G. Downton and W. H. Nicholls. Improvements in process and apparatus for the destruction of night soil and noxious matters. Complete Specification. Nov. 15.

26,821. W. A. Clark. Improvements in the method of and apparatus for the treatment of sewage. Nov. 16.

26,959. G. Hinchcliffe, H. T. Hirst, and J. R. Hinchcliffe. An improved treatment for precipitating, clarifying, and purifying sewage and waste or foul water. Nov. 18.

27,004. J. G. Dornig, F. H. Rachlitz, and T. Zennert. Improved process and means for the sanitary disposal of human excrement, and the production therefrom of gas and by-products serving for manure and other purposes. Complete Specification. Nov. 18.

27,026. H. McPhail. Improvements in heating, softening, and purifying water, and apparatus therefor. Nov. 18.

27,306. H. Morris. Improvements in and connected with the treatment of sewage and other waste liquids. Nov. 22.

27,349. D. Cameron, F. J. Commin, and A. J. Martin. Improvements in apparatus for the treatment of sewage or other liquids. Nov. 22.

28,164. G. Krell and M. Elb. An improved disinfecting process by means of methylaldehyde or polymeric methylaldehydes, and method of employing same. Nov. 30.

29,090. A. B. Ogden. New process or system of sewage and foul water purification by electrical action. Dec. 9.

29,160. J. Hardwick. Improvements in or relating to the treatment of sewage. Dec. 9.

Complete Specifications Accepted.

A.—Foods.

1897.

4447. J. Imray.—From La Soc. Anonyme des Matières Colorantes et Produits Chimiques de St. Denis and D. A. Rosenstiehl. Process for preserving alimentary substances. Nov. 24.

B.—Sanitation.

1896.

27,513. F. P. Candy. Purification of sewage and polluted waters. Dec. 15.

XIX.—PAPER, PASTEBOARD, Etc.

Applications.

27,477. A. S. Caldwell. Improvements in and relating to the production of water marks in paper. Nov. 23.

28,187. A. S. Caldwell. Improvements in and relating to the production of water marks in paper. Nov. 30.

28,982. F. Langley, J. Whittingham, and J. Harris. Improvements in the process of and apparatus for sizing paper. Dec. 8.

29,032. A. J. Boult.—From R. Scholich and O. Kunze, Germany. Improved manufacture of sanitary paper of material for toilet purposes. Dec. 8.

Complete Specification Accepted.

1897.

24,560. E. Cox, F. Kornacher, and M. Brumm. Improved writing paper, and ink therefor. Dec. 15.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

Applications.

27,032. H. J. H. Fenton and F. S. St. Barbe Sladen. An improved process for the production of dihydroxytartaric acid. Nov. 18.

27,161. A. Classen. Manufacture of compounds of starch-like bodies and gums with acetaldehyde and paraldehyde, and processes therefor. Nov. 19.

27,403. A. J. Boult.—From A. Reychler, Belgium. Improved manufacture of camphor sulphonic acids. Nov. 22.

28,167. O. Imray.—From La Société Fabriques de Produits Chimiques de Thann et de Mulhouse, Germany. Improvements in the manufacture of artificial musk. Dec. 2.

29,061. S. Pitt.—From The Parmaceutische Institut Ludwig W. Gans, Germany. The production of the operative substance from the thyroid glands and other organs in an unaltered uncoagulable state. Dec. 8.

29,308. A. Goldberg, W. Siepermann, and H. Flemming. A process for the preparation of guanidine salts. Dec. 10.

Complete Specifications Accepted.

1896.

26,078. W. B. Bishop. Treatment of piperazine. Nov. 24.

1897.

2472. J. Y. Johnson.—From C. F. Boehringer and Soehne. Preparation of hydroxycaffeine by the direct methylation of uric acid or of certain mono- and dimethyl-derivatives thereof also obtainable in the process. Dec. 8.

2556. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of nitroal phylpyruvic acids. Dec. 8.

3957. J. Y. Johnson.—From C. F. Boehringer and Soehne. Manufacture or preparation of dichloroxy purine. Dec. 15.

25,945. W. P. Thompson.—From J. Brissonnet, France. Improved creosote compound. Dec. 15.

XXI.—PHOTOGRAPHY.

Applications.

27,033. J. C. Fell.—From M. W. McDonald, United States. Matt surface photographic paper, and process of making same. Complete Specification. Nov. 18.

28,596. J. Hauff. Photographic developers. Complete Specification. Dec. 3.

28,920. J. W. Bennetto. Improvements in the production of coloured photographs, and apparatus for that purpose. Dec. 7.

29,093. K. Märten. An improved method of producing pictures upon glass by lithography in one or more colours. Dec. 9.

XXII.—EXPLOSIVES, MATCHES, Etc.

Applications.

26,896. C. A. McEvoy. Improvements in time and percussion fuses. Nov. 17.

26,962. J. C. Hamilton. Improvements in the manufacture of blasting compounds. Nov. 18.

27,100. T. Loehtie. Safety waterproof couplings for detonators and fuses. Nov. 19.

27,174. H. de Stedingk. An improvement connected with explosive cartridge fuses. Nov. 19.

27,234. J. Fraser. Improvements in mining and like fuses, and means for igniting same. Nov. 20.

27,520. J. Craveri. Improvements relating to the manufacture of safety matches and rubbing surfaces therefor. Complete Specification. Nov. 23.

27,912. J. C. Hamilton. Improvements in the detonation of certain blasting compounds. Nov. 27.

28,152. J. C. Hamilton. Improvements in or relating to blasting compounds. Nov. 30.

28,476. R. T. Maguire and D. P. Maguire. Improvements in or relating to lucifer matches. Dec. 2.

28,807. D. W. Nightingale. Improvements in explosives. Dec. 6.

29,005. L. Davies. Improvements in explosives. Dec. 8.

29,064. F. A. Debize and L. F. Chasseigne. Improvements in machinery for the continuous manufacture of wooden matches. Complete Specification. Dec. 8.

29,202. J. Joscht. Improvements in the manufacturing of matches. Dec. 9.

29,204. J. Bown. Improvements in fog signals. Dec. 9.

29,389. F. L. Thomas.—From La Société des Poudres et Dynamites, France. An improvement in explosives. Dec. 11.

Complete Specifications Accepted.

1896.

27,197. A. Nobel. Manufacture of explosive compounds. Dec. 8.

28,890. G. Kynoch and Co., Ltd., and A. T. Cocking. Manufacture of nitroglycerin. Dec. 8.

1897.

651. W. Greaves and E. M. Hann. Improvements in explosives. Dec. 15.

2557. M. Bieldefeldt. Manufacture of explosives. Dec. 8.

3135. H. R. von Dalmen. Improvements in blasting compositions. Dec. 8.

25,204. J. C. Chapman.—From C. W. Volney, United States. Manufacture of gunpowder. Dec. 8.

25,413. J. C. Chapman.—From C. W. Volney, United States. An improved gunpowder. Dec. 8.

XXIII.—ANALYTICAL CHEMISTRY.

Application.

27,373. C. E. Müller and R. F. Hurran. *See* Class I.

PATENTS UNCLASSIFIABLE.

Applications.

27,121. W. Leuscher. A process for the manufacture of burnt filtering mass. Complete Specification. Nov. 19.

27,661. M. Weishaus. Process for regenerating used whalebone and for dressing fin-whalebone without rendering the same brittle. Complete Specification. Nov. 24.

27,877. J. Rodgers. Improvements in the petrifaction or incrustation of porous or other substances. Nov. 26.

29,059. A. J. de Hailes. Improvements in preparations for rendering inflammable materials resistant to fire. Dec. 8.

29,387. L. C. Reese. Improvements in the extraction from liquids of matters therein contained. Dec. 11.

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